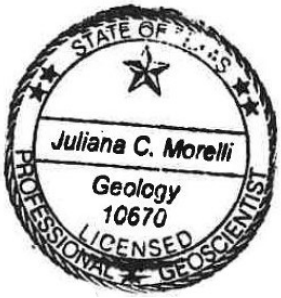
# MUNICIPAL SOLID WASTE PERMIT

# MAJOR AMENDMENT

## Part III – Attachment F

## Groundwater Characterization Report




Julie Morelli
9/15/2023

***NAME OF PROJECT: Beck Landfill***

***MSW PERMIT APPLICATION NO.: 1848A***

***OWNER: Nido, LTD (CN603075011)***

***OPERATOR: Beck Landfill (RN102310968)***

***CITY, COUNTY: Schertz, Guadalupe County***

*Major Amendment: September 2023*

Prepared by:

PROJECT NUMBER: 150051.05.01
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# TABLE OF CONTENTS

**1.0 Groundwater Certification Process for Arid Exemption (§330.63(e)(6)) .....1**
1.1. Groundwater Sampling and Analysis Plan (§330.63(f)) ..... 1
1.1.1 Applicability Statement (§330.401(f)) ..... 1
1.2. Groundwater Monitoring System (§330.403) ..... 2
1.3. Groundwater Monitoring at Type IV Landfills (§330.417) ..... 3
1.4. Monitor Well Construction Specifications (30 TAC §330.421) ..... 5
1.4.1 Monitoring Well and Piezometer Data Sheets ..... 7

## TABLES:

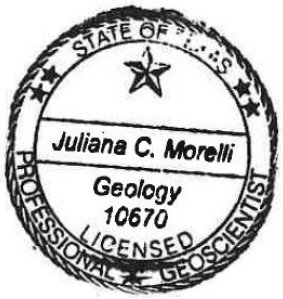
TABLE 1 MONITOR AND PIEZOMETER WELLS AT BECK LANDFILL ..... 3
TABLE 2 WATER WELLS AT BECK LANDFILL ..... 7

## FIGURES:

FIGURE 3-F-1 GROUNDWATER GRADIENT MAP

## APPENDICES

APPENDIX F-1 MONITOR WELL INSTALLATION INFORMATION
APPENDIX F-2 HISTORIC GROUNDWATER DATA
APPENDIX F-3 GROUNDWATER SAMPLING AND ANALYSIS PLAN

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9/15/2023

# 1.0 Groundwater Certification Process for Arid Exemption (§330.63(e)(6))

Not applicable - Beck is not seeking an arid exemption for the landfill, therefore this section does not apply.

## 1.1. Groundwater Sampling and Analysis Plan (§330.63(f))

*(f) Groundwater sampling and analysis plan. The groundwater sampling and analysis plan for landfills and if otherwise requested by the executive director for other MSW units must be prepared in accordance with Subchapter J of this chapter (relating to Groundwater Monitoring and Corrective Action).*

Beck Landfill is a Type IV Landfill subject to the groundwater monitoring requirements promulgated in 30 TAC 330, Subchapter J, and more specifically those outlined in 30 TAC 330.417. The Facility revised the Groundwater Sampling and Analysis Plan (GWSAP) (TCEQ Minor Modification approved 2013) in compliance with the monitoring requirements for Type IV Landfills in 30 TAC §330 Subchapter J. The full GWSAP is attached as **Appendix F-3**, for consistency with the application format.

### 1.1.1 Applicability Statement (§330.401(f))

*(f) Once established at a solid waste management unit, groundwater monitoring must be conducted throughout the active life and any required post-closure care period of that solid waste management unit as specified in §330.463 of this title (relating to Post-Closure Care Requirements).*

Beck Landfill has an existing groundwater monitoring system, installed in 1998 and 2000. Background monitoring was performed quarterly from August 2000 to August 2001. Annual detection monitoring has been performed each year since then. Beck Landfill will conduct groundwater monitoring throughout the active life and any required post-closure care period, as required by MSW Permit No. 1848A.

## 1.2. Groundwater Monitoring System (§330.403)

*(a) A groundwater monitoring system must be installed that consists of a sufficient number of monitoring wells, installed at appropriate locations and depths, to yield representative groundwater samples from the uppermost aquifer as defined in §330.3 of this title (relating to Definitions)*

An existing, TCEQ-approved groundwater monitoring system is in place and in use at the Facility (TCEQ Class I Permit Modification dated July 12, 2000). The System is comprised of five (5) monitoring wells installed on the outside of the flood control dike (impermeable barrier to prevent migration of contaminants from with the landfill) and installed at a depth to intersect the confining layer (the Navarro Formation) of the perched alluvial water table. The monitor wells are screened to intercept the saturated zone of the alluvium. Wells are provided with a protective, steel collar and stick up approximately 36" from the concrete pad. Each well is protected with a lockable, water-tight cap and enclosed within a lockable steel collar.

In addition, Beck Landfill installed five (5) piezometer wells in correlation with the five (5) monitor wells. The piezometer wells are installed between the landfill and the flood control dike (inside the landfill), at a depth to intersect the confining layer (the Navarro Formation), identical to its corresponding monitor well. These wells are similarly screened. No concrete pad was installed with the piezometer wells. Each well is flush-mounted and is protected with a lockable, water-tight cap. The well is protected by a flush mount iron collar with a bolted on lid.

All parts of the monitoring system shall be operated and maintained so they perform as designed. **Table 1** below documents the relevant information regarding the monitor and piezometer wells approved for use at Beck Landfill.

Beck proposes to plug and abandon MW-D and install a replacement well along Line E (MW-E) in accordance with the design criteria established above. The current MW-D well location is situated in proximity to the proposed stormwater collection pond and may not be as representative of groundwater conditions due to potential influence from the proposed pond.

**TABLE 1 MONITOR AND PIEZOMETER WELLS AT BECK LANDFILL**

| Well ID No. | Installation Date | Well Pad Elevation (ft. above msl) | Well Depth Elevation (ft. above msl) | Total Depth (feet) | Monitoring Performed |
|---|---|---|---|---|---|
| MW-A | May 20, 1998 | 712.61 | 673.93 | 38.68 | Annual Detection Monitoring; Background in 2000 |
| 712.61PZ-A | May 20, 1998 | 712.59 | 673.13 | 39.46 | Informational only |
| MW-C | May 20, 1998 | 712.65 | 666.56 | 46.09 | Annual Detection Monitoring |
| PZ-C | May 20, 1998 | 712.85 | 671.46 | 41.39 | Informational only |
| MW-D (to be replaced by MW-E) | February 29, 2000 | 708.05 | 665.67 | 42.39 | Annual Detection Monitoring |
| PZ-D (to be replaced by PZ-E) | May 20, 1998 | N/A | | 38.15 | Informational only |
| MW-E | Proposed | TBD | TBD | TBD | To replace MW-D |
| PZ-E | Proposed | TBD | TBD | TBD | To replace PZ-D |
| MW-F | May 20, 1998 | 702.52 | 666.00 | 36.52 | Annual Detection Monitoring |
| PZ-F | May 20, 1998 | 702.51 | 669.2 | 33.31 | Informational only |
| MW-G | May 20, 1998 | 700.59 | 663.61 | 36.98 | Annual Detection Monitoring |
| PZ-G | May 20, 1998 | 700.54 | 668.09 | 32.45 | Informational only |

## 1.3. Groundwater Monitoring at Type IV Landfills (§330.417)

*(b) At the discretion of the executive director, the owner or operator of a Type IV landfill may be required to installed groundwater monitoring systems and to monitor on a regular basis the quality of groundwater at the point of compliance.*

See Section 1.2 above.

*(3) Groundwater sampling and analysis requirements shall be in accordance with §330.405(a)-(d) of this title (relating to Groundwater Sampling and Analysis Requirements).*

The GWSAP conforms to the requirements set forth in 30 TAC 330.405(a)-(d) (see **Appendix F-3**).

*(4) Each monitoring well or other sampling point shall be sampled and analyzed annually, or on some other schedule but not less frequently than annually as determined by the executive director, for the following constituents: chloride, iron, manganese, cadmium, zinc, total dissolved solids, specific conductance (field and laboratory measurements), pH (field and laboratory measurements), and non-purgeable organic commands.*

The GWSAP identifies annual detection monitoring and includes required parameters as outlined in this rule.

*(5) Not later than 60 days after each sampling event, the owner or operator shall determine whether the landfill has released contaminants to the uppermost aquifer. The owner or operator shall provide an annual detection monitoring report within 60 days after the facility's annual groundwater monitoring event that includes the following information determined since the previously submitted report:*

*(A) the results of all monitoring, testing, and analytical work obtained or prepared in accordance with the requirements of this permit, including a summary of background groundwater quality values, groundwater monitoring analyses, any statistical calculations, graphs, and drawings;*

*(B) the groundwater flow rate and direction in the uppermost aquifer. The groundwater flow rate and direction of groundwater flow shall be established using the data collected during the preceding calendar year's sampling events from the monitoring wells of the Detection Monitoring Program. The owner or operator shall also include in the report all documentation used to determine the groundwater flow rate and direction of groundwater flow;*

*(C) a contour map of piezometric water levels in the uppermost aquifer based at a minimum upon concurrent measurement in all monitoring wells. All data or documentation used to establish the contour map should be included in the report;*

*(D) recommendation for any changes; and*

*(E) any other items requested by the executive director.*

Beck Landfill submits an Annual Groundwater Monitoring Event Report that conforms with the required elements above.

*(6) The executive director may require additional sampling, analyses of additional constituents, installation of additional monitoring wells or other sampling points, and/or other hydrogeological investigations if the facility appears to be contaminating the uppermost aquifer.*

No additional constituents are included in MSW Permit No. 1848A.

## 1.4. Monitor Well Construction Specifications (30 TAC §330.421)

Monitor wells were installed for the purpose of sampling and testing groundwater adjacent to the landfill as a provision of quality assurance. The protection of the groundwater quality in the area of the landfill is a major concern of the landfill operator, the TCEQ, and the public. Monitor wells on this site were installed by Jedi Drilling, a licensed Texas Water Well Driller in February 1998, with a replacement of Monitor Well D (MW-D) installed on February 20, 2000. The wells were completed in accordance with Texas Water Commission regulations in place at the time of installation. The wells are used to monitor the quality of water found in the shallow, perched Alluvial system. Water associated with the Edwards Aquifer, located approximately 500 feet beneath the site, is not to monitored, as interconnection is not anticipated.

The gradient of the shallow groundwater beneath the landfill site currently exists as depicted in **Part III-F, Figure 3-F-1,** based on historic annual detection monitoring at the landfill. The installation of the slurry wall creates a hydraulic barrier between the Landfill and the Cibolo Creek, effectively stopping the hydraulic connection inside the Landfill. Groundwater is directed around the slurry wall rather than beneath the site.

Monitor well MW-A as depicted on **Part III, Figure 3-F-1** is the primary upgradient well. Wells MW-C and MW-G are predominately upgradient but are situated so as to detect and aid in isolating any leachate, should such ever become apparent. Wells MW-D and MW-F are downgradient.

The monitor wells are variable in depth corresponding to the existing strata variations in the alluvial aquifer and underlying shale. An approximate 20-foot depth plus the height of the dike,

was considered as an average for the proposed wells, or an average of 40 feet. The static water table, or the first potable aquifer being the Alluvial aquifer comprised of the sand and gravel deposits overlying the shales beneath the site, is the zone to be monitored. The rate of groundwater flow relates to the flow of Cibolo Creek and is variable.

Details of proposed monitor well construction were provided by Snowden. These well construction details have been updated to more closely represent the wells installed at the Landfill, based on surface observations (see **Appendix F-1**). The top of the wells were completed a minimum of 24 inches above the finish grade of the dike, which at the time, required the dike to be above the (then) 100-year flood plain. A 4-ft square by 4-inch minimum thickness sloped concrete sealing block was cast around the monitor wells at the top of the dike. Other construction parameters were as per the Water Well Drillers Act, Chapter 319-Standards for Completion with the most stringent of these standards being applicable. Permanent well identification plates are installed on each stick-up on each well.

The monitor wells were located upon an extended section of the dike. Such location did not comply with the specifications of the Water Well Drillers Act in terms of horizontal separation. The location is however the only method by which the monitor wells could be maintained above the 100-year flood plain and allow accessibility for sample extraction. The required horizontal separation is further inappropriate and otherwise differed as said separation would require location in Cibolo Creek and/or beyond the boundaries of the landfill property.

The monitor wells have an extended screened or blank section of schedule 40-ft PVC extending below the saturated zone to a depth equivalent to that of the slurry wall key. Said extended screen-blank section of pipe is a minimal provision of storage, as it is possible that during certain periods of any given year a low yield characteristic could occur in the vicinity of some monitor wells. Provisions to assure sample freshness, with regards to the blank section, are addressed within Groundwater Sampling and Analysis Plan (GWSAP) (**Appendix F-3** of this Report).

Background data was generated through the use of samples recovered directly from Cibolo Creek. Records of these samples were not located in this amendment application and are believed to have been destroyed during flooding. However, background monitoring is included, as well as all detection data gathered since the monitor wells were installed.

### 1.4.1 Monitoring Well and Piezometer Data Sheets

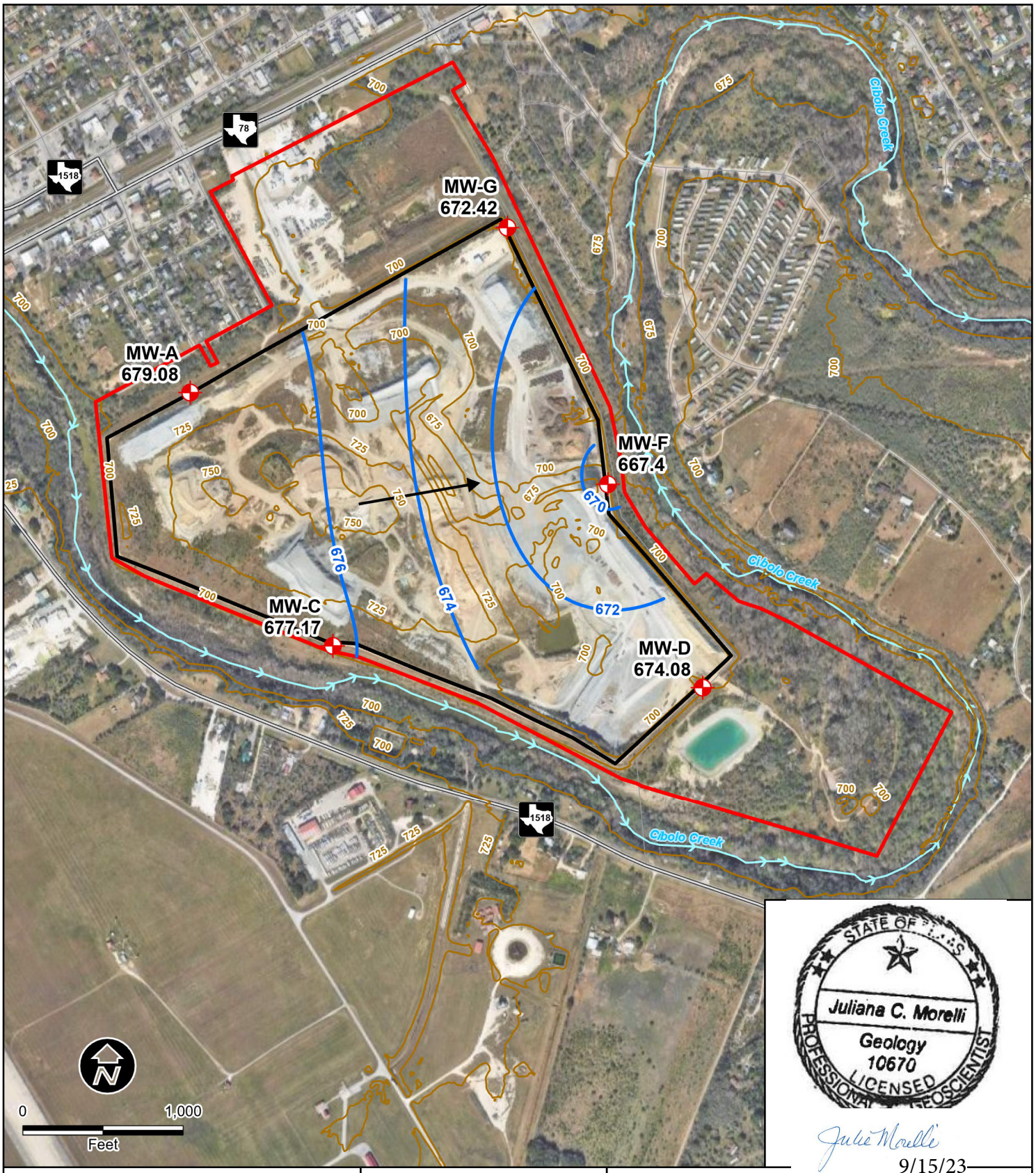
On May 20th, 1998, Jedi (TNRCC Driller License No. 50205-M) installed a series of five monitoring wells and five piezometers at the Beck Landfill under the supervision of Harley Weld. The well on Line D (MW-D) was replaced on February 20, 2000. The TNRCC MSW-SE67 monitor well data sheets for each monitoring well and piezometer are attached as **Appendix F-1** and have been updated with survey elevations on the stick-up collars taken on September 10, 2023. Included in the TNRCC data sheets is relevant information pertaining to the construction of monitoring well and piezometer on-site including elevations, depths, cross sections, and dimensions. Each monitoring well and piezometer was reported to have been dry following installation.

The locations of all existing and abandoned wells at the Beck Landfill are depicted in **Table 2** below. The on-site wells are utilized for groundwater quality monitoring in accordance with the existing MSW permit. No other active or historical wells within the Beck Landfill facility are depicted on the Texas Water Development Board (TWDB) Groundwater Data Viewer (TWDB, accessed September 6, 2022). Beck will replace MW-D and Piezometer D with a similar well installed along Line E to accommodate the installation of the proposed stormwater drainage pond.

**TABLE 2 WATER WELLS AT BECK LANDFILL**

| Well | Use | Latitude and Longitude |
|---|---|---|
| MW-A | Groundwater monitoring of perched aquifer outside of landfill dike-line. | 29.548880°, -98.268411° |
| MW-C | Groundwater monitoring of perched aquifer outside of landfill dike-line. | 29.544524°, -98.265643° |
| MW-D | Groundwater monitoring of perched aquifer outside of landfill dike-line. | 29.543768°, -98.258393° |
| MW-F | Groundwater monitoring of perched aquifer outside of landfill dike-line. | 29.547263°, -98.260227° |
| MW-G | Groundwater monitoring of perched aquifer outside of landfill dike-line. | 29.551674°, -98.262166° |
| Piezometer A | Groundwater monitoring of leachate inside of the landfill dike-line | 29.548868°, -98.268394° |
| Piezometer C | Groundwater monitoring of leachate inside of the landfill dike-line | 29.544557°, -98.265645° |
| Piezometer D | Groundwater monitoring of leachate inside of the landfill dike-line | 29.543796°, -98.258427° |
| Piezometer F | Groundwater monitoring of leachate inside of the landfill dike-line | 29.547273°, -98.260264° |
| Piezometer G | Groundwater monitoring of leachate inside of the landfill dike-line | 29.551662°, -98.262213° |

**FIGURE 3-F-1 GROUNDWATER GRADIENT MAP**

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9/15/23

**Legend**

- Permit Boundary
- Landfill Boundary
- Monitoring Well
- Surface Elevation (ft)
- Generalized Piezometric Surface (ft)
- Inferred Groundwater Flow Direction
- NHD River or Stream

Map Sources: Google Earth Imagery


NIDO, LTD
BECK LANDFILL

# FIGURE 3-F-1
# GROUNDWATER GRADIENT MAP

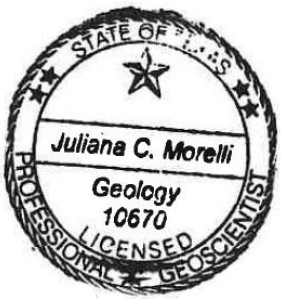
SCHERTZ, GUADALUPE COUNTY, TEXAS

POWER ENGINEERS

Date: 9/15/2023
Rev. 0

Path: G:\Projects\0_Beck\150051 Landfill GIS\150051_Landfill_GIS\150051_Landfill_GIS.aprx

# APPENDIX F-1 MONITOR WELL INSTALLATION INFORMATION




Julie Morelli
9/15/2023

This seal indicates that the following sheets are as provided to TCEQ at well installation.

Additional survey information has been obtained to clarify the elevations of the stick-up collars and lockable metal protective covers that are installed to prevent inundation to the existing wells.

The following table provides the survey information collected on September 10, 2023.

| NORTHING | EASTING | ELEVATION | DESCRIPTION |
|---|---|---|---|
| 13748867.35 | 2203077.243 | 700.82 | MONITORING WELL-G |
| 13747267.63 | 2203707.327 | 702.657 | MONITORING WELL-F |
| 13745998.19 | 2204296.054 | 710.028 | MONITORING WELL-D |
| 13746249.68 | 2201982.801 | 711.914 | MONITORING WELL-C |
| 13747842.9 | 2201097.146 | 712.164 | MONITORING WELL-A |

# A. Monitor Well Data Sheet

TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.

MSW PERMIT NO: 1848

County: Guadalupe

Monitor Well I.D. No.: A-22+25W

Date of Monitor Well Installation: 5-20-98

Date of Monitor Well Development: _____

Monitor Well: Latitude: _____ Longitude: _____

Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___

Monitor Well Driller Name: JEDI

License No.: 50205-M

NOTE:

(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid

Static Water Level Elevation (with respect to MSL) after Well Development: dry

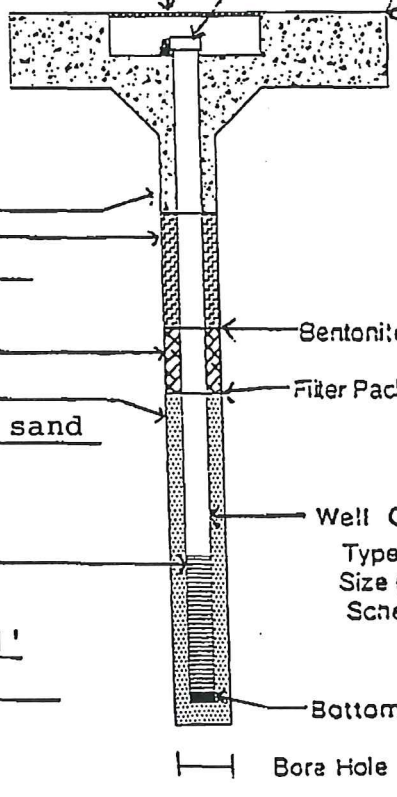
Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor

Type of Locking Device: top lock cap & bolted metal lid

Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.
Surface Pad Dimensions: 6' x 6'

Surface Elevation: 712.61'



Top of Protective Collar Elevation: 712.61'

Top of Casing Elevation: 712.28'

Surveyor's Pin Elevation: 712.61'

Concrete Seal Depth: 0' to 26'

Casing Seal (Backfill) Material: cement

Bentonite Seal

Bentonite Seal Top Depth: 26' Elevation: 686.61'

Filter Pack

Filter Pack Top Depth: 28' Elevation: 684.61'

Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Well Casing Type: 0.010 PVC
Size (diameter): 4"
Schedule or Thickness: Sch. 40

Well Screen
Top Depth: 30'
Top Elevation: 682.61'
Type of Well Screen: PVC
Screen Opening Size: 4"

Bottom Cap (Depth: 40')

Bore Hole Diameter: 8"

# A. Monitor Well Data Sheet

TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.

MSW PERMIT NO: 1848

County: Guadalupe

Monitor Well I.D. No.: C-14+50W

Date of Monitor Well Installation: 5-20-98

Date of Monitor Well Development: _____

Monitor Well: Latitude: _____ Longitude: _____

Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___

Monitor Well Driller Name: JEDI

License No.: 50205-M

NOTE:

(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid

Static Water Level Elevation (with respect to MSL) after Well Development: dry

Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor

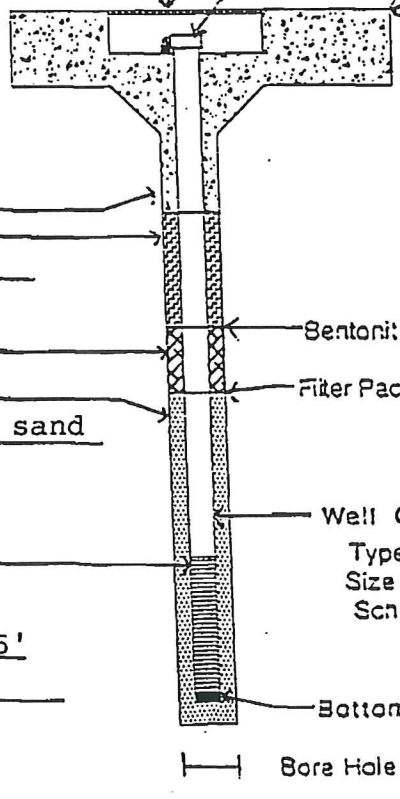
Type of Locking Device: top lock cap & bolted metal lid

Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.

Surface Pad Dimensions: 6' x 6'

Surface Elevation: 712.65'



Top of Protective Collar Elevation: 712.65'

Top of Casing Elevation: 712.32'

Surveyor's Pin Elevation: 712.65'

Concrete Seal Depth: 0' to 32'

Casing Seal (Backfill) Material: cement

Bentonite Seal

Filter Pack

Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Bentonite Seal Top Depth: 32' Elevation: 680.65'

Filter Pack Top Depth: 34' Elevation: 678.65'

Well Casing
Type: 0.010 PVC
Size (diameter): 4"
Schedule or Thickness: Sch. 40

Well Screen
Top Depth: 36'
Top Elevation: 676.65'
Type of Well Screen: PVC
Screen Opening Size: 4"

Bottom Cap (Depth: 46')

Bore Hole Diameter: 8"

# A. Monitor Well Data Sheet

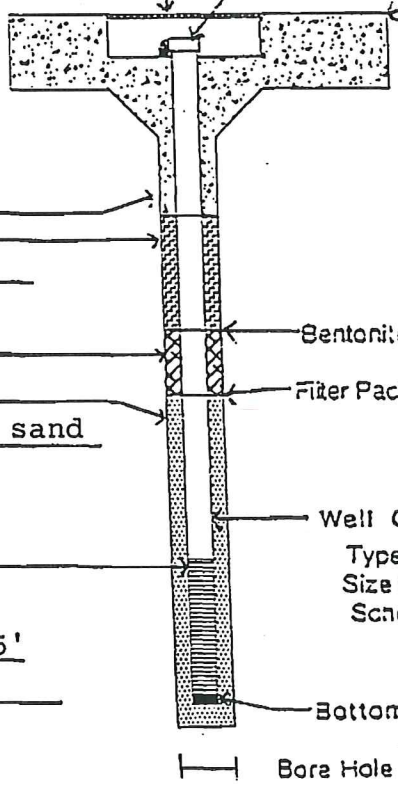
TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.
MSW PERMIT NO: 1848
County: Guadalupe
Monitor Well I.D. No. B-7+25W
Date of Monitor Well Installation: 5-20-98
Date of Monitor Well Development:
Monitor Well: Latitude: _____ Longitude: _____
Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___
Monitor Well Driller Name: JEDI
License No.: 50205-M

NOTE:
(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid
Static Water Level Elevation (with respect to MSL) after Well Developement: dry
Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor
Type of Locking Device: top lock cap & bolted metal lid
Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.
Surface Pad Dimensions: 6' x 6'
Surface Elevation: 708.05'



Top of Protective Collar Elevation: 708.05'
Top of Casing Elevation: 707.62'
Surveyor's Pin Elevation: 708.05'

Concrete Seal
Depth: 0' to 25'
Casing Seal (Backfill) Material: cement

Bentonite Seal
Filter Pack
Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Bentonite Seal Top Depth: 25' Elevation: 683.05'
Filter Pack Top Depth: 27' Elevation: 681.05'

Well Casing
Type: 0.010 PVC
Size (diameter): 4"
Schedule or Thickness: Sch. 40

Well Screen
Top Depth: 29'
Top Elevation: 679.05'
Type of Well Screen: PVC
Screen Opening Size: 4"

Bottom Cap (Depth: 39')

Bore Hole Diameter: 8"

# A. Monitor Well Data Sheet

TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.

MSW PERMIT NO: 1848

County: Guadalupe

Monitor Well I.D. No.: F-2+00W

Date of Monitor Well Installation: 5-20-98

Date of Monitor Well Development: _____

Monitor Well: Latitude: _____ Longitude: _____

Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___

Monitor Well Driller Name: JEDI

License No.: 50205-M

NOTE:

(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid

Static Water Level Elevation (with respect to MSL) after Well Developement: dry

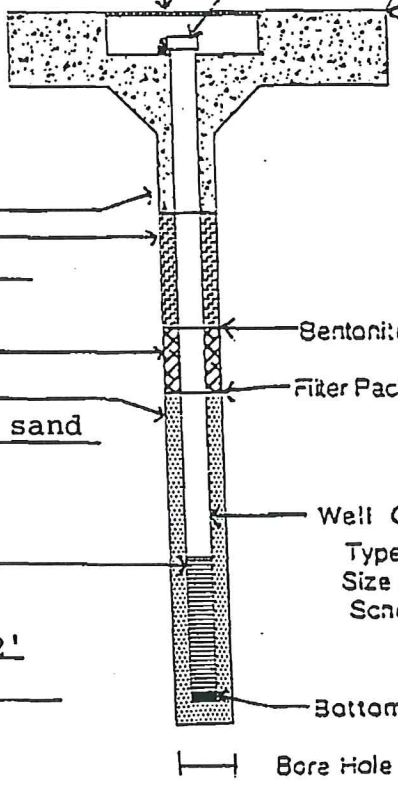
Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor

Type of Locking Device: top lock cap & bolted metal lid

Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.
Surface Pad Dimensions: 6' x 6'

Surface Elevation: 702.52'



Top of Protective Collar Elevation: 702.52'

Top of Casing Elevation: 702.19'

Surveyor's Pin Elevation: _____

Concrete Seal Depth: 0' to 20'

Casing Seal (Backfill) Material: cement

Bentonite Seal

Filter Pack

Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Bentonite Seal Top Depth: 20' Elevation: 682.52'

Filter Pack Top Depth: 22' Elevation: 680.52'

Well Casing
Type: 0.010 PVC
Size (diameter): 4"
Schedule or Thickness: Sch. 40

Well Screen
Top Depth: 24'
Top Elevation: 678.52'
Type of Well Screen: PVC
Screen Opening Size: 4"

Bottom Cap (Depth: 34')

Bore Hole Diameter: 8"

# A. Monitor Well Data Sheet

TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.

MSW PERMIT NO: 1848

County: Guadalupe

Monitor Well I.D. No.: G-13+25W

Date of Monitor Well Installation: 5-20-98

Date of Monitor Well Development: _____

Monitor Well: Latitude: _____ Longitude: _____

Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___

Monitor Well Driller Name: JEDI

License No.: 50205-M

NOTE:

(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

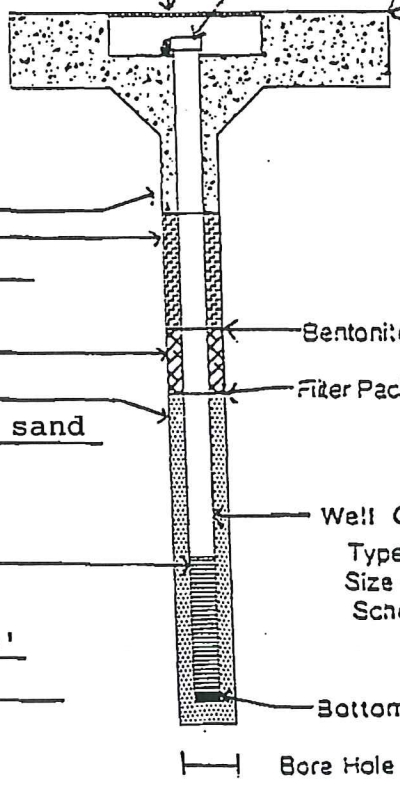
Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid

Static Water Level Elevation (with respect to MSL) after Well Developement: dry

Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor

Type of Locking Device: bolted metal lid top lock cap & Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.
Surface Pad Dimensions: 6' x 6'
Surface Elevation: 700.59



Top of Protective Collar Elevation: 700.59
Top of Casing Elevation: 700.26'
Surveyor's Pin Elevation: 700.59'

Concrete Seal Depth: 0' to 23'
Casing Seal (Backfill) Material: cement

Bentonite Seal
Filter Pack
Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Bentonite Seal Top Depth: 23' Elevation: 677.59'
Filter Pack Top Depth: 25' Elevation: 675.59'

Well Casing
Type: 0.010 PVC
Size (diameter): 4"
Schedule or Thickness: Sch. 40

Well Screen
Top Depth: 27'
Top Elevation: 673.59'
Type of Well Screen: PVC
Screen Opening Size: 4"

Bottom Cap (Depth: 37')

Bore Hole Diameter: 8"

# A. Monitor Well Data Sheet

TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.

MSW PERMIT NO: 1848

County: Guadalupe

Monitor Well I.D. No.: A-22+25P

Date of Monitor Well Installation: 5-20-98

Date of Monitor Well Development: _____

Monitor Well: Latitude: _____ Longitude: _____

Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___

Monitor Well Driller Name: JEDI

License No.: 50205-M

NOTE:

(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

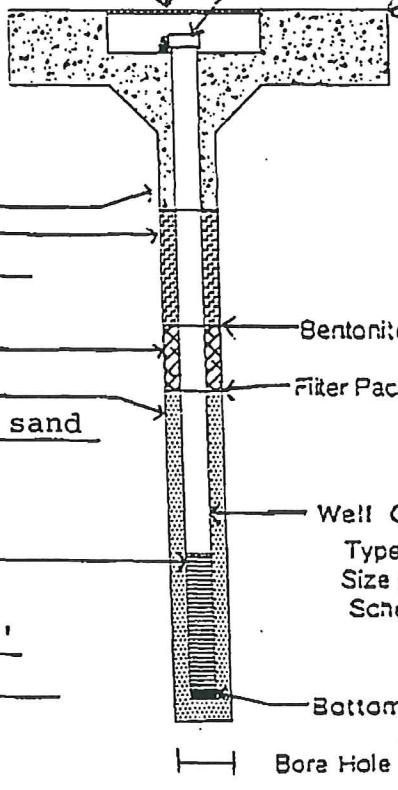
Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid

Static Water Level Elevation (with respect to MSL) after Well Developement: dry

Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor

Type of Locking Device: bolted metal lid top lock cap & Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.
Surface Pad Dimensions: 6' x 6'
Surface Elevation: 712.59'



Top of Protective Collar Elevation: 712.59'
Top of Casing Elevation: 712.26'
Surveyor's Pin Elevation: 712.59'

Concrete Seal Depth: 0' to 25'
Casing Seal (Backfill) Material: cement

Bentonite Seal
Filter Pack
Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Bentonite Seal Top Depth: 25' Elevation: 687.59'
Filter Pack Top Depth: 27' Elevation: 685.59'

Well Casing
Type: 0.010 PVC
Size (diameter): 4"
Schedule or Thickness: Sch. 40

Well Screen
Top Depth: 29'
Top Elevation: 683.59'
Type of Well Screen: PVC
Screen Opening Size: 4"

Bottom Cap (Depth: 39')

Bore Hole Diameter: 8"

# A. Monitor Well Data Sheet

TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.

MSW PERMIT NO: 1848

County: Guadalupe

Monitor Well I.D. No.: C-14+50P

Date of Monitor Well Installation: 5-20-98

Date of Monitor Well Development: _____

Monitor Well: Latitude: _____ Longitude: _____

Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___

Monitor Well Driller Name: JEDI

License No.: 50205-M

NOTE:

(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid

Static Water Level Elevation (with respect to MSL) after Well Development: dry

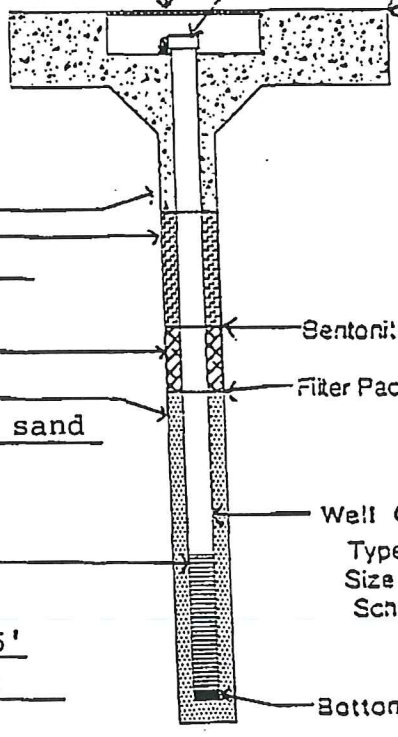
Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor

Type of Locking Device: top lock cap & bolted metal lid

Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.
Surface Pad Dimensions: 6' x 6'

Surface Elevation: 712.85'



Top of Protective Collar Elevation: 712.85'

Top of Casing Elevation: 712.52'

Surveyor's Pin Elevation: 712.85'

Concrete Seal Depth: 0' to 30'

Casing Seal (Backfill) Material: cement

Bentonite Seal

Filter Pack

Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Bentonite Seal Top Depth: 30' Elevation: 682.85'

Filter Pack Top Depth: 32' Elevation: 680.85'

Well Casing
Type: 0.010 PVC
Size (diameter): 4"
Schedule or Thickness: Sch. 40

Well Screen
Top Depth: 34'
Top Elevation: 678.85'
Type of Well Screen: PVC
Screen Opening Size: 4"

Bottom Cap (Depth: 44')

Bore Hole Diameter: 8"

# A. Monitor Well Data Sheet

TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.
MSW PERMIT NO: 1848

County: Guadalupe
Monitor Well I.D. No: D-7+25P

Date of Monitor Well Installation: 5-20-98
Date of Monitor Well Development: _____

Monitor Well: Latitude: _____ Longitude: _____

Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___

Monitor Well Driller Name: JEDI
License No.: 50205-M

NOTE:

(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

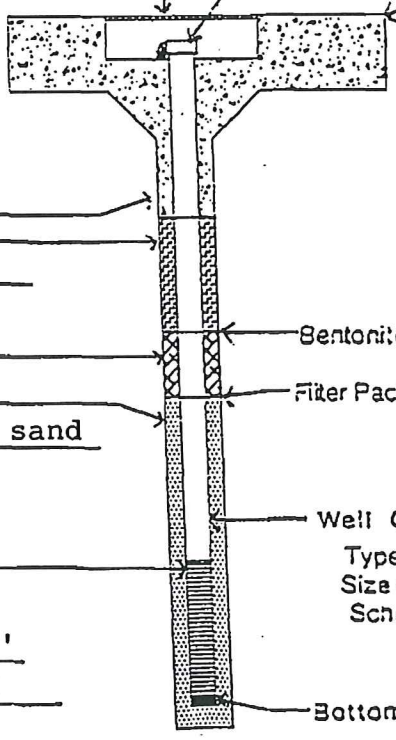
Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid

Static Water Level Elevation (with respect to MSL) after Well Developement : dry

Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor

Type of Locking Device: top lock cap & bolted metal lid
Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.
Surface Pad Dimensions: 6' x 6'
Surface Elevation: 706.49'



Top of Protective Collar Elevation: 708.49'
Top of Casing Elevation: 708.16'
Surveyor's Pin Elevation: 708.49'

Concrete Seal
Depth: 0' to 20'
Casing Seal (Backfill) Material: cement

Bentonite Seal
Filter Pack
Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Bentonite Seal Top Depth: 20' Elevation: 688.49'
Filter Pack Top Depth: 22' Elevation: 686.49'

Well Casing
Type: 0.010 PVC
Size (diameter) : 2"
Schedule or Thickness: Sch. 40

Well Screen
Top Depth: 24'
Top Elevation: 684.49'
Type of Well Screen: PVC
Screen Opening Size: 4"

Bottom Cap (Depth: 34')

Bore Hole Diameter: 8"

# A. Monitor Well Data Sheet

TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.
MSW PERMIT NO: 1848

County: Guadalupe
Monitor Well I.D. No.: F-2+00P

Date of Monitor Well Installation: 5-20-98
Date of Monitor Well Development: _____

Monitor Well: Latitude: _____ Longitude: _____

Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___

Monitor Well Driller Name: JEDI
License No.: 50205-M

NOTE:

(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

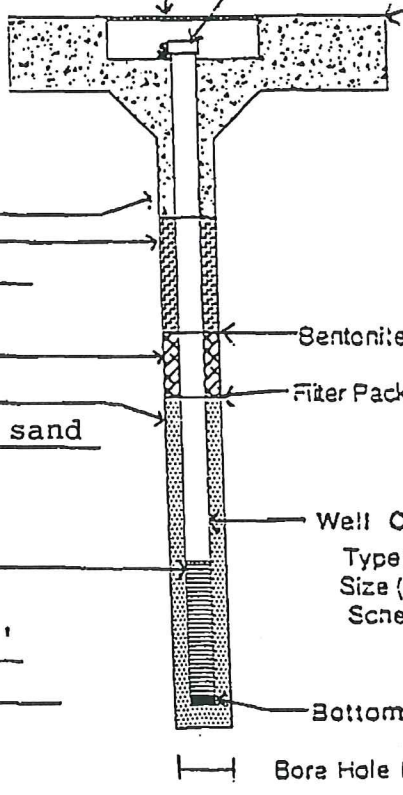
Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid

Static Water Level Elevation (with respect to MSL) after Well Developement: dry

Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor

Type of Locking Device: top lock cap & bolted metal lid
Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.
Surface Pad Dimensions: 6' x 6'
Surface Elevation: 702.51'



Top of Protective Collar Elevation: 702.51'
Top of Casing Elevation: 702.18'
Surveyor's Pin Elevation: 702.51'

Concrete Seal
Depth: 0' to 20'
Casing Seal (Backfill) Material: cement

Bentonite Seal
Filter Pack
Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Bentonite Seal Top Depth: 20' Elevation: 682.51'
Filter Pack Top Depth: 22' Elevation: 680.51'

Well Casing
Type: 0.010 PVC
Size (diameter): 2"
Schedule or Thickness: Sch. 40

Well Screen
Top Depth: 24'
Top Elevation: 678.51'
Type of Well Screen: PVC
Screen Opening Size: 4"

Bottom Cap (Depth: 34')

Bore Hole Diameter: 8"

# A. Monitor Well Data Sheet

TEXAS NATURAL RESOURCE CONSERVATION COMMISSION
MSWD-SE67

Permittee or Site Name: Beck Readymix Concrete Co.

MSW PERMIT NO: 1848

County: Guadalupe

Monitor Well I.D. No.: G-13+25P

Date of Monitor Well Installation: 5-20-98

Date of Monitor Well Development: _____

Monitor Well: Latitude: _____ Longitude: _____

Monitor Well Groundwater Gradient: Upgradient ___ Downgradient ___

Monitor Well Driller Name: JEDI

License No.: 50205-M

NOTE:

(A) The information shown in the sketch below should be considered the minimum required for an installed ground-water monitor well.
(B) Report All Depths from Surface Elevation and all Elevations relative to Mean Sea Level.
(C) The minimun distance between the inside wall of the Bore Hole and the outside of the Well Casing shall be 3".
(D) Use Flush Screw Joint Casing only, 2" diameter or larger. Recommend 4" diameter minimum & Teflon Taping Casing Joints.
(E) Well development should continue until water is clear, and pH and conductivity are stable.

Geologist, Hydrologist or Engineer Supervising Well Installation: Harley Weid

Static Water Level Elevation (with respect to MSL) after Well Developement: dry

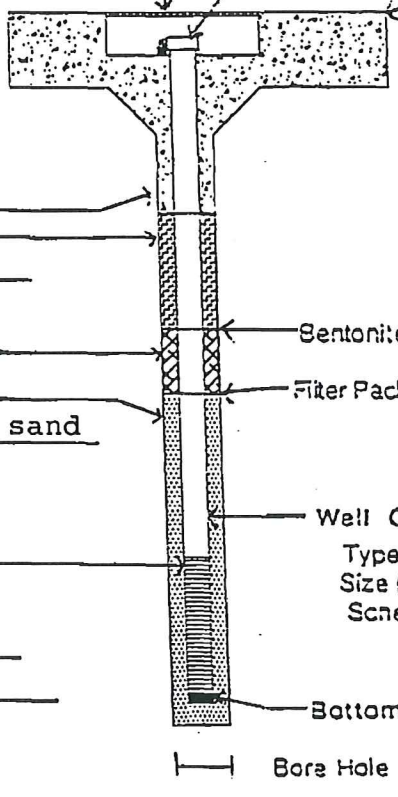
Name of Geologic Formation(s) in which Well is completed: Navarro/Taylor

Type of Locking Device: top lock cap & bolted metal lid

Type of Casing Protection: stand up well cover

Concrete Surface Pad - Recommend steel reinforcement in the Surface Pad.
Surface Pad Dimensions: 6' x 6'

Surface Elevation: 700.54'



Top of Protective Collar Elevation: 700.54'

Top of Casing Elevation: 700.21'

Surveyor's Pin Elevation: 700.54'

Concrete Seal Depth: 0' to 25'

Casing Seal (Backfill) Material: cement

Bentonite Seal

Filter Pack

Filter Pack Material: 20/40 sand
Sterilized Sand or Glass Beads

Bentonite Seal Top Depth: 25' Elevation: 675.54'

Filter Pack Top Depth: 27' Elevation: 673.54'

Well Screen

Top Depth: 29'

Top Elevation: _____

Type of Well Screen: PVC

Screen Opening Size: 4"

Well Casing
Type: 0.010 PVC
Size (diameter): 4"
Schedule or Thickness: Sch. 40

Bottom Cap (Depth: 39')

Bore Hole Diameter: 8"

Jan 19 99 8:56 P.01

Send original copy by certified return receipt requested mail to: CC, MC 177, P.O. Box 13087, Austin, TX 78711-3087

ATTENTION OWNER: Confidentiality Privilege Notice on on reverse side of Well Owner's copy (pink)

# State of Texas
# WELL REPORT

Texas Water Well Drillers Advisory Council
MC 177
P.O. Box 13087
Austin, TX 78711-3087
512-239-6530

1) OWNER Ben Davis (Name) ADDRESS P.O. Box 790641 S.A. (Street or RFD) Tx. 78279-0641 (City) (State) (Zip)

2) ADDRESS OF WELL: County Guadalupe 550 Fm 78 (Street, RFD or other) Shertz (City) Tx (State) 78108 (Zip) GRID # 68-30-6

3) TYPE OF WORK (Check): [X] New Well [ ] Deepening [ ] Reconditioning [ ] Plugging

4) PROPOSED USE (Check): [X] Monitor [ ] Environmental Soil Boring [ ] Domestic [ ] Industrial [ ] Irrigation [ ] Injection [ ] Public Supply [ ] De-watering [ ] Test well
If Public Supply well, were plans submitted to the TNRCC? [ ] Yes [ ] No

5)

6) WELL LOG: [redacted]
Date Drilling: [illegible]
Started 5-18 19 98
Completed 5-20 19 98

| DIAMETER OF HOLE | | |
|---|---|---|
| Dia. (in.) | From (ft.) | To (ft.) |
| 8 | Surface | [redacted] 44 |

7) DRILLING METHOD (Check): [ ] Driven [ ] Air Rotary [ ] Mud Rotary [ ] Bored [ ] Air Hammer [ ] Cable Tool [ ] Jetted [X] Other HSA

N

| From (ft.) | To (ft.) | Description and color of formation material |
|---|---|---|
| 0 | 2 | Roadbase gravel |
| 2 | [illegible]12 | Tan sandy clay fill |
| 12 | 44 | blue shale |

(Use reverse side of Well Owner's copy, if necessary)

8) Borehole Completion (Check): [ ] Open Hole [ ] Straight Wall [ ] Underreamed [ ] Gravel Packed [X] Other 2"dia SCH 40
If Gravel Packed give interval ... from _____ ft. to _____ ft.

CASING, BLANK PIPE, AND WELL SCREEN DATA:

| Dia. (in.) | New or Used | Steel, Plastic, etc. Perf., Slotted, etc. Screen Mfg., if commercial | Setting (ft.) From | Setting (ft.) To | Gage Casing Screen |
|---|---|---|---|---|---|
| 2 | N | Blank | [illegible] | [illegible] | [illegible] |
| 2 | N | Screen | [illegible] | [illegible] | 0.010 |

all wells

9) CEMENTING DATA [Rule 338.44(1)]
Cemented from 0 ft. to 20 ft. No. of sacks used 1.5
_____ ft. to _____ ft. No. of sacks used _____
Method used bentonite
Cemented by Eric Jones / JEDI
Distance to septic system field lines or other concentrated contamination _____ ft.
Method of verification of above distance _____

10) SURFACE COMPLETION
[X] Specified Surface Slab Installed [Rule 338.44(2)(A)]
[ ] Specified Steel Sleeve Installed [Rule 338.44(3)(A)]
[ ] Pitless Adapter Used [Rule 338.44(3)(D)]
[ ] Approved Alternative Procedure Used [Rule 338.71]

11) WATER LEVEL:
Static level Dry ft. below land surface Date 5-20-98
Artesian flow _____ gpm. Date _____

12) PACKERS: N/A Type Depth

13) TYPE PUMP: N/A
[ ] Turbine [ ] Jet [ ] Submersible [ ] Cylinder
[ ] Other _____
Depth to pump bowls, cylinder, jet, etc. _____ ft.

14) WELL TESTS: N/A
Type test: [ ] Pump [ ] Bailer [ ] Jetted [ ] Estimated
Yield: _____ gpm with _____ ft. drawdown after _____ hrs.

15) WATER QUALITY:
Did you knowingly penetrate any strata which contained undesirable constituents?
[ ] Yes [X] No If yes, submit "REPORT OF UNDESIRABLE WATER"
Type of water? _____ Depth of strata _____
Was a chemical analysis made? [ ] Yes [X] No

I hereby certify that this well was drilled by me (or under my supervision) and that each and all of the statements herein are true to the best of my knowledge and belief. I understand that failure to complete items 1 thru 15 will result in the log(s) being returned for completion and resubmittal.

COMPANY NAME JEDI (Type or print) WELL DRILLER'S LICENSE NO. 50205-m

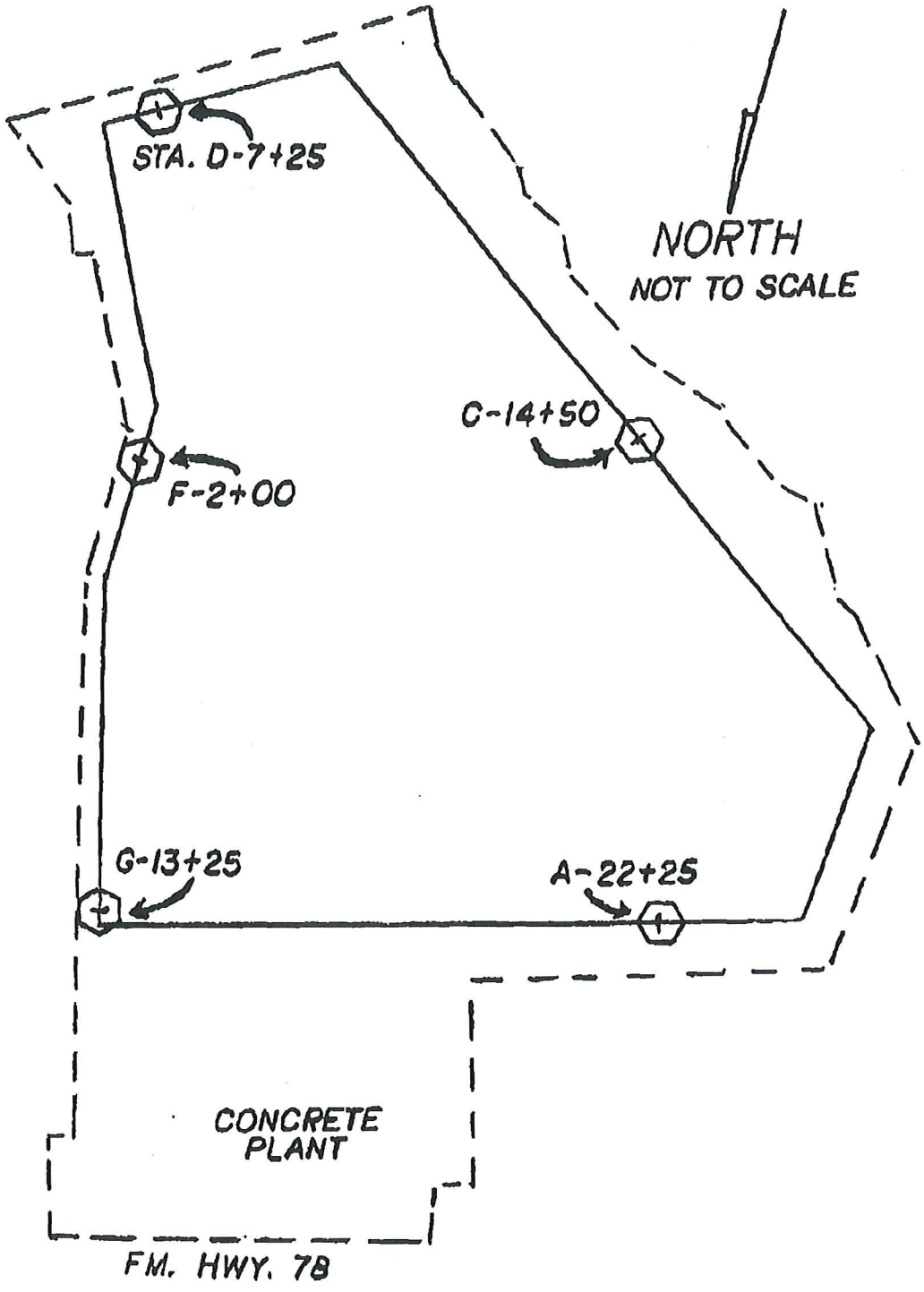
ADDRESS 5026 N. Main (Street or RFD) Cibolo (City) Tx (State) 78108 (Zip)

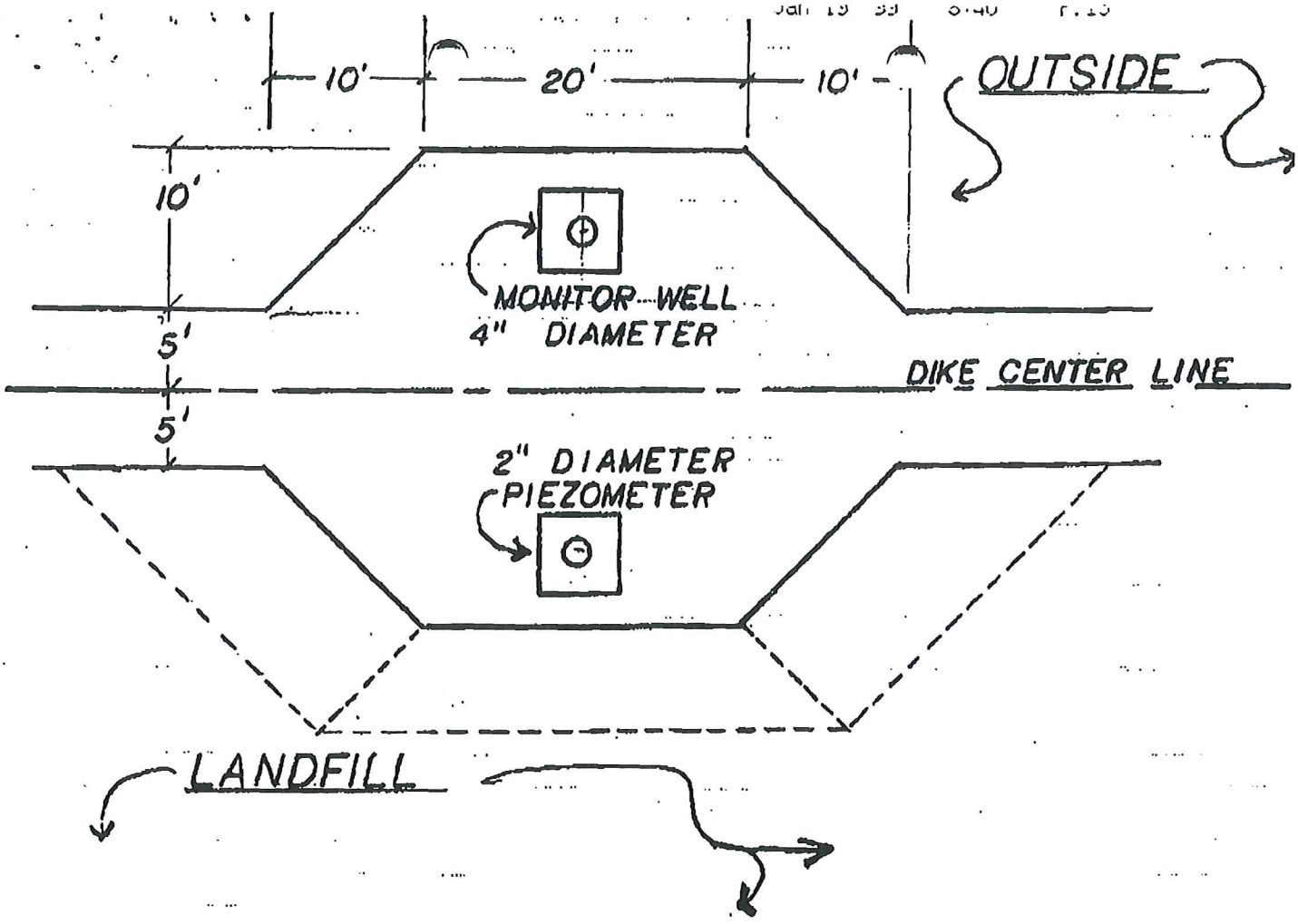
(Signed) [signature] (Licensed Well Driller) (Signed) _____ (Registered Driller Trainee)

Please attach electric log, chemical analysis, and other pertinent information, if available.

TNRCC-0199 (Rev. 05-21-96) White - TNRCC Yellow - DRILLER Pink - WELL OWNER

# MONITOR WELL AND PIEZOMETER LOCATIONS





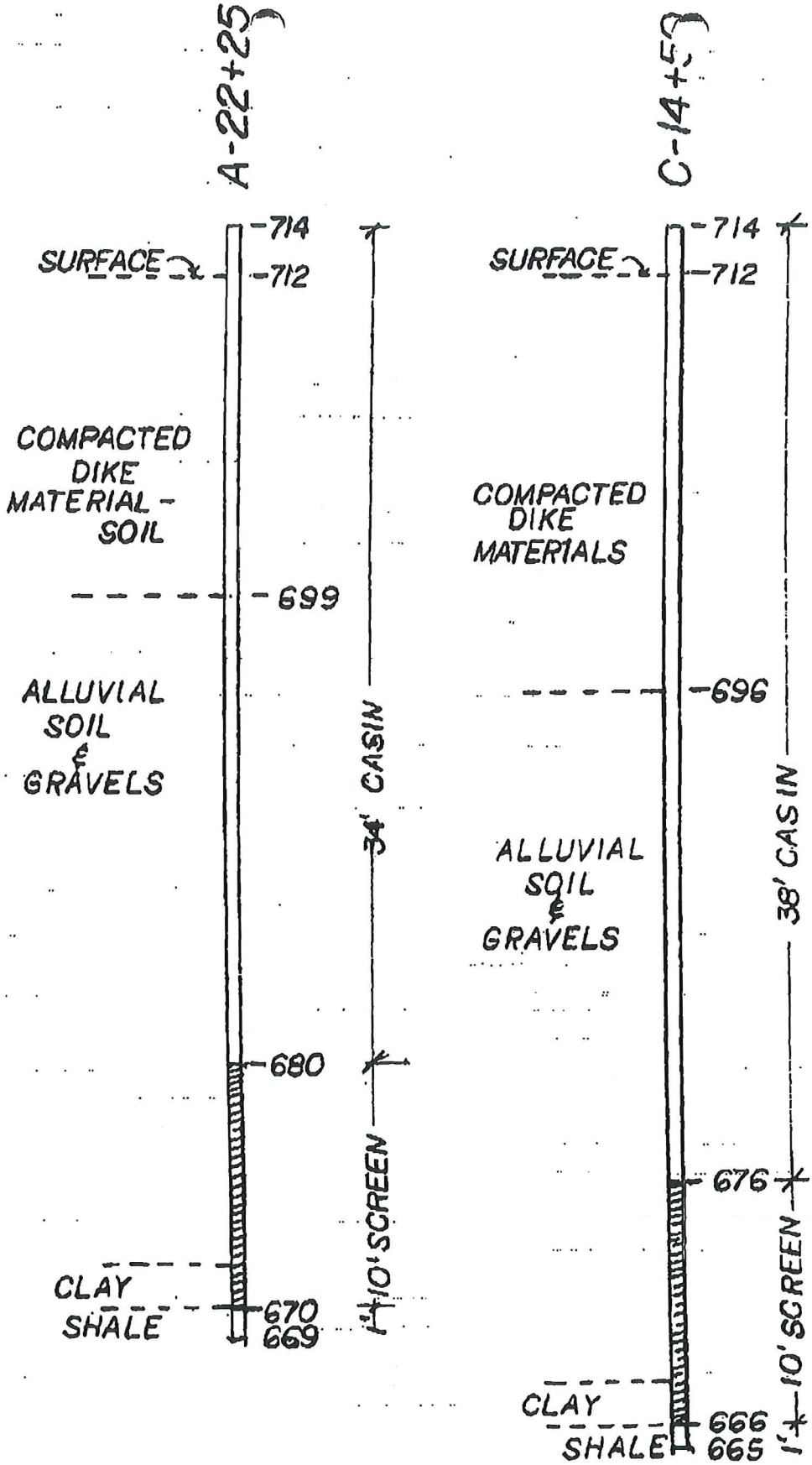

NOTE: LINE-STATION DESIGNATION SHALL BECOME IDENTIFICATION NUMBER

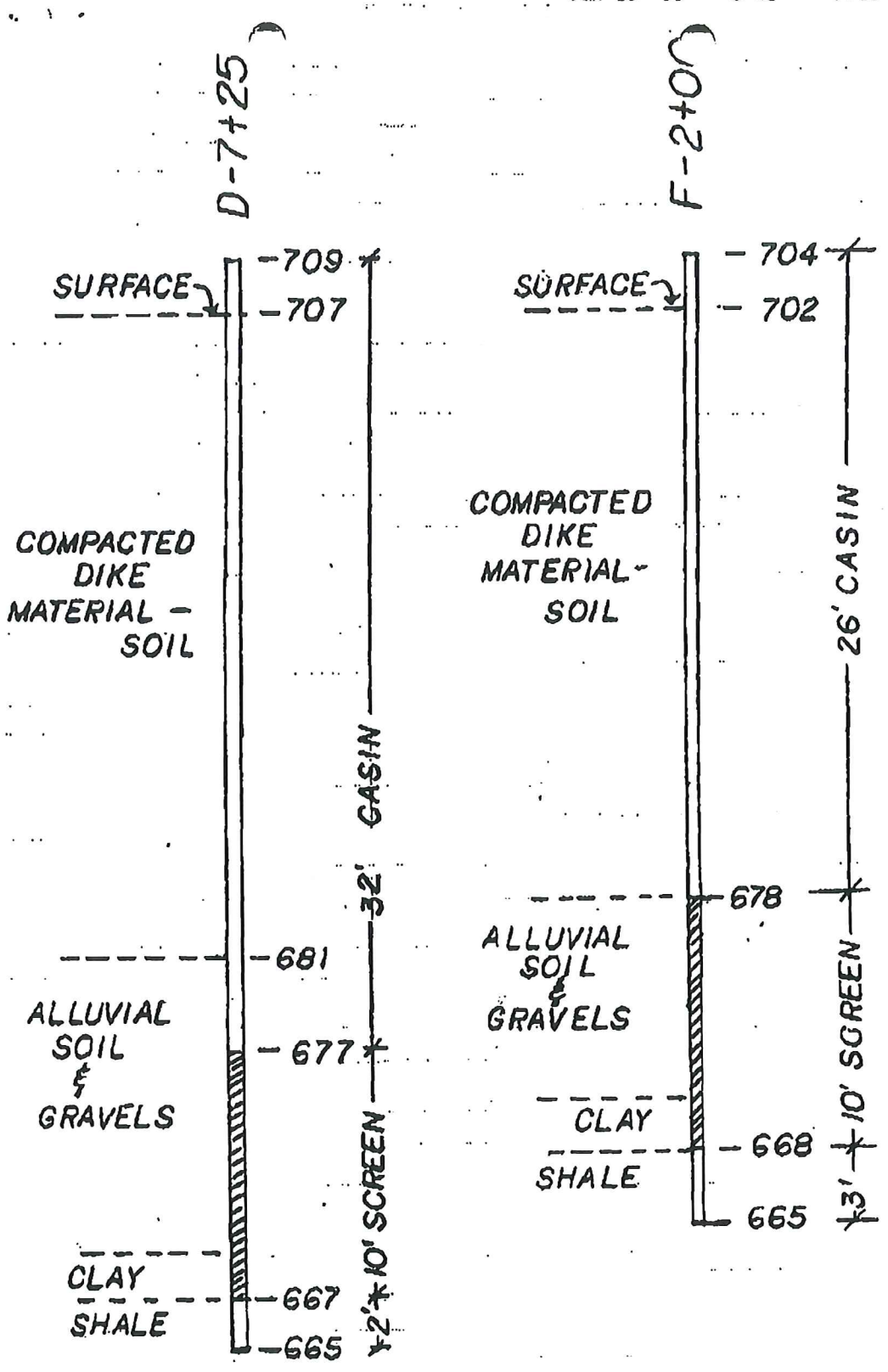
"W" SHALL INDICATE MONITOR WELL (X-0+00W)

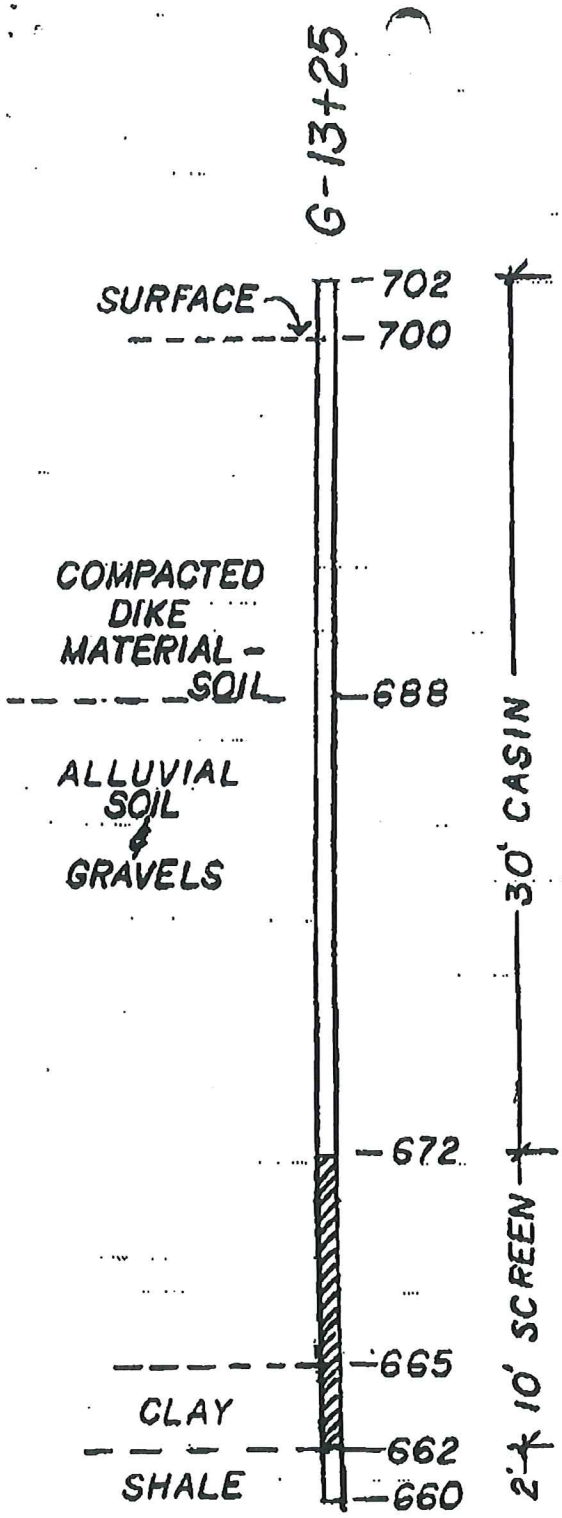
"P" SHALL INDICATE PIEZOMETER (X-0+00P)

# TYPICAL DETAIL:

## MONITOR WELL / PIEZOMETER DIKE EXTENSIONS


A-22+25
714
SURFACE
712
COMPACTED DIKE MATERIAL - SOIL
699
ALLUVIAL SOIL & GRAVELS
34' CASIN
680
10' SCREEN
CLAY SHALE
670
669
C-14+57
714
SURFACE
712
COMPACTED DIKE MATERIALS
696
ALLUVIAL SOIL & GRAVELS
38' CASIN
676
10' SCREEN
CLAY SHALE
666
665


D-7+25
709
SURFACE
707
COMPACTED DIKE MATERIAL - SOIL
681
ALLUVIAL SOIL & GRAVELS
677
CLAY
667
SHALE
665
32' CASIN
2'+10' SCREEN
F-2+00
704
SURFACE
702
COMPACTED DIKE MATERIAL - SOIL
678
ALLUVIAL SOIL & GRAVELS
CLAY
668
SHALE
665
26' CASIN
3'+10' SCREEN


G-13+25
SURFACE
702
700
COMPACTED DIKE MATERIAL - SOIL
688
ALLUVIAL SOIL & GRAVELS
30' CASIN
672
10' SCREEN
665
CLAY
662
SHALE
660
2'

# APPENDIX F-2 HISTORIC GROUNDWATER DATA

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848

| Date | Monitor Well | Cadmium (mg/L) | Iron (mg/L) | Manganese (mg/L) | Zinc (mg/L) | Chloride (mg/L) | Conductivity (umhos/cm) | pH (s.u.) | TDS (mg/L) | TOC (3) (mg/L) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 6/3/2005 | A | <5 | <0.05 | <0.005 | 20 | 20 | 1170 | 6.5 | 805 | <0.5 | <0.5 | <0.5 |
| | C | <5 | <0.05 | 0.197 | <20 | 18 | 1390 | 6.37 | 1000 | <0.5 | <0.5 | <0.5 |
| | D | <5 | <0.05 | 0.113 | <20 | 22 | 787 | 6.72 | 502 | <0.5 | <0.5 | <0.5 |
| | F | <5 | 0.09 | 0.06 | 34 | 35 | 3150 | 6.47 | 3010 | <0.5 | <0.5 | <0.5 |
| | G | <5 | <0.05 | <0.005 | <20 | 34 | 1650 | 6.59 | 1320 | <0.5 | <0.5 | <0.5 |
| 6/6/2006 | A | <5 | <0.05 | 0.014 | <20 | 32 | 1280 | 6.63 | 902 | 4.23 | 4.33 | 3.85 |
| | C | <5 | 0.17 | 1.35 | <20 | 50 | 1530 | 6.59 | 1050 | 4.07 | 3.69 | 3.78 |
| | D | <5 | 0.07 | 0.491 | 30 | 31 | 1080 | 6.72 | 786 | 2.61 | 2.55 | 2.33 |
| | F | <5 | 0.06 | 0.036 | 20 | 37 | 4780 | 6.58 | 3050 | 1.2 | 1.1 | 0.92 |
| | G | <5 | <0.05 | <0.005 | <20 | 42 | 1480 | 6.68 | 1120 | <0.5 | <0.5 | <0.5 |
| 6/13/2007 | A | <0.003 | <0.03 | 0.006 | <0.02 | 24 | 1240 | 6.7 | 911 | 30.1 | 28.4 | 28.9 |
| | C | <0.003 | <0.03 | 0.992 | 0.02 | 36 | 1480 | 6.56 | 1040 | 57.7 | 63.9 | 57.8 |
| | D | <0.003 | 0.07 | 0.828 | <0.02 | 32 | 1340 | 6.97 | 1030 | 21.5 | 19.6 | 20.2 |
| | F | <0.003 | <0.03 | 0.089 | 0.04 | 36 | 4400 | 6.48 | 2860 | 45.5 | 43.7 | 41 |
| | G | <0.003 | <0.03 | 0.014 | <0.02 | 32 | 2340 | 6.75 | 1430 | 32 | 30.2 | 28.2 |
| 12/12/2007 | A | | | | | | 870 | 6.33 | | 130 | 134 | 136 |
| | C | | | | | | 1440 | 6.18 | | 203 | 205 | 181 |
| | D | | | | | | 1400 | 6.5 | | 45.2 | 46.1 | 41 |
| | F | | | | | | 2890 | 6.26 | | 74.9 | | 80.6 |
| | G | | | | | | N/A | N/A | | N/A | N/A | N/A |

4/30/2008 VOC Sampling Only
All results below detection

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848

| Date | Monitor Well | Cadmium (mg/L) | Iron (mg/L) | Manganese (mg/L) | Zinc (mg/L) | Chloride (mg/L) | Conductivity (umhos/cm) | pH (s.u.) | TDS (mg/L) | TOC (3) (mg/L) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 6/12/2009 | A | <0.0017 | <0.0130 | 0.363 | <0.0080 | 63.3 | 1590 | 6.8 | 1010 | 24.9 | 24.6 | 24.8 |
| | C | <0.0017 | 0.29 | 1.41 | <0.0080 | 40.1 | 1500 | 6.9 | 1000 | 15 | 14.1 | 13.7 |
| | D | <0.0017 | <0.0130 | 0.337 | <0.0080 | 21.7 | 1020 | 7.2 | 760 | 2.48 | 2.21 | 2.24 |
| | F | <0.0017 | <0.0130 | 0.058 | 0.04 | 39.1 | 3920 | 7.2 | 3110 | 2.95 | 2.49 | 2.42 |
| | G | <0.0017 | 0.09 | 0.594 | <0.0080 | 13.4 | 2500 | 6.9 | 1570 | 0.3 | 0.134 | 0.117 |
| 4/14/2010 | A | <0.0003 | <0.03 | <0.005 | <0.02 | | 1440 | 6.82 | 948 | 0.98 | 0.97 | 0.87 |
| | C | <0.0003 | 0.09 | 1.97 | <0.02 | | 1520 | 6.52 | 1000 | 11.9 | 11.3 | 11.1 |
| | D | <0.0003 | 0.97 | 0.767 | <0.02 | | 1780 | 6.86 | 1170 | 1.22 | 1.19 | 1.08 |
| | F | <0.0003 | 0.03 | 0.056 | <0.02 | | 3580 | 6.44 | 2360 | 2.09 | 1.96 | 2.03 |
| | G | <0.0003 | 0.19 | 5.42 | <0.02 | | 2960 | 6.1 | 1950 | 4.76 | 4.62 | 4.39 |
| 8/24/2011 | A | <0.0003 | 0.45 | 0.14 | <0.02 | | 1460 | 7.49 | 1030 | 4.32 | 4.18 | 4.3 |
| | C | <0.0003 | 53.2 | 3.91 | <0.02 | | 1540 | 7.07 | 1080 | 42.3 | 41.9 | 42.2 |
| | D | 0.005 | 2.6 | 1.09 | 0.02 | | 1340 | 7.48 | 936 | 7.42 | 7.28 | 7.36 |
| | F | 0.0003 | 0.16 | 0.06 | 0.07 | | 2800 | 7.3 | 1960 | 4.16 | 4.2 | 4.22 |
| | G | <0.0003 | 23 | 5.84 | <0.02 | | 2010 | 6.92 | 1400 | 4.2 | 4.24 | 4.21 |
| 7/18/2012 | A | <0.003 | 1.25 | 0.79 | 0.053 | 33.9 | 1570 | 6.5 | 960 | 7.8 | 7.96 | 8.04 |
| | C | 0.005 | 37 | 2.01 | 0.118 | 41.6 | 1560 | 6.57 | 960 | 17.1 | 17.5 | 17.6 |
| | D | <0.003 | 0.269 | 0.46 | 0.051 | 26.3 | 1690 | 7.05 | 1200 | 3.84 | 3.89 | 3.87 |
| | F | <0.003 | 0.112 | 0.08 | 0.079 | 25.8 | 2970 | 6.86 | 2040 | 2.26 | 2.3 | 2.32 |
| | G | 0.003 | 27.4 | 4.29 | 0.061 | 9.52 | 1640 | 6.43 | 1000 | 3.59 | 3.72 | 3.76 |

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848

| Date | Monitor Well | Cadmium (mg/L) | Iron (mg/L) | Manganese (mg/L) | Zinc (mg/L) | Chloride (mg/L) | Conductivity (umhos/cm) | pH (s.u.) | TDS (mg/L) | TOC (3) (mg/L) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 7/13/2013 | A | <0.005 | <0.050 | 0.912 | <0.010 | 76.7 | 1790 | 6.31 | 1110 | 29.8 | 30.2 | 30.4 |
| | C | <0.005 | 6.69 | 2.46 | <0.010 | 29.5 | 1410 | 6.49 | 888 | 8.3 | 8.4 | 8.7 |
| | D | <0.005 | <0.050 | 0.725 | <0.010 | 27.1 | 1680 | 6.77 | 1220 | 3.2 | 3.24 | 3.24 |
| | F | <0.005 | <0.050 | 0.049 | 0.022 | 24.8 | 2900 | 6.45 | 1890 | 2.22 | 2.25 | 2.65 |
| | G | <0.005 | 14.6 | 3.32 | <0.010 | 10.5 | 1700 | 6.25 | 1050 | 3.38 | 3.4 | 3.39 |
| 7/9/2014 | A | <0.005 | 0.184 | 2.45 | 0.051 | 96.8 | 1950 | 6.48 | 1200 | 1.3 | 1.31 | 1.35 |
| | C | <0.005 | 4.45 | 2.11 | 0.043 | 26.6 | 1470 | 6.66 | 891 | 1.34 | 1.35 | 1.36 |
| | D | <0.005 | 0.121 | 0.503 | 0.03 | 20.8 | 1450 | 6.91 | 1010 | 2.38 | 2.36 | 2.37 |
| | F | <0.005 | 0.092 | 0.075 | 0.022 | 25.1 | 3370 | 6.53 | 2860 | 3.07 | 3.12 | 3.08 |
| | G | <0.005 | 14.0 | 1.99 | <0.010 | 17.1 | 2840 | 6.41 | 2150 | <0.10 | <0.10 | <0.10 |
| 6/8/2015 | A | <0.005 | <0.050 | 3 | 0.025 | 48.4 | 1680 | 6.52 | 1260 | 19.8 | 19 | 18.3 |
| | C | <0.005 | <0.050 | 1.18 | 0.019 | 20.8 | 1480 | 6.48 | 1150 | 8.1 | 7.94 | 8.02 |
| | D | <0.005 | <0.050 | 0.128 | 0.024 | 12.1 | 1030 | 6.5 | 823 | 1.63 | 1.59 | 2.4 |
| | D (Duplicate) | <0.005 | <0.050 | 0.097 | 0.028 | 14.2 | 1150 | 6.81 | 875 | 1.74 | 1.7 | 2.44 |
| | F | <0.005 | <0.050 | 0.024 | 0.029 | 17.2 | 2110 | 6.51 | 1830 | 1.62 | 1.66 | 2.04 |
| | G | <0.005 | 1.74 | 3.11 | 0.012 | 12.0 | 1440 | 6.37 | 1040 | 3.04 | 3.03 | 3.23 |
| 5/26/2016 | A | <0.005 | <0.050 | 0.581 | <0.010 | 28.0 | 1570 | 6.64 | 1210 | 1.10 | 1.09 | 1.03 |
| | C | <0.005 | 59.1 | 1.92 | 0.270 | 36.9 | 1550 | 6.65 | 1120 | 8.43 | 8.60 | 8.71 |
| | C- Duplicate | <0.005 | 20.9 | 0.813 | 0.088 | 37.0 | 1560 | 6.74 | 1160 | 8.84 | 8.74 | 8.83 |
| | D | <0.005 | 0.083 | 0.151 | <0.010 | 16.2 | 840 | 7.04 | 567 | 1.21 | 1.20 | 0.86 |
| | F | <0.005 | 0.063 | 0.052 | 0.012 | 21.2 | 2150 | 6.68 | 1780 | 2.03 | 2.06 | 1.37 |
| | G | <0.005 | 22.2 | 2.55 | 0.012 | 15.5 | 1500 | 6.57 | 1020 | 3.81 | 3.89 | 3.90 |
| 5/24/2017 | A | <0.005 | 0.065 | 0.605 | <0.010 | 14.0 | 1540 | 6.37 | 1080 | 1.14 | 1.14 | 1.14 |
| | A- Duplicate | <0.005 | <0.050 | 0.655 | 0.013 | 14.2 | 1560 | 6.48 | 1090 | 1.17 | 1.17 | 1.20 |
| | C | <0.005 | 24.5 | 2.01 | 0.048 | 49.6 | 1730 | 6.45 | 1060 | 44.5 | 40.6 | 41.5 |
| | D | <0.005 | 0.575 | 0.215 | 0.013 | 20.7 | 1250 | 6.73 | 847 | 1.59 | 1.58 | 1.59 |
| | F | <0.005 | 0.163 | 0.101 | 0.018 | 30.4 | 3350 | 6.45 | 2940 | 2.31 | 2.30 | 2.26 |
| | G | <0.005 | 24.2 | 2.36 | 0.017 | 14.0 | 1480 | 6.34 | 932 | 4.03 | 4.05 | 4.07 |

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848

| Date | Monitor Well | Cadmium (mg/L) | Iron (mg/L) | Manganese (mg/L) | Zinc (mg/L) | Chloride (mg/L) | Conductivity (umhos/cm) | pH (s.u.) | TDS (mg/L) | TOC (3) (mg/L) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 6/13/2018 | A | <0.005 | 1.15 | 1.14 | 0.019 | 67.5 | 2080 | 6.31 | 1290 | 24.9 | 25.7 | 25.5 |
| | A- Duplicate | <0.005 | 0.965 | 1.06 | 0.014 | 66.1 | 2080 | 6.4 | 1280 | 25.0 | 23.9 | 23.6 |
| | C | <0.005 | 42.0 | 2.96 | 0.053 | 51.4 | 1830 | 6.47 | 918 | 51.2 | 49.6 | 49.3 |
| | D | <0.005 | 0.545 | 0.197 | 0.029 | 16.2 | 1170 | 6.86 | 765 | 1.93 | 1.90 | 1.93 |
| | F | <0.005 | 0.089 | 0.068 | 0.024 | 25.5 | 3570 | 6.51 | 2940 | 2.52 | 2.53 | 2.54 |
| | G | <0.005 | 38.0 | 2.64 | 0.016 | 65.7 | 1980 | 6.31 | 1200 | 40.5 | 40.6 | 40.5 |
| 10/2/2018 | A | N/A | N/A | N/A | N/A | N/A | N/A | N/A | N/A | 54.4 | 54.8 | 54.7 |
| TOC only | A- Duplicate | N/A | N/A | N/A | N/A | N/A | N/A | N/A | N/A | 54.3 | 54.4 | 54.7 |
| | C | N/A | N/A | N/A | N/A | N/A | N/A | N/A | N/A | 7.3 | 7.2 | 7.1 |
| | G | N/A | N/A | N/A | N/A | N/A | N/A | N/A | N/A | 53.1 | 52.7 | 52.6 |
| 5/22/2019 | A | <0.005 | 1.43 | 2.02 | 0.020 | 38.1 | 1720 | 6.29 | 1110 | 12.4 | 12.8 | 12.9 |
| | A- Duplicate | <0.005 | 0.615 | 1.92 | 0.019 | 45.8 | 1700 | 6.39 | 1160 | 13.8 | 14.0 | 14.1 |
| | C | <0.005 | 7.56 | 1.31 | 0.014 | 38.9 | 1610 | 6.59 | 1080 | 21.8 | 21.5 | 21.2 |
| | D | <0.005 | 0.069 | 0.066 | 0.011 | 17.1 | 887 | 6.92 | 594 | 1.43 | 1.42 | 1.37 |
| | F | <0.005 | 0.064 | 0.056 | 0.023 | 18.2 | 2440 | 6.58 | 2090 | 1.98 | 1.99 | 2.00 |
| | G | <0.005 | 25.0 | 2.18 | 0.021 | 75.3 | 1780 | 6.33 | 1120 | 41.9 | 41.6 | 41.5 |
| 6/18/2020 | A | <0.005 | 0.964 | 1.73 | 0.019 | 36.9 | 1740 | 6.40 | 1330 | 9.44 | 9.59 | 9.46 |
| | A- Duplicate | <0.005 | 0.848 | 1.63 | <0.010 | 36.2 | 1770 | 6.40 | 1270 | 9.04 | 8.99 | 9.24 |
| | C | <0.005 | 25.0 | 1.62 | 0.027 | 47.8 | 1620 | 6.56 | 1120 | 18.9 | 19.9 | 20.0 |
| | D | <0.005 | 0.132 | 0.094 | <0.010 | 13.6 | 935 | 6.94 | 513 | 1.26 | 1.15 | 1.12 |
| | F | <0.005 | 0.188 | 0.052 | 0.018 | 20.4 | 2870 | 6.65 | 2750 | 2.08 | 2.17 | 2.16 |
| | G | <0.005 | 25.2 | 2.04 | <0.010 | 84.4 | 1900 | 6.33 | 1300 | 4.33 | 4.46 | 4.51 |
| 6/16/2021 | A | <0.005 | 0.608 | 1.54 | <0.010 | 72.2 | 1830 | 6.17 | 1300 | 20.6 | 20.0 | 20.0 |
| | C | <0.005 | 17.3 | 1.16 | 0.039 | 38.1 | 1620 | 6.54 | 1140 | 11.4 | 10.5 | 11.1 |
| | D | <0.005 | 0.120 | 0.237 | <0.010 | 11.5 | 765 | 6.94 | 555 | 2.51 | 2.55 | 2.51 |
| | D - Duplicate | <0.005 | 0.114 | 0.210 | 0.012 | 9.30 | 601 | 7.10 | 395 | 2.06 | 1.72 | 1.58 |
| | F | <0.005 | 0.182 | 0.080 | 0.013 | 24.4 | 3000 | 6.48 | 2720 | 6.22 | 4.88 | 5.20 |
| | G | <0.005 | 23.1 | 1.89 | <0.010 | 107 | 2090 | 6.24 | 1450 | 68.6 | 68.1 | 68.2 |

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848

| Date | Monitor Well | Cadmium (mg/L) | Iron (mg/L) | Manganese (mg/L) | Zinc (mg/L) | Chloride (mg/L) | Conductivity (umhos/cm) | pH (s.u.) | TDS (mg/L) | TOC (3) (mg/L) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 7/1/2022 | A | <0.005 | 0.458 | 1.75 | 0.048 | 42.1 | 1910 | 6.42 | 1340 | 11.9 | 12.6 | 11.9 |
| | A - Duplicate | <0.005 | 0.564 | 1.57 | 0.062 | 40.4 | 1890 | 6.46 | 1330 | 11.5 | 10.0 | 11.0 |
| | C | <0.005 | 17.8 | 1.29 | 0.046 | 46.1 | 1700 | 6.44 | 977 | 33.2 | 31.8 | 30.0 |
| | D | <0.005 | 0.110 | 0.051 | 0.054 | 11.9 | 837 | 6.76 | 465 | 1.26 | 1.27 | 1.31 |
| | F | <0.005 | 0.104 | <0.010 | 0.05 | 24.5 | 4370 | 6.92 | 2920 | 2.86 | 2.97 | 2.91 |
| | G | <0.005 | 24.2 | 2.22 | 0.053 | 128 | 2230 | 6.36 | 1500 | 92.2 | 83.5 | 87.4 |
| 6/14/2023 | A | <0.005 | 0.895 | 1.73 | 0.018 | 86.1 | 1940 | 7.12 | 1230 | 40.3 | 41.4 | 41.0 |
| | C | <0.005 | 37.3 | 1.11 | 0.067 | 31.9 | 1450 | 7.29 | 941 | 14.3 | 13.4 | 13.1 |
| | D | <0.005 | 0.195 | 0.138 | 0.021 | 9.22 | 774 | 8.02 | 563 | 1.44 | 1.42 | 1.35 |
| | D - Duplicate | <0.005 | 0.053 | 0.048 | 0.013 | 8.38 | 732 | 7.77 | 437 | 1.18 | 1.20 | 1.22 |
| | F | <0.005 | 0.134 | 0.054 | 0.031 | 22.8 | 3180 | 7.60 | 2850 | 2.49 | 2.48 | 2.49 |
| | G | <0.005 | 22.5 | 1.57 | 0.016 | 132 | 2290 | 6.55 | 1460 | 105 | 97.0 | 97.3 |

# APPENDIX F-3 GROUNDWATER SAMPLING AND ANALYSIS PLAN

# MUNICIPAL SOLID WASTE PERMIT
# MAJOR AMENDMENT

## Groundwater Sampling and Analysis Plan

## (TAC Title 30 Rule §330.63(f))

***NAME OF PROJECT: Beck Landfill***

***MSW PERMIT APPLICATION NO.: 1848A***

***OWNER: Nido, LTD (CN603075011)***

***OPERATOR: Beck Landfill (RN102310968)***

***CITY, COUNTY: Schertz, Guadalupe County***

*Major Amendment: September 2022*

Prepared by:

*PROJECT NUMBER: 150051.05.01*
*PROJECT CONTACT: Julie Morelli*
*EMAIL: Julie.Morelli@powereng.com*
PHONE: 210-951-6424

# Groundwater Sampling and Analysis Plan

## OVERVIEW

The following Groundwater Sampling and Analysis Plan (GWSAP) is prepared for the Beck Landfill, Nido, LTD. Type IV Landfill (Beck Landfill), MSW Permit No. 1848A, located in Schertz,, Guadalupe County, Texas in accordance with the regulations in 30 TAC §330.417 (relating to Groundwater Monitoring at Type IV Landfills).

This GWSAP is included as Attachment F, Appendix F-3 of Part III of the Beck Landfill permit application submitted in September 2022. It is intended to provide a consistent sampling and analysis procedure and is designed to ensure that ground-water data accurately represents actual groundwater quality and can be used to reliably evaluate the groundwater conditions at this site.

# Table of Contents

PROCEDURES: ..... 4
I Timing and Order of Purging or Sampling ..... 4
II Well Inspection ..... 4
III Water-Level Measurements ..... 5
IV Well Purging ..... 5
V Sample Collection and Preservation ..... 6
V.1 Sample Collection and Preparation ..... 6
V.2 Field Measurements ..... 7
V.3 Sample Containers ..... 7
V.4 Sample Containers, Preservation and Holding Times ..... 8
V.5 QC Samples (Trip Blanks, Field Blanks, Replicates) ..... 9
V.6 Sample Storage and Transport ..... 9
V.7 Chain-of-Custody Documentation ..... 9
V.8 Documentation of Sampling ..... 10
VI Sample Filtration ..... 10
VII Analytical Parameters ..... 11
VIII Analytical Methods ..... 11
IX Background Samples – Not Revised during January 2008 Updates ..... 12
X Detection Monitoring ..... 13
XI Corrective Action ..... 14
XII Quality Assurance and Quality Control (QA/QC) ..... 14
XIII Reporting and Submittals ..... 15
XIV Safety Plan ..... 16
Attachment 1 – Field Log Data Sheets for Purging and for Sampling
Attachment 2 – Chain of Custody Form for San Antonio Testing Lab
Attachment 3 – San Antonio Testing Laboratories, Ltd.
Quality Assurance Plan (QAP) Standard
Operating Procedures (SOPs)

Beck Landfill, Nido, LTD. has developed the following Groundwater Sampling and Analysis Plan (GWSAP) for the Guadalupe County Landfill in Schertz, MSW Permit No. 1848, in accordance with the regulations in 30 TAC §330.417 (relating to Groundwater Monitoring at Type IV Landfills). This GWSAP is submitted as a modification to the Site Operating Plan and is intended to provide a consistent sampling and analysis procedure. It is designed to ensure that ground-water data accurately represents actual groundwater quality and can be used to reliably evaluate the groundwater conditions at this site.

PROCEDURES:

*I Timing and Order of Purging or Sampling*

The elapsed time between well purging and sample collection should be as short as possible to avoid temporal variations in water levels and water chemistry. Sampling should be done preferably within 24 hours of purging. If a well is very slow to recharge, it should be sampled as soon as practicable; a maximum of seven days may be acceptable with prior TCEQ approval.

The wells will be sampled from the up-gradient well to the down-gradient well, sequentially beginning with the well on Line A and proceeding as follows: Line A to Line C to Line D to Line F to Line G. See gradient map attached directly behind this page.

If contamination is known to be present, sampling should proceed from the monitoring well least or not contaminated to the well with the most contamination.

*II Well Inspection*

Inspect the integrity of the monitoring well prior to commencement of purging and/or sampling the well. The inspection of the well should be documented on a Field Log Data Sheet.

- Check the casing and concrete pad for cracks or fissures. Be sure that vandalism, animals, heavy equipment, etc have not damaged the well.
- Check that the cap is locked.
- Check that the well plug cap is tightened to prevent surface runoff infiltration into the well.
- Note the proximity of the well to potential sources of contamination on a Field Log Data Sheet.
- If insects are found in or on the well casing, do NOT use organic sprays or other potential contaminants to remove them.
- Similarly, organic lubricants should not be used on well components such as locks.

## III *Water-Level Measurements*

Prior to purging or sampling of a well, measure the depth to water to determine water level and to be sure that enough water is present for sampling. Follow these steps for proper measurements.

- Decontaminate the measurement probe prior to use in each well by washing with a phosphate-free soap and rinsing with reagent grade water, obtained from the laboratory, or commercially distilled water.
- Calibrate measurement probes regularly to determine the stretch of suspended measuring tapes, wires, or cables.
- Measure from the top of the well casing, identified on the Monitor Well Data Sheets, for each well. Record the depth to water to the nearest hundredth of a foot.
- Calculate the elevation of the water level with respect to mean sea level (msl) and record it to the nearest hundredth of a foot.

## IV *Well Purging*

- Wells should be purged of stagnant water with a bailer (or a pump) 24 hours prior to sampling to obtain a chemically representative ground water sample from each well.
- To assure comparability of the ground-water samples collected from the site, the same type of purging equipment should generally be used in each of the site wells.
- Each well will be purged with a disposable bailer or using a submersible pump and disposable tubing, so that the well does not become contaminated during sampling.
- Bailers should be bottom-emptying devices, so that the bailer can be emptied slowly, with minimum aeration.
- Care should be taken during purging to avoid introducing contaminants to the water in the well. Use disposable, plastic or vinyl gloves, changed between each well, to avoid cross-contamination. Latex gloves can cause contamination.
- Purging should be performed in such a way as to minimize the stirring of sediments with the waters in the well. Lower the bailer (or pump) gently. Do NOT drop the bailer (or pump) to the bottom of the screen in the well. Pull the bailer (or pump) to the surface slowly. (If a pump is used, pump intakes should not be set too close to the bottom of the well.)
- If possible, purge at least three times the total volume of water determined to be in the well casing from the measurements made in Section II.

Example: Volume = pi * r2 * h

Where -
pi = 3.14159265
r = radius of the casing
h = height of the water column in the well

V = pi * (.17')2 * (4') = .36 cu. ft.

Conversion to gallons (7.48052 gallons per cubic foot)
0.36 cu. ft * 7.48052 = 2.7 gallons Volume *
3 = 8.1 gallons

Note: The casing volume is the amount of water in the casing itself prior to purging and does not include the volume of water in the filter pack.

These wells recharge very slowly. If insufficient water is available to be removed from the well, purging to dryness is sufficient to remove stagnant water.

Allow the well to recover enough to allow collection of samples. Where possible, the water level should be allowed to recover to within 90% of the water level established prior to purging.

Record the following data collected on a Field Purging Log Data Sheet (See Attachment 1):

- The initial depth to water (DTW),
- measured well depth (total depth (TD)),
- height of the water column,
- well purging time,
- volume of water purged from the well,
- purging discharge rate, and
- information from the well inspection.

Purged water should be containerized and disposed of through the local POTW, with written permission.

## V *Sample Collection and Preservation*

Sample collection, preservation and shipment to the laboratory are important steps in the sampling process. Physical or chemical changes occur in ground-water samples no matter how carefully sampling is done. Inappropriate sampling devices, collection procedures, preservatives and temperature controls, or inadequate shipment can damage sample quality, giving inaccurate results.

### V.1 Sample Collection and Preparation

The need to minimize turbulence and aeration of the sample can not be overemphasized.

- Fill sample containers directly from the bailer (or pump tubing) when possible. Transfer containers are not recommended for sample collection because of the likelihood of cross-contamination.

- Do not reuse soiled sample containers, bailers and bailer rope, disposable tubing, or plastic (or vinyl) gloves.
- Where possible, keep clean equipment off the ground to prevent contamination once the equipment is cleaned.
- Handle water removed during sampling and not saved in the same way as purged water.
- Do not allow the sampling device to touch the sampling container, but hold the two as close as possible to reduce aeration.
- Check the area around the sampling point for possible sources of air contamination.

## V.2 Field Measurements

- The equipment used for field measurements should be calibrated at least daily during sampling.
- Slowly pour an unfiltered portion into a clean container for field measurement of temperature, specific conductance, and pH.
- Measure and record the temperature immediately.
- Measure and record the specific conductance of the sample to avoid any effect on the sample from salts from the pH probe.
- Measure and record the pH.
- Record the color, odor, foaming, presence of more than one phase of liquid, and turbidity of the sample.

## V.3 Sample Containers

The volume of samples and types of sample containers needed are described in Table 1 below. Volumes and containers have been selected in accordance with methods specified in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" (United States Environmental Protection Agency (EPA) Publication Number SW-846). To avoid confusion, the number of containers collected from each well will be minimized.

Label all sample containers with indelible ink for identification purposes. Alternatively, cover the sample label with clear packing tape and place the sample container inside a ziplock bag before placing on ice. The label information should include:

- sample number,
- well number,
- site identification,
- analysis to be performed,
- preservatives used,
- date and time of sample collection, and
- name of sampler.

Fill the sample containers in the following order:

1) Non-Purgeable Organics (NPOC)
2) Metals
3) Other Inorganic Parameters

Fill replicate sample containers for NPOC from a single bailer to improve homogeneity in the samples.

## V.4 Sample Containers, Preservation and Holding Times

Holding times and sample volumes required for each analysis have been reviewed with the laboratory. Sample preservation is intended to 1) retard biological action, 2) retard hydrolysis, and 3) reduce sorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and protection from light. Specific preservation methods presented in Table 1, below, are in accordance with the EPA requirements of SW-846, "Test Methods for Evaluating Solid Waste", 3rd Edition as revised and updated or Standard Methods for the Examination of Water and Wastewater, 21st Edition as revised and updated.

**Table 1 Annual Detection Monitoring Sample Containers, Preservation & Holding Time**

| Parameter | Sample Container | Preservative | Replicates | Holding Time |
|---|---|---|---|---|
| pH | 1 Liter Glass Bottle | Ice | No | Analyze Immediately |
| Specific Conductance | 1 Liter Plastic Bottle | Ice | No | 28 days |
| Non-Purgeable Organics (TOC) | 100 mL Amber VOA | Ice, HCL or H2SO4 | Three | 2 hours (28 days if acidified) |
| Total Dissolved Solids | 1 Liter Plastic Bottle | Ice | No | 7 days |
| Chloride | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| Iron (dissolved) | 1 Liter Plastic Bottle | Ice, (HNO3 if filtered) | No | 6 Months |
| Manganese (dissolved) | 1 Liter Plastic Bottle | Ice, (HNO3 if filtered) | No | 6 Months |
| Cadmium (dissolved) | 1 Liter Plastic Bottle | Ice, (HNO3 if filtered) | No | 6 Months |
| Zinc (dissolved) | 1 Liter Plastic Bottle | Ice, (HNO3 if filtered) | No | 6 Months |

Note: See Table 4 at the end of this report for Background Parameters

## V.5 QC Samples (Trip Blanks, Field Blanks, Replicates)

- One field blank will be used during each sampling event to identify possible sources of air pollutant contamination originating at the onsite ready mix plant.
- Three Replicate samples will be collected during each sampling event for analysis of Non-Purgeable Organic Compounds.
- One sample duplicate will be collected for analysis of Volatile Organic Compounds during Background Sampling.

## V.6 Sample Storage and Transport

- All samples should be kept cold, ideally at 4°C, and transported to the laboratory within 2 days of sampling.
- Samples should be kept in re-sealable bags, then in an ice chest and packed with sufficient ice or re-freezeable materials to keep then as near 4°C as possible. DON'T USE DRY ICE TO CHILL THE SAMPLES BECAUSE THE SAMPLES WILL FREEZE AND THE CONTAINERS
- WILL BREAK.
- If the samples are shipped, they and the insulated container should first be chilled with ice. Pour off the ice and water, and keep cold during shipment with frozen packages of re-freezeable materials such as "blue ice."
- The insulated container needs to be packed inside with foam, newspaper, or an absorbent material such as vermiculite to prevent or minimize the likelihood of container breakage, then thoroughly sealed with cloth tape or reinforced shipping tape.
- Inexpensive foam chests are NOT suitable for shipping.
- Under NO circumstances, should water, ice, or dry ice be used for samples shipped via public transportation (i.e. the bus).

## V.7 Chain-of-Custody Documentation

- A suitable chain-of-custody (COC) document must accompany the samples at every step from field to laboratory and must be signed by each party handling the samples, from sampler through transporter to the laboratory, to document the possession of the samples at all times. Proper COC procedures are essential to ensure sample integrity and to provide legally and technically defensible data.
- The person collecting the sample starts the COC procedure.
- Individuals relinquishing and receiving the samples sign, date, and note the time of the transfer on the COC form (see attachment 2).
- Packages sent by mail should be certified with return receipt requested to document shipment.
- For packages sent by common carrier, a copy of the bill of lading will suffice.

- Copies of the return receipt or bill of lading should be attached to the COC document.
- The COC document must accompany the sample during transport and shipping, and should be protected from moisture using sealable plastic bags.

### V.8 Documentation of Sampling

- Information related to a sampling event should be recorded in a bound, permanent field log book or on Field Sampling Log Data Sheets (see Attachment 1).
- All entries should be legible and made in indelible ink.
- Entry errors should be crossed out with a single line, dated, and initialed by the person making corrections.
- Record sufficient information so that the sampling situation can be reconstructed without relying on the sampler's memory.
- Location, date, time, weather conditions, name and identity of sampling personnel, all field measurements, including numerical values and units, comments about the integrity of the well, etc., should be recorded.
- These records may be the only acceptable record for legal purposes. Protect it and keep it in a safe place.

## *VI Sample Filtration*

As stated in §330.405(c), samples shall **not** be field filtered prior to laboratory analysis. Laboratory filtering of samples for metals analysis is permitted if necessary to protect analytical equipment. Because of chemical or physical changes that may occur during shipping or transport, the interpretation of “total” metals is questionable if the samples are filtered in the laboratory. Dissolved metals are better indicators than "total" metals, and owners and operators are encouraged to analyze samples for both "total" and dissolved metals, especially for sites that have large amounts of suspended sediments in the samples. If dissolved metals are to be analyzed, the samples should be properly filtered in the field. If field filtering is not practical, the samples should be filtered in the lab as soon as possible. Samples to be analyzed for inorganic parameters other than metals may also be filtered for the sake of consistency. A note indicating whether or not the samples were filtered and the place where they were filtered must accompany the results of the ground-water analyses.

- The metals (Fe, Mn, Cd, and Zn) to be analyzed as total metals at this site will not be filtered in the field.
- Neither field nor lab filtering is permitted for samples that are to be analyzed for NPOC. Many organic compounds are attached to solid particles, and filtering would remove them, yielding false, negative results.

## VII *Analytical Parameters*

Ground-water sampling and analysis requirements shall be in accordance with §330.417 of this title (relating to Ground-Water Monitoring at Type IV Landfills).

The following constituents will be tested for: chloride, iron (total), manganese (total), cadmium (total), zinc (total), total dissolved solids, specific conductance (field and laboratory measurements), pH (field and laboratory measurements), and non-purgeable organic compounds (analysis of three replicate samples).

Not later than 60 days after each sampling event, the owner or operator shall submit to the Executive Director for review and approval a report containing the results of the analyses. If the facility is found to have contaminated or be contaminating the shallow water-bearing zones, the Executive Director may order corrective action appropriate to protect human health and the environment up to and including that in §§330.411, 330.412, and 333.415 of this title (relating to Assessment of Corrective Measures; Selection of Remedy; and Implementation of Corrective Action Program). See Section XI of this report for a discussion of Corrective Action.

## VIII *Analytical Methods*

This ground-water monitoring program will incorporate appropriate analytical methods that accurately measure monitoring parameters in ground-water samples.
Among acceptable analytical methods are those in Standard Methods for the Examination of Water and Wastewater, 21st Edition, or those listed in SW-846.

- EPA Method 8270 may be used to analyze samples for Non-Purgeable Organic Compounds
- Most heavy metals can be analyzed by inductively coupled plasma-atomic emission spectrometry (ICP).
- Other metals will be analyzed using anion chromatography.
- Attachment 3 contains the Laboratory Standard Operating Procedures for methods employed.

**Table 2 Annual Detection Monitoring Methods and Reporting Limits (RL)**

| Parameter | Method | RL (mg/L) |
|---|---|---|
| Chloride | Method E300 | 1 |
| Iron (total) | Method E200.7 | 0.03 |
| Manganese (total) | Method E200.7 | 0.005 |
| Cadmium (total) | Method E200.7 | 0.002 |
| Zinc (total) | Method E200.7 | 0.001 |
| Total Dissolved Solids | Method E160.1 | 10 |
| Specific Conductance | Method E120.1 | 1 umhos/cm |
| pH | Method E150.1 | 1 |
| Non-purgeable Organic Compounds | Method E415.1 | 0.5 |

## *IX Background Samples*

Four background samples, one per calendar quarter, were taken, for one year. As required, 45 days existed between sampling events. The following table lists the background parameters that were analyzed for during this first year.

**Table 3 Background Sampling Parameters**

| Parameter | Total or Dissolved | Method | MDL mg/L | RL mg/L |
|---|---|---|---|---|
| Cobalt | Total | 219.1 | 0.04 | 0.10 |
| Arsenic | Total | 206.2 | 0.01 | 0.02 |
| Mercury | Total | 245.1 | * | 0.0005 |
| Barium | Total | 208.1 | * | 1.0 |
| Silver | Total | 272.1 | 0.02 | 0.10 |
| Chromium | Total | 218.1 | 0.05 | 0.10 |
| Zinc | Total | 289.1 | 0.05 | 0.10 |
| Lead | Total | 239.2 | 0.004 | 0.015 |
| Cadmium | Total | 213.2 | 0.001 | 0.005 |
| Selenium | Total | 270.2 | 0.01 | 0.02 |
| Copper | Total | 220.1 | * | 0.10 |
| Manganese | Total | 243.1 | 0.02 | 0.05 |
| Iron | Total | 236.1 | 0.14 | 0.3 |
| Alkalinity | N/A | 310.1 | NA | 5 |
| Carbonate | N/A | 310.1 | NA | 5 |
| Hardness | N/A | Calculation | NA | 10 |
| Potassium | N/A | 258.1 | * | 1.0 |
| Phenophthalein alkalinity | N/A | 310.1 | NA | 5 |
| Bicarbonate | N/A | 310.1 | NA | 5 |
| anion-cation ration | N/A | Calc. | NA | NA |
| Calcium | N/A | 215.1 | * | 1.0 |

| **Parameter** | **Total or Dissolved** | **Method** | **MDL mg/L** | **RL mg/L** |
|---|---|---|---|---|
| Magnesium | N/A | 242.1 | 0.24 | 1.0 |
| Sulfate | N/A | 375.4 | 0.84 | 5.0 |
| total dissolved solids | N/A | 160.1 | NA | 10 |
| | N/A | 4500-Cl- B | | |
| Sodium | N/A | 273.1 | 2.3 | 5.0 |
| Fluoride | N/A | 340.2 | 0.02 | 0.10 |
| pH (field & lab) | | | | 1.0 S.U. |
| Specific Conductance (field & | | | | 10umhos |
| nitrate as nitrogen or ammonia as | N/A | | | |
| total organiccarbon (3 replicates) | | | See | See LSOP |
| VOCs | N/A | Best Available | ** | ** |

*Current MDL not available.
**See Table 5: VOC Breakdown and Reporting Limits

X *Detection Monitoring*

Twelve months after the completion of the last quarterly background sampling event, annual monitoring will begin. Analysis will be in accordance with the requirements of 30 TAC §330.417. The monitoring parameters are discussed in Section VII.

The goal of detection monitoring is finding specific constituents that may be leaking from the site. If a breach is suspected, leachate may be analyzed for the detection monitoring parameters. Leachate analysis data can be helpful in supporting a reduction of the number of parameters monitored from the monitoring wells and may be crucial in showing that an anomalous reading was probably not from the landfill.

## XI *Corrective Action*

The Executive Director may require additional sampling, analyses of additional constituents, installation of additional monitoring wells or other sampling points, and/or other hydro-geological investigations if the facility appears to be contaminating the shallow water-bearing zone(s).

If the facility is found to have contaminated or be contaminating the shallow water-bearing zone(s), the Executive Director may order corrective action appropriate to protect human health and the environment up to and including that in §§§§330.411, 330.412, and 333.415 of this title (relating to Assessment of Corrective Measures; Selection of Remedy; and Implementation of Corrective Action Program).

## XII *Quality Assurance and Quality Control (QA/QC)*

All analytical data submitted under the requirements of this permit will be examined by the owner and/or operator to ensure that the data quality objectives are considered and met prior to submittal for the commission to review. The owner or operator will determine if the results representing the sample are accurate and complete. The quality control results, supporting data, and data review by the laboratory must be included when the owner/operator reviews the data. Any potential impacts will be reported such as the bias on the quality of the data, footnotes in the report, and anything of concern that was identified in the laboratory case narrative.

The owner or operator will ensure that the laboratory documents and reports all problems observed anomalies associated with the analysis. If analysis of the data indicates that the data fails to meet the quality control goals for the laboratory's analytical data analysis program, the owner or operator will determine if the data is usable. If the owner and/or operator determines the analytical data may be utilized, any and all problems and corrective action that the laboratory identified during the analysis will be included in the report submitted to the TCEQ.

A Laboratory Case Narrative (LCN) report for all problems and anomalies observed must be submitted by the owner and/or operator. The LCN will report the following information:

1. The exact number of samples, testing parameters and sample matrix.
2. The name of the laboratory involved in the analysis. If more than one laboratory is used, all laboratories shall be identified in the case narrative.
3. The test objectives regarding samples.
4. Explanation of each failed precision and accuracy measurement determined to be outside of the laboratory and/or method control limits.
5. Explanation if the effect of the failed precision and accuracy measurements on the results induces a positive or negative bias.

6. Identification and explanation of problems associated with the sample results, along with the limitations these problems have on data usability.
7. A statement on the estimated uncertainty of analytical results of the samples when appropriate and/or when requested.
8. A statement of compliance and/or non-compliance with the requirements and specifications. Exceedance of holding times and identification of matrix interferences must be identified. Dilutions shall be identified and if dilutions are necessary, they must be done to the smallest dilution possible to effectively minimize matrix interferences and bring the sample into control for analysis.
9. Identification of any and all applicable quality assurance and quality control samples that will require special attention by the reviewer.
10. A statement on the quality control of the analytical method of the permit and the analytical recoveries information shall be provided when appropriate and/or when requested.

The San Antonio Testing Laboratory Quality Assurance Plan (QAP) and Standard Operating Procedures (SOPs) are included as Attachment 3 to this GWSAP.

## XIII *Reporting and Submittals*

The results of the analyses of ground-water samples collected during detection monitoring will be submitted to the Commission that includes all information required by §330.417(b)(5)(A)-(E). Not later than 60 days after each sampling event, Beck Landfill shall determine whether the landfill has released contaminants to the uppermost aquifer. . Triplicate copies of the results are to be submitted.

In addition to the LCN, the following information must be submitted for all analytical data:

1. A table identifying the field sample name with the sample identification in the laboratory report.
2. Chain of custody.
3. An analytical report that documents the results and methods for each sample and analyte to be included for every analytical testing event. These test reports must document the reporting limit/method detection limit the laboratory used.
4. A release statement must be submitted from the laboratory. This statement must state, "I am responsible for the release of this laboratory data package. This data package has been reviewed by the laboratory and is complete and technically compliant with the requirements of the methods used, except where noted by the laboratory in the attached exception reports. By my signature below, I affirm to the best of my knowledge, all problems/anomalies, observed by the laboratory as having the potential to affect the quality of the data, have been identified by the laboratory in the Laboratory Review Checklist, and no information or data have been knowingly withheld that would affect the quality of the data."
5. A laboratory checklist. For every response of "No, NA, or NR" that is reported on the checklist, the permittee will ensure the laboratory provides a detailed description of the "exception report" in the summary of the LCN. The permittee will

require that the laboratory use the checklist and do an equivalent of an EPA level 3 review regarding quality control analysis.

The submittal, including a cover letter, will be in triplicate (one original and two copies). The original is to be filed in TCEQ Central Records in Austin, one copy is sent to the appropriate Regional office, and one copy is used as a work copy by the Commission staff.

## XIV *Safety Plan*

Beck Landfill and/or all of its subcontractors performing functions specific to activities associated with and identified in the GWSAP will establish, implement, and maintain appropriate health and safety plans.

- When sampling at the site, avoid the introduction of contaminants into the body by ingestion, absorption, or respiration.
- Smoking, chewing, drinking, and eating are all prohibited at a waste site.
- Monitor-well water should not be allowed to come in contact with the eyes, mouth, or skin.
- Special care is necessary when handling sample containers, some cleaning solutions, and sample preservatives.
- Combination of reagents may result in a violent reaction.
- Read all warning labels carefully.
- Walk carefully and be aware of steep slopes, unstable ground, poison ivy, fire ant mounds, debris piles, poisonous snakes and spiders, stinging insects, ticks, and mosquitoes.
- Wear proper garments such as boots, hats, gloves, and safety glasses, to protect from exposure.
- Watch out for heavy equipment moving around the site.
- Bring a partner who can help with sampling and transport and will be ready to render aid to the second person or go for help if it becomes necessary.

| **Table 4: Background Sampling** | | | | |
|---|---|---|---|---|
| Parameter | Sample Container | Preservative | Replicates | Holding Time |
| Cobalt | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Arsenic | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Mercury | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 28 Days |
| Barium | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Silver | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Chromium | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Zinc | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Lead | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Cadmium | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Selenium | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Copper | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Manganese | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Iron | 1 Liter Plastic Bottle | Ice (HNO3 if filtered) | No | 6 Months |
| Alkalinity | 1 Liter Plastic Bottle | Ice | No | 200 mL |
| Carbonate | 1 Liter Plastic Bottle | Ice | No | 6 Months |
| Hardness | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| Potassium | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| Phenophthtalein alkalinity | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| Bicarbonate | 1 Liter Plastic Bottle | Ice | No | 28 Days |

| **Table 4: Background Sampling** | | | | |
|---|---|---|---|---|
| Parameter | Sample Container | Preservative | Replicates | Holding Time |
| anion-cation ration | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| Calcium | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| Magnesium | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| Sulfate | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| total dissolved solids | 1 Liter Plastic Bottle | Ice | No | 7 Days |
| Chloride | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| Sodium | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| Fluoride | 1 Liter Plastic Bottle | Ice | No | 28 Days |
| pH (field & lab) | 25 mL Plastic Bottle | None | No | Immediately |
| Specific Conductance (field & lab) | 100 mL Plastic Bottle | None | No | Immediately |
| nitrate as nitrogen or ammonia as nitrogen | 100 mL Plastic Bottle | Ice | No | 48 Hours |
| total organic carbon (3 replicates) | 100 mL Amber Glass | Ice, (HCl, if filtered) | One | 48 Hours (28 Days if acidified) |
| VOCs | 40 mL glass, Teflon lined septa | Ice, (HCl, if filtered) | Two | 48 Hours (28 Days if acidified) |

| **Table 5: VOCs and RLs** | |
|---|---|
| | **Reporting Limit** |
| **Analysis:** | **ug/L** |
| 1,1,1,2 Tetrachloroethane | 5 |
| 1,1 ,1-Trichloroethane | 5 |
| 1,1 ,2,2-Tetrachloroethane | 5 |
| 1,1 ,2-Trichloroethane | 5 |
| 1 ,1-Dichloroethane | 5 |
| 1 ,1-Dichloroethene | 5 |
| 1,2 Dichloropropane | 5 |
| 1 ,2,3-Trichloropropane | 5 |
| 1 ,2-Dibromo-3-Chloropropane | 2* |
| 1 ,2-Dibromoethane | 2* |
| 1 ,2-Dichlorobenzene | 5 |
| 1 ,2-Dichloroethane | 5 |
| 1 ,4-Dichlorobenzene | 5 |
| 2-Butanone (MEK) | 10 |
| 2-hexanone | 10 |
| 4-Methyl-2pentanone | 10 |
| Acetone | 10 |
| Acrylonitrile | 30 |
| Benzene | 5 |
| Bromochloromethane | 5 |
| Bromodichloromethane | 5 |
| Bromoform | 5 |
| Bromomethane | 10 |
| Carbon Disulfide | 5 |
| Carbon tetrachloride | 5 |
| Chlorobenzene | 5 |
| Chlorodibromomethane | 5 |
| Chloroethane (Ethyl Chloride) | 10 |
| Chloroform | 5 |
| Chloromethane | 10 |
| cis-1 ,2-Dichloroethene | 5 |
| cis-1 ,3-Dichloropropene | 5 |
| Dibromomethane | 5 |
| Dichloromethane | 5 |
| Ethylbenzene | 5 |
| Iodomethane | 5 |
| Styrene | 5 |

| **Table 5: VOCs and RLs Continued** | **Reporting Limit** |
|---|---|
| **Analysis:** | **ug/L** |
| Tetrachloroethene | 5 |
| Toluene | 5 |
| trans-1 ,2-Dichloroethene | 5 |
| trans-1 ,3-Dichloropropene | 5 |
| trans-1 ,4-Dichloro-2-Butene | 10 |
| Trichloroethene | 5 |
| Trichlorofluoromethane | 5 |
| Vinyl Acetate | 5 |
| Vinyl Chloride | 2* |
| Xylene | 10* |

* Lower reporting limits are available using a purge volume of 25mL (Cost of analysis will increase) J-Flags (Data Flag) are also possible to indicate the compound is present but below reporting limit.

# Attachment 1 – Purging Worksheets and Sampling Worksheets (24 hours after Purging)

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Purging Field Data Collection Form

Date:_____ Monitor Well No. MW-A

Names:_____ _____

Well Inspection:

Concrete Pad (cracks, fissures, etc.)_____

Casing: _____

Stick Up Locked?_____ Well Cap Locked?_____

Plug Cap Tightened?_____ Insects/Other Issues?_____

Proximity and direction to sources of contamination:_____

_____

Water Level Meter: _____

Decontamination Method: _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (32.98')

(B) Depth to Bottom (nearest 0.01'): (38.82')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

(F) PURGE VOLUME = 3 X (E) _____

**Purge Rate:**

Start Time:_____ End Time:_____ Total Time:_____

(G) PURGE RATE = (F)/TOTAL TIME _____

**Purged Dry?** Yes or No

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Purging Field Data Collection Form

Date:_____ Monitor Well No. MW-C

Names:_____ _____

Well Inspection:

Concrete Pad (cracks, fissures, etc.)_____

Casing: _____

Stick Up Locked?_____ Well Cap Locked?_____

Plug Cap Tightened?_____ Insects/Other Issues?_____

Proximity and direction to sources of contamination:_____

_____

Water Level Meter: _____

Decontamination Method: _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (35.32')

(B) Depth to Bottom (nearest 0.01'): (47.71')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

(F) PURGE VOLUME = 3 X (E) _____

**Purge Rate:**

Start Time:_____ End Time:_____ Total Time:_____

(G) PURGE RATE = (F)/TOTAL TIME _____

**Purged Dry?** Yes or No

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Purging Field Data Collection Form

Date:_____ Monitor Well No. MW-D

Names:_____ _____

Well Inspection:

Concrete Pad (cracks, fissures, etc.)_____

Casing: _____

Stick Up Locked?_____ Well Cap Locked?_____

Plug Cap Tightened?_____ Insects/Other Issues?_____

Proximity and direction to sources of contamination:_____

_____

Water Level Meter: _____

Decontamination Method: _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (33.94')

(B) Depth to Bottom (nearest 0.01'): (42.60')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

(F) PURGE VOLUME = 3 X (E) _____

**Purge Rate:**

Start Time:_____ End Time:_____ Total Time:_____

(G) PURGE RATE = (F)/TOTAL TIME _____

**Purged Dry?** Yes or No

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Purging Field Data Collection Form

Date:_____ Monitor Well No. MW-F

Names:_____ _____

Well Inspection:

Concrete Pad (cracks, fissures, etc.)_____

Casing: _____

Stick Up Locked?_____ Well Cap Locked?_____

Plug Cap Tightened?_____ Insects/Other Issues?_____

Proximity and direction to sources of contamination:_____

_____

Water Level Meter: _____

Decontamination Method: _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (31.68')

(B) Depth to Bottom (nearest 0.01'): (36.65')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

(F) PURGE VOLUME = 3 X (E) _____

**Purge Rate:**

Start Time:_____ End Time:_____ Total Time:_____

(G) PURGE RATE = (F)/TOTAL TIME _____

**Purged Dry?** Yes or No

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Purging Field Data Collection Form

Date:_____ Monitor Well No. MW-G

Names:_____ _____

Well Inspection:

Concrete Pad (cracks, fissures, etc.)_____

Casing: _____

Stick Up Locked?_____ Well Cap Locked?_____

Plug Cap Tightened?_____ Insects/Other Issues?_____

Proximity and direction to sources of contamination:_____

_____

Water Level Meter: _____

Decontamination Method: _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (28.06')

(B) Depth to Bottom (nearest 0.01'): (37.04')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

(F) PURGE VOLUME = 3 X (E) _____

**Purge Rate:**

Start Time:_____ End Time:_____ Total Time:_____

(G) PURGE RATE = (F)/TOTAL TIME _____

**Purged Dry?** Yes or No

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Sampling Field Data Collection Form

Date:_____ Monitor Well No. MW-A

Names:_____ _____

Water Level Meter: _____

Decontamination Method: _____

Water Quality Meter: _____

Decontamination Method: _____

Calibration Date and Results (attach results if necessary): _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (33.02')

(B) Depth to Bottom (nearest 0.01'): (39.80')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

**Field Measurements:**

Sample Collection Start Time:_____ End Time:_____

pH (s.u.) _____

Specific Conductivity (umhos/sec) _____

Temperature (ºF) _____

**Field Duplicate:** Yes or No

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Sampling Field Data Collection Form

Date:_____ Monitor Well No. MW-C

Names:_____ _____

Water Level Meter: _____

Decontamination Method: _____

Water Quality Meter: _____

Decontamination Method: _____

Calibration Date and Results (attach results if necessary): _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (37.10')

(B) Depth to Bottom (nearest 0.01'): (46.24')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

**Field Measurements:**

Sample Collection Start Time:_____ End Time:_____

pH (s.u.) _____

Specific Conductivity (umhos/sec) _____

Temperature (ºF) _____

**Field Duplicate:** Yes or No

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Sampling Field Data Collection Form

Date:_____ Monitor Well No. MW-D

Names:_____ _____

Water Level Meter: _____

Decontamination Method: _____

Water Quality Meter: _____

Decontamination Method: _____

Calibration Date and Results (attach results if necessary): _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (34.05')

(B) Depth to Bottom (nearest 0.01'): (42.43')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

**Field Measurements:**

Sample Collection Start Time:_____ End Time:_____

pH (s.u.) _____

Specific Conductivity (umhos/sec) _____

Temperature (ºF) _____

**Field Duplicate:** Yes or No

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Sampling Field Data Collection Form

Date:_____ Monitor Well No. MW-F

Names:_____ _____

Water Level Meter: _____

Decontamination Method: _____

Water Quality Meter: _____

Decontamination Method: _____

Calibration Date and Results (attach results if necessary): _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (35.05')

(B) Depth to Bottom (nearest 0.01'): (36.55')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

**Field Measurements:**

Sample Collection Start Time:_____ End Time:_____

pH (s.u.) _____

Specific Conductivity (umhos/sec) _____

Temperature (ºF) _____

**Field Duplicate:** Yes or No

Beck Landfill, Nido, LTD.
Type IV Landfill
MSW Permit 1848
Well Sampling Field Data Collection Form

Date:_____ Monitor Well No. MW-G

Names:_____ _____

Water Level Meter: _____

Decontamination Method: _____

Water Quality Meter: _____

Decontamination Method: _____

Calibration Date and Results (attach results if necessary): _____

**Data Collection:** (From top of well casing)

(A) Depth to Water (nearest 0.01'): (28.02')

(B) Depth to Bottom (nearest 0.01'): (37.04')

**Calculations:**

(C) DEPTH OF WATER COLUMN (FT) = (B) – (A) _____

(D) CUBIC FEET OF WATER IN CASING = PI *R2*(C)

= (3.14 *( 0.17')$^2$ ) * (C) = **0.0872 SFT** *(C) = _____

(E) CONVERSION TO GALLONS =(D) * 7.48 _____

**Field Measurements:**

Sample Collection Start Time:_____ End Time:_____

pH (s.u.) _____

Specific Conductivity (umhos/sec) _____

Temperature (ºF) _____

**Field Duplicate:** Yes or No

# Attachment 2 – Chain of Custody Form

# CHAIN-OF-CUSTODY RECORD


**SAN ANTONIO TESTING LABORATORY, LLC**

1610 S. Laredo Street, San Antonio, Texas 78207
(210) 229-9920 • Fax (210) 229-9921
www.satestinglab.com


| REPORT TO: | INVOICE TO: | P.O. # |
|---|---|---|
| COMPANY | COMPANY | REPORT NUMBER |
| ADDRESS | ADDRESS | |
| CITY STATE ZIP | CITY STATE ZIP | E-MAIL |
| ATTN: PHONE # | ATTN: PHONE # | |

REQUESTED TURNAROUND TIME IN BUSINESS DAYS & SURCHARGE: ☐ 7-10 Days REG ☐ 5 Days +25% ☐ 4 Days +50% ☐ 3 DAYS +75% ☐ 2 DAYS +100% ☐ Next Day +150% ☐ SAME DAY WHEN POSSIBLE +300%

PROJECT NAME/LOCATION/SITE

THE TURNAROUND TIME FOR SAMPLES RECEIVED AFTER 3:00 PM SHALL BEGIN AT 8:00 AM THE FOLLOWING BUSINESS DAY / SPECIAL REQ.:

FOR STATE COMPLIANCE ☐ YES ☐ NO

PROJECT NO.

SAMPLE TEMPERATURE WITHIN COMPLIANCE (> 0°C ≤ 6°C) ☐ YES ☐ NO
PROPER CONTAINERS ☐ YES ☐ NO
INITIAL TO AUTHORIZE BULK ANALYSIS _____
IF NO, INITIAL HERE TO AUTHORIZE ANALYSIS _____
INSUFFICIENT SAMPLE AMOUNT: ☐ YES ☐ NO AUTHORIZE TO PROCEED_____

SAMPLED BY

OBSERVED TEMP. / CORRECTED TEMP. / TEMP. I.R. GUN #_____ / SAMPLE ICED ☐ YES ☐ NO

TRRP 13 ☐ LPST PCLS ☐ PST ☐ TSDF Class 2 ☐

| SAMPLE NUMBER | COLLECTED: DATE | COLLECTED: TIME | MATRIX: DRINKING WATER | MATRIX: NON-POTABLE WATER | MATRIX: PAINT CHIPS | MATRIX: OIL | MATRIX: ORGANIC LIQUID | MATRIX: SOLID | MATRIX: SLUDGE | MATRIX: OTHER | SAMPLING METHOD: COMPOSITE | SAMPLING METHOD: GRAB | SAMPLING METHOD: CORE | SAMPLING METHOD: OTHER | SAMPLE IDENTIFICATION | NUMBER OF CONTAINERS | SAMPLE AMOUNT | CONTAINER SIZE | ANALYSIS REQUESTED: BTEX/MTBE 8260 / TPH TX1005/TX1006 | Metals 8 / 11 / 12 / 13 / TCLP / SPLP / Total | PAH / SVOC / 8270 / 625 / TCLP / SPLP / Total | VOC / 8260 / 624 / TCLP / SPLP / Total | Water Quality - Drinking / Livestock / Irrigation | Coli / TC / FC / HPC / EColi / Enterococci / Q-Tray | Br / Cl / F / NO3 / NO2/O-P / SO4 | PCB / 608 / 8082A | OC Pest / 608 / 8081A / TCLP / SPLP / Total | PRESERVED WITH: ICE | HCl | HNO3 | H2SO4 | NaOH | NaOH+ZnAc | MeOH | HNO3 | PRESERVED IN THE LAB | COMPOSITED IN THE LAB | FILTERED IN THE LAB | FILTERED IN THE FIELD | REMARKS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| RELINQUISHED BY (SIGNATURE) DATE / TIME | RECEIVED BY (SIGNATURE) DATE / TIME | RELINQUISHED BY (SIGNATURE) DATE / TIME | RECEIVED BY (SIGNATURE) DATE / TIME |
|---|---|---|---|
| RELINQUISHED BY (PRINT NAME) DATE / TIME | RECEIVED BY (PRINT NAME) DATE / TIME | RELINQUISHED BY (PRINT NAME) DATE / TIME | RECEIVED BY (PRINT NAME) DATE / TIME |
| RELINQUISHED BY (SIGNATURE) DATE / TIME | RECEIVED BY (SIGNATURE) DATE / TIME | METHOD OF SHIPMENT | SUBCONTRACTED ☐ YES ☐ NO |
| RELINQUISHED BY (PRINT NAME) DATE / TIME | RECEIVED BY (PRINT NAME) DATE / TIME | SAMPLED IN 5035 CONTAINERS ☐ YES ☐ NO ☐ N/A | CUSTODY SEAL IN PLACE & INTACT ☐ YES ☐ NO |

# Attachment 3 – QAPP and SOP

# Quality Assurance Manual (QAM) Rev. 5 Training

# Contents of Quality Assurance Manual Rev. 4.1

- Section 1: Cover pages
- Section 2: Table of Contents
- Sections 3-29
- Appendices A-G

# Section 3: Introduction and Scope

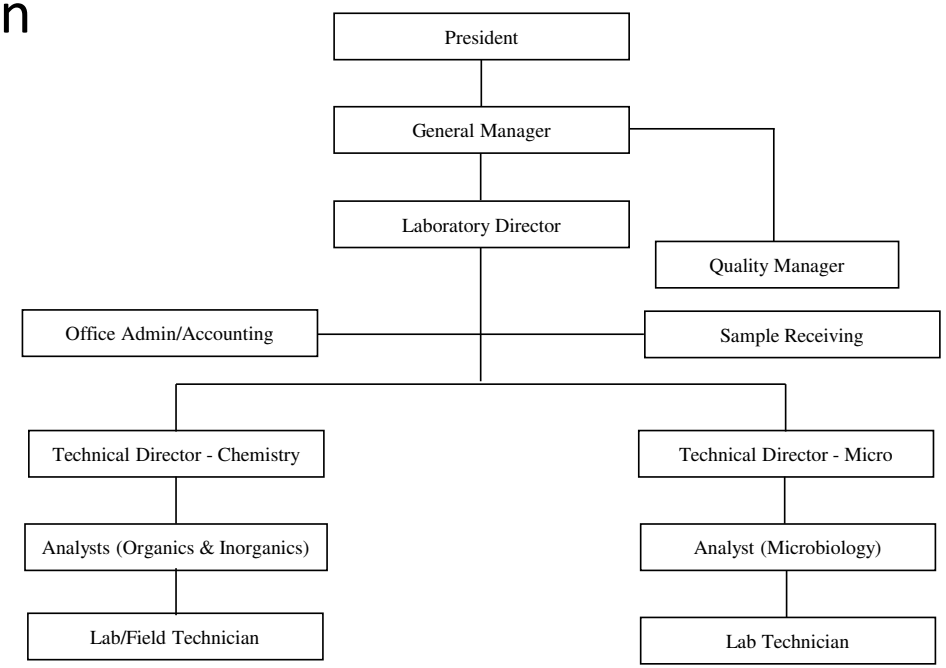
- Purpose: Outline the quality system for SATL to provide clients with data of known and documented quality
- Policy: Understand and meet regulatory requirements while providing clients with independent, reliable, accurate, legally defensible analytical services with fast turnaround times
- Reference: Modules 1, 2, 4 and 5 of the 2016 TNI Environmental Laboratory Sector Standard
- Acronyms provided
- The QA Manager maintains the current version of the QA Manual
- The QA Manual is reviewed at least once every 2 years

# Section 4: Organization

- SATL is organized into 4 departments:
  - Administrative
  - Organics
  - Inorganics (metals & wet chemistry)
  - Microbiology
- The laboratory assures that it is impartial and that personnel are free from undue commercial, financial or other undue pressures that might influence their technical judgement
- Ethics and data integrity policies (Appendix E and Sections 5 and 19) ensure that personnel do not engage in activities that diminish confidence in the laboratory's capabilities
- All employees must sign a Conflict of Interest statement form (Appendix F) and the Ethics Policy (Appendix E)

# SATL Organization

## SATL Organization



# Section 5: Management

- Laboratory Director has overall responsibility and authority for technical operations of the laboratory
- QA Manager has overall responsibility for required quality of laboratory operations
- Management is responsible for meeting the requirement of the TNI Standard 2016 and the needs of the client
- Technical Director has education and experience requirements (see Section 5.2.6.1 of the TNI V1M2-2016 standard)
- Quality Policy: The objective of the quality system, and the commitment of management is to consistently provide customers with data of known and documented quality that meets their requirements (see page 15 of QAM for full policy)

# Section 5: Management (cont.)

- The Ethics Policy is documented in Appendix E. The Ethics and Data Integrity program, training and investigation is documented in QAM Section 19.
- The quality system is documented in the QA Manual and written SOPs.
- There are technical and general SOPs
  - Technical SOPs are divided into Inorganics-Wet Chemistry, Inorganics-Metals, Organics-Semivolatiles, Organics-Volatiles and Microbiology
  - General SOPs are for front office and disposal
- In the event of a conflict or discrepancy, the order of Precedence is: TNI Modules, QA Manual, Methods and SOPs and Policies

# Section 6: Document Control

- SATL has 3 types of documents:
  - Controlled (QA Manual, SOPs, Forms and Methods)
  - Approved (Work Orders, Test Reports)
  - Obsolete (Documents superseded by more recent version or those no longer in use needed)
- Controlled documents are reviewed at least every 2 years or as needed
- Controlled documents are available on the Q drive/Controlled Documents/SOPs (pdf versions) or Forms
- Document changes are approved by President, Technical Directors and QA Manager
- Electronic signatures are used on laboratory documents, quality system documents and test reports

# Section 7: Review of Requests, Tenders and Contracts

- The Lab Director determines if the laboratory has the necessary accreditations, resources and personnel to meet work requests
- The President makes the decision whether to accept or forego the work
- For new, complex or large projects, the proposed contract is given to the President and Lab Director for review
- Records are maintained for every contract or work request
- Records of all project-related communication with clients is kept in the final report folder for each client

# Section 8: Subcontracting Environmental Tests

- When subcontracting analytical services, the lab assures that work requiring accreditation is sent to an appropriately accredited lab
- The certificate of accreditation is reviewed by the QA Manager and/or Lab Director and/or President to ensure that the subcontracting lab has the appropriate accreditation to do the work
- Subcontractor accreditation certificates are available on the Q drive/Accreditation and Certifications/Subcontractors
- Approved subcontractors are in Q drive/Controlled Documents/Quality Manual/QAM Appendices
- Subcontract details are documented on the COC and the Sample Receipt Checklist
- The lab performing subcontracting is identified in the final report

# Section 9: Purchasing Services and Supplies

- SATL ensures that purchased supplies and services that affect quality of environmental tests are of the required or specified quality by using approved suppliers and products
- The Lab Director reviews and approves suppliers and approves the technical content of purchasing documents prior to ordering
- Approved vendors are in Q drive/Controlled Documents/Quality Manual/QAM Appendices
- Supplies received are inspected for breakage, leaks or damages. Supplies are checked in (dated and initialed) on the packing slip
- Certificates of Analysis (COAs) are kept with the department and scanned into Element
- Supplies are stored according to the manufacturer's instructions, laboratory SOP or test method specifications
- Chemicals, standards and reagents are logged into the Element LIMS database which creates a unique ID which is used on the containers and logbooks.

# Section 10: Service to the Client

- SATL collaborates with clients in clarifying their requests and in monitoring laboratory performance related to their work
- The SATL client confidentiality policy is to not divulge or release any information to a third party with proper authorization
- A confidentiality statement accompanies all electronic mail to clients
- Communication with clients is maintained to provide proper instruction and modification for testing; delays or major deviations are communicated to the client immediately by President or Lab Director
- SATL seeks both positive and negative feedback following completion of projects and may use a survey request; negative feedback is documented as a customer complaint

# Section 11: Complaints

- Complaints by customers or other parties are reviewed by SATL management (President and Lab Director) and an appropriate action is determined
- All customer complaints are documented by the person receiving the complaint and addressed to the responsible manager
- Initial evaluation of the complaint may result in using the Customer Complaint form
- Complaints are resolved as soon as practically possible
- If it is determined that the complaint has merit, then corrective action will be utilized
- A complaint such as a concern that data is repeatedly late is reviewed for preventive action to minimize a future occurrence

# Section 12 – Control of Nonconforming Work

- Non-conforming work is work that does not meet acceptance criteria or requirements
- Non-conforming work can come through customer complaints, QA, instrument data, calibration data, staff observation, final report reviews, management reviews and internal and external audits
- The procedure for investigating and taking appropriate corrective action on non-conforming work is described in Section 14
- Employees shall notify the QA Manager or Technical Director of any nonconformance as soon as it is noticed/observed/detected
- The QA Manager/Laboratory Director/Technical Director reviews the nonconformance and determines a course of action
- A Stop Work Order may be used if a method is restricted or not used until modifications are implemented

# Section 13: Improvement

- Improvement in the overall effectiveness of the lab's quality system may result from implementation of the lab's management system:
  - Quality policy and objectives
  - Corrective action
  - Preventive action
  - Internal auditing
  - Ethics and data integrity program
  - Review and analysis of data
  - Annual management review of the quality management system

# Section 14: Corrective Action

- Corrective action is the action taken to eliminate the cause of an existing nonconformance, defect or other undesirable situation
- Deficiencies cited in external assessments, internal audits, data reviews, customer feedback/complaints, control of nonconforming work or managerial reviews are documented and require corrective action
- Corrective Action Form is used to document and track corrective action
- Root cause analysis is used to determine the cause of the nonconformance
- Corrective action needs to be appropriate to correct the problem and prevent recurrence
- QA Manager will monitor the implementation and effectiveness of the corrective action

# Section 15: Preventive Action

- Preventive action is a proactive process to identify opportunities for improvement
- All personnel have the authority to offer suggestions for improvement
- Preventive action includes
  - Review of QC data to identify trends
  - Regularly scheduled staff quality meetings to ensure staff is knowledgeable in quality procedures
  - Annual budget and managerial reviews
  - Review of proficiency testing data to identify near misses
  - Scheduled instrument maintenance

# Section 16: Control of Records

- Records include data recordings, laboratory forms, list, spreadsheets, analyst notes; Records are electronic and hard copy.
- SATL records all laboratory activities in order to establish an audit trail
- SATL retains all original observations, calculations and derived data, calibration records and test reports for a minimum of 5 years
- Sample records are organized by year and client name
- A backup of electronic data is performed on a weekly basis and an automatic incremental backup is done on a daily basis

# Section 17: Audits

- Audits measure laboratory performance and verify compliance with accreditation and project requirements. Audits can be internal, external, performance and system
- Internal
  - Conducted at least annually
  - May be conducted by a consultant
- External audits
  - Accreditation or client audits
- Performance audits
  - Proficiency test samples
- System audits
  - Annual management review meetings
- Audit findings are handled through the corrective action process

# Section 18: Management Reviews

- Management reviews are conducted in the first quarter of the year and review the following for suitability and effectiveness:
  - Policies and procedures
  - Reports from managerial and supervisory personnel
  - Outcome of recent internal audits
  - Corrective and preventive actions
  - Assessments by external bodies
  - Results of proficiency tests
  - Customer feedback and complaints
  - Recommendations for improvement
  - Review of data integrity procedure
  - Quality control activities, resources, facility and staff training
  - Ethics and data integrity program

# Section 19: Data Integrity Investigations

- Ethics and Data Integrity Program
  - Documented data integrity procedures
  - Ethics Policy signed by all management and staff annually
  - Ethics and data integrity training is provided for new employees within 3-5 days of hire and annually for all personnel
  - Procedures for confidential reporting of alleged data integrity issues
  - Audit program that monitors data integrity
  - Procedures for handling data integrity investigations and client notifications

# Section 19: Data Integrity Investigations (cont.)

- Examples of unethical behavior
  - Fabricating results
  - Altering instrument settings
  - Altering the Chain of Custody record
  - Altering calculations
  - Altering approved SOPs
  - Lack of reporting unethical behavior by others

# Section 19: Data Integrity Investigations (cont.)

- Data integrity training includes:
  - SATL organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting
  - How and when to report data integrity issues
  - Recordkeeping
  - Data integrity procedures
  - Data integrity training documentation
  - In-depth data monitoring and data integrity procedure documentation
  - Specific examples of breaches of unethical conduct, such as improper data manipulations, adjustments of instrument time clocks, inappropriate changes in concentrations of standards

# Section 19: Data Integrity Investigations (cont.)

- Confidential reporting of ethics and data integrity issues is assured through:
  - Unrestricted access to senior management
  - Assurance that personnel will not be treated unfairly for reporting instances of ethics and data integrity issues
  - Anonymous reporting
- Investigations
  - Documented and conducted confidentially
  - Allegations are investigated
  - Affected clients notified

# Section 20: Personnel

- All personnel are responsible for complying with all quality and data integrity policies and procedures relevant to their area of responsibility
- Initial, ongoing and refresher training is provided as needed
- Personnel are qualified to perform tasks they are responsible for based on education, training, experience and demonstrated skills
- The Laboratory Director is responsible for the laboratory operations and staff supervision
- The QA Manager is responsible for ensuring that the quality system is implemented and followed
- The Technical Director is responsible for day to day supervision of technical laboratory operations

# Section 20: Personnel - Training

- Training for new staff
  - All associated documentation with the task must be read and understood
  - Hands on training will be provided under the direct supervision of a qualified senior analyst or Laboratory Director
  - The trainee must demonstrate competency in the new task before they can operate independently
  - Approval of competency is documented by the Technical/Laboratory Director on the training form

# Section 20: Personnel – Training (cont.)

- Ongoing training
  - The analyst attests that they have read, understood and agree to perform according to the latest version of the Quality Manual and method SOPs
  - Semiannually, the analyst will show continued proficiency by analyzing PT samples for the tests that they are responsible for
  - Proof of acceptable on-going training is documented by annual demonstrations of capability by each analyst and each method
- Refresher training
  - Will be provided as needed based on nonconformances, audit findings, PT study failures or customer complaints

# Section 21: Accommodations and Environmental Conditions

- Environmental conditions that are controlled and monitored include temperature, humidity, voltage, biological sterility, dust, light, sound and vibration levels
- Access to areas affecting the quality of results such as sample storage, records, laboratory facility, LIMS system is restricted to authorized personnel only
- Chemicals are stored in appropriate areas; acids are stored in cabinets under fume hoods, solvents are stored in metal cabinets, standards and reference materials are stored in separate refrigerators from sample extracts

# Section 21: Accommodations and Environmental Conditions (cont.)

- Laboratory space is arranged to minimize cross-contamination; microbiology, volatiles, semivolatiles and metals are in separate areas
- Electric balances are kept away from drafts and vibrations
- A janitorial service is used for general housekeeping
- Periodic cleanup days are used to help clean up clutter
- Each employee is responsible for housekeeping in their work area at the end of the day
- Smoking/eating/drinking are prohibited in the laboratory area
- Building security includes locks, alarm system and cameras

# Section 22: Environmental Methods and Method Validation

- Reference methods and/or procedures are available for all activities associated with the preparation and analysis of samples
- Reference methods are validated by a **demonstration of capability (DOC)** which is a procedure to establish the ability of the analyst to generate data of acceptable precision and accuracy
- A DOC is performed whenever the method, analyst or instrument type is changed
- DOC: 4 replicates prepared at the mid-point of the calibration or LCS spike level from a certified reference standard (QC sample) purchased from an approved vendor. The QC sample is prepared in a clean matrix such as DI water, Ottawa sand or clean sodium sulfate

# Section 22: Environmental Methods and Method Validation (cont.)

- The analysis of 4 DOC replicates is compared to established control limits and checked for precision and accuracy
  - If the results of 4 replicate analyses fall within the method control limits, then the analyst has demonstrated their capability in that method
  - If 1 or more analytes fail to meet the acceptance criteria, then the replicate analyses are repeated. A second failure indicates a potential problem that needs corrective action
  - In the case of microbiology, presence/absence is demonstrated using a set of 10 replicate samples
  - For enumeration techniques, 4 samples inoculated with microorganisms of known CFU range are analyzed or commercially available enumeration QC samples are used
- The DOC results are documented in the training file for each analyst
- After the initial DOC is completed, on-going proficiency is demonstrated by analysis of single blind samples, performing another DOC or using 4 consecutive LCSs

# Section 22: Environmental Methods and Method Validation (cont.) NEW

- **Method Detection Limit (MDL)** is an estimate of the minimum amount of an analyte that an analytical process can reliably detect.
- MDL values are generated in accordance with 40 CFR Part 136 Appendix B Revision 2 which includes a minimum of 7 spiked samples at 2-10 times the estimated MDL and a minimum of 7 method blank samples.
- The samples used for the initial MDL must be prepared in at least 3 batches on 3 separate calendar days and analyzed on 3 separate calendar days (preparation and analysis may be on the same day).
- If any result for any individual analyte does not provide a numerical result greater than 0, then repeat the spiked samples at a higher concentration.
- See attachment for the $MDL_s$ and $MDL_b$ calculations. Select the greater of $MDL_s$ and $MDL_b$ as the initial MDL.

# Section 22: Environmental Methods and Method Validation (cont.) NEW

**MDL**

- **During each quarter**, prepare and analyze a minimum of 2 spiked samples on each instrument, in separate batches, using the same spiked concentration as the initial MDLs.
- Ensure that at least 7 spiked samples and 7 method blanks are completed for the annual verification.
- **At least once per year**, reevalute the spiking level – if more than 5% of the spiked samples do not yield results greater than 0, then the spiking level must be increased and the initial MDL redetermined.
- **At least once every 13 months**, recalculate $MDL_s$ and $MDL_b$.

# Section 22: Environmental Methods and Method Validation (cont.)

- The Limit of Quantitation (LOQ) or Practical Quantitation Limit (PQL) is an estimate of the minimum amount of an analyte that can be reported with a specified degree of confidence
- The lowest calibration standard is equal to the LOQ
- The LOQ/PQL is always greater than the LOD/MDL
- Precision is the degree to which a set of measurements of the same property obtained under similar conditions conform the themselves.
- Bias is the systematic error that contributes to the difference between the mean of a significant number of test results and the accepted reference value
- Precision and bias are determined through performance of a DOC

# Section 22: Environmental Methods and Method Validation (cont.)

- Selectivity is the capability of a test method or instrument to respond to targeted analytes in the presence of non-target analytes
- Estimation of uncertainty is sum of the uncertainties of the numerous steps of the analytical process
- Control of Data
  - Automated and manual procedures are used to check calculations and data transfers
  - Excel spreadsheet formulas are validated and locked
  - Commercial off-the-shelf software is used and is considered validated
  - Access to application software is by a user name and password
  - Access to the building is by means of a key and security code

# Section 22: Environmental Methods and Method Validation (cont.)

- Control of Data (continued)
  - Most instruments the laboratory uses have the capability to export data out of the instrument software and into the LIMS software
  - All reports to clients and quality control measures are reviewed prior to reporting to clients through the use of a Final Review Checklist
- Procedure for Minimizing Errors
  - Transcription errors are minimized by secondary review
  - Calculation errors are minimized by the use of automated spreadsheets
  - Manual integration criteria are addressed in SOP003D

# Section 23: Calibration Requirements

| Instrument | Activity | Frequency | Documentation |
|---|---|---|---|
| Balance | 1. Clean<br>2. Check alignment<br>3. Calibration & Service | 1. Before use<br>2. Before use<br>3. Annual | Log book<br><br>Post annual service date on balance |
| ASTM Class 1Weights | 1. Only use for the intended purpose<br>2. Use plastic forceps to handle<br>3. Keep in case | Once every 5 years | Keep certificate |
| Thermometers: Glass and electronic | Check bracketing the temperature of use, against a reference NIST certified thermometer | Annual for glass and electronic within ± 7 days of last calibration | Calibration factor and date of calibration on thermometer, Log book and calibration form |
| pH electrometers | Calibration:<br>1. pH buffer aliquot are used only once<br>2. Buffers used for calibration will bracket the pH of the media, reagent, or sample tested. | Before use | Logbook |
| pH probe | Maintenance:<br>Use manufacturer's specifications | As needed | Logbook |
| Conductivity meter | Calibration:<br>Conductivity standard will bracket the conductivity of the media, reagent, or sample tested. | Before use | Log book |

Support equipment such as balances, ovens, refrigerators, freezers and water baths are verified with an NIST traceable reference, each day prior to use, to ensure operation is within the expected range.

# Section 23: Calibration Requirements (cont.)

| | | | |
|---|---|---|---|
| Spectrophotometer | 1. Keep cells clean | As needed | Logbook |
| Automatic or digital type pipettes | Calibrate for accuracy and precision using reagent water and analytical balance | Quarterly | Logbook |
| Refrigerators, Freezers, and BOD incubators | 1. Thermometers are immersed in liquid to the appropriate immersion line<br>2. The thermometers are graduated in increments of 1°C or less | Temperatures are recorded each day in use by an analysts. The min/max digital thermometer is use to record temperatures for units containing samples or reagents used for analytical procedures during the weekend and holidays. | Logbook |
| Sterilizer [microbiology] | 1. Use a maximum-temperature-registering thermometer<br>2. Use spore strips or ampules.<br>3. Service contract | 1. Each cycle<br>2. One sterilizing cycle per month<br>3. As needed | Logbook |
| Microbiological incubators, and water baths | 1. Thermometers in each unit are immersed in liquid to the appropriate immersion line<br>2. The thermometers will be graduated in increments of 0.5°C (0.2°C increments for tests which are incubated at 44.5°C) or less | Temperature of incubators and water baths will be recorded twice a day for each day in use with readings separated by at least four hours | Logbook |
| DO probe | Maintenance as specified by manufacturer | As needed | Logbook |
| TKN Digestion Block | Internal thermocouple is checked at the programmed temperatures of 225°C and 380°C | Annually | Log book |
| COD Digester Block | Internal thermocouple is checked at the end of analytical cycle at 150°C. | Annually | Log book |

# Section 23: Calibration Requirements (cont.)

NEW

- For regression or average response /calibration factor calibrations, the following minimum number of non-zero calibration standards shall be used, in accordance with Section 1.7.1.1.f the TNI 2016 standard:

| Type of Calibration Curve | Minimum Number of Calibration Standards |
|---|---|
| Threshold testing | 1 |
| Average response | 4 |
| Linear fit | 5 |
| Quadratic fit | 6 |

The lowest calibration standard shall be at or below the lowest concentration for which quantitative data are reported without qualification. The highest calibration standard shall be at or above the highest concentration for which quantitative data are reported without qualification.

# Section 23: Calibration Requirements (cont.)

# NEW

As per Volume 1, Module 4, Section 1.7.1.1.e of the TNI 2016 standard, the following is the **policy on removal and replacement of calibration standards**:

- i. The laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of interior levels is not permitted.
- ii. The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. A laboratory that chooses to remove a calibration standard from the interior of the calibration shall remove that particular standard calibration level for all analytes. Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.
- iii. The laboratory shall adjust the LOQ/reporting limit and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.
- iv. The laboratory shall ensure that the remaining initial calibration standards are sufficient to meet the minimum requirements for number of initial calibration points as mandated by this Standard, the method, or regulatory requirements.
- v. The laboratory may replace a calibration standard provided that:
- a. the laboratory analyzes the replacement standard within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;
- b. the laboratory replaces all analytes of the replacement calibration standard if a standard within the interior of the calibration is replaced; and
- c. the laboratory limits the replacement of calibration standards to one calibration standard concentration.
- vi. The laboratory shall document a technically valid reason for either removal or replacement of any interior calibration point.

# Section 23: Calibration Requirements (cont.)

# NEW

- The laboratory shall use and document a measure of **relative error in the calibration**.
- i. for calibrations evaluated using an average response factor, the determination of the relative standard deviation (RSD) is the measure of the relative error;
- ii. for calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory shall evaluate relative error by either:
- a. measurement of the Relative Error (%RE). See attachment for the calculation.
- This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level.
- The RE at both of these levels shall meet the criteria specified in the method. If no criterion for the lowest calibration level is specified in the method, the criterion and the procedure for deriving the criterion shall be specified in the laboratory SOP.

or,

- b. measurement of the Relative Standard Error (%RSE). See attachment for the calculation.
- The RSE shall meet the criterion specified in the method. If no criterion is specified in the method, the maximum allowable RSE shall be numerically identical to the requirement for RSD in the method. If there is no specification for RSE or RSD in the method, then the RSE shall be specified in the laboratory SOP.

# Section 23: Calibration Requirements (cont.)

- Continuing Calibration Verification (CCV): The validity of the initial calibration shall be verified prior to sample analysis by a CCV with each analytical batch
- The CCV concentration shall be equal to or less than half of the highest level of calibration
- Instrument CCV shall be performed at the beginning and end of each analytical batch, and at frequency defined in the method
- If routine corrective action for an instrument CCV fails to produce an acceptable CCV, then a new initial calibration shall be performed

# Section 24: Measurement Traceability

- Measurement quality comes in part from traceability of standards to certified materials/standards
- All equipment is calibrated and traceable to national standards of measurement where available
- All equipment that affects the quality of test results is calibrated according to the minimum frequency specified by the manufacturer, regulation or method
- **Reference standards** are standards of the highest quality available, such as ASTM Class 1 weights or NIST reference thermometers (weights are calibrated every 5 years and NIST thermometers are calibrated every 2 years)

# Section 24: Measurement Traceability (cont.)

- **Reference materials** are traceable to national standards of measurement or to Certified Reference Materials, by a Certificate of Analysis (CoA)
- Reference standards and materials are tracked from purchase, receipt and storage through disposal
- Records for all standards, reagents, reference materials and media shall include the vendor name, the CoA, date of receipt, date of preparation, expiration date and recommended storage conditions
- *******All containers of standards, reagents or materials, whether original or prepared shall be logged into Element and assigned a unique ID – this unique ID is used in all associated data, logs, and spreadsheets *******
- **CoAs shall be labeled with the unique ID and scanned and uploaded in Element**

# Section 24: Measurement Traceability (cont.)

- Records for prepared standards, reagents, reference materials and media shall include:
  - Traceability to purchased stock or neat analytes
  - The manufacturer’s CoA or purity
  - The date of receipt
  - Reference to the method of preparation
  - Date of preparation
  - Recommended storage conditions
  - Expiration date
  - Preparer’s initials

# Section 25: Collection of Samples

- SATL provides sampling services including sampling containers
- Sample kits include:
  - Appropriate container with preservative, if required
  - Sample labels
  - Chain of Custody forms
  - Custody seals
  - Cooler
- Sampling records include:
  - Sampling procedure
  - Date and time of sampling
  - Matrix type
  - Identification of the sample and sampler
  - Sampling location and environmental conditions

# Section 26: Handling Samples and Test Items

- When samples are received at the lab:
  - Their condition is documented on the Chain of Custody form
  - They are assigned a unique report number and sample identifier (2 digits for year, 2 digits for month and 3 digits for sequential number, i.e., 2010203)
  - The work orders are logged in a logbook and into Element
  - A Sample Receipt Checklist is completed
- Clients will be notified of any deviations and they will need to sign the COC or send email authorization to proceed
- COCs and any additional records received with the samples are maintained electronically (P drive) as well as in a client file folder

# Section 26: Handling Samples and Test Items (cont.)

- Sample Preservation Checks
  - Thermal preservation – checked for samples requiring temperature preservation (>0 $^{o}$C to ≤ 6 $^{o}$C); record temperature on the Sample Receipt Checklist and note if ice was present
  - Chlorine checks – chlorine is checked on potable water samples
  - pH checks – performed by the analyst for samples requiring acid/base preservation and documented in the logbook, electronic spreadsheet or benchsheet
- Sample Identification
  - All samples, including subsamples, extracts and digestates are uniquely identified in a permanent chronological record
- Sample Storage
  - Samples are held secure in temperature controlled refrigerators and/or freezers. The temperature is monitored and recorded daily. Limits are >0 to 6 degrees C.

# Section 26: Handling Samples and Test Items (cont.)

- Sample disposal
  - Samples are disposed of according to Federal, State and local regulations
  - Waste is segregated into 3 main categories (liquid waste, solid waste and organic waste) with subcategories based on the process it was generated from and stored in various sized drum
  - The waste list and codes are in form SATL MISC004

# Section 27: Quality Assurance for Environmental Testing

- Quality control measurements:
  - Blanks
  - Laboratory control samples (LCS)
  - Matrix spikes (MS)
  - Duplicates
  - Surrogates and internal standards
- Proficiency testing samples also assess laboratory performance
  - Water Pollution (WP) – 2x/year in March-May and Sept.-Nov.
  - Water Supply (WS) – 2x/year in March-May and Sept.-Nov
  - Hazardous Waste (HW) – 2x/year in March-May and Sept.-Nov
  - Two out of three PT studies in a row must pass to be in compliance with TCEQ & TNI

# Section 27: Quality Assurance for Environmental Testing – for Chemistry

| Item | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Method blank (negative control) | Every 20 samples or 1/batch | Method specific or Reporting limit | Qualify data and take corrective action |
| LCS (positive control) | Every 20 samples or 1/batch | Method specific or as determined by the lab | Reprocess, reanalyze or qualify data |
| MS/MSD | Every 20 samples or per method requirement | Method specific or as determined by the lab | Qualify data and take corrective action |
| Duplicates | Every 20 samples or per method of SOP requirement | Method specific or as determined by the lab | Qualify data and take corrective action |
| Surrogates | Every organic sample and QC sample | Method specific or as determined by the lab | Qualify data and take corrective action |
| ICV | Initially and on CCV failure | Method specific or as determined by the lab | Reanalyze standard and take corrective action |
| CCV | Per test method or SOP requirement | Method specific or as determined by the lab | Reanalyze standard and take corrective action |

# Section 27: Quality Assurance for Environmental Testing – for Microbiology

| Item | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Sterility check | Each lot of media prior to use | No growth | Investigate cause |
| Sterility check containers | One container for each lot or batch sterilized | No growth | Investigate cause |
| Sterility check dilution water | One per batch of dilution water | No growth | Investigate cause |
| Positive control | Prior to first use of medium | Positive reaction | Investigate cause. If necessary, reject medium |
| Negative control | Prior to first use of medium | Negative reaction | Investigate cause. If necessary, reject medium |
| Duplicate MPN counts | Monthly on one positive sample for each month | Same analyst <5%D between counts (2 analysts 10%D) | Investigate cause<br>Qualify data |
| Quanti-tray seal check | Once per month | No leaks | Investigate cause |

# Section 28: Reporting the Results

- The result of each test performed must be reported accurately, clearly, unambiguously and objectively to comply with all specific instructions contained in the test method
- Laboratory results are reported in a test report that includes all information requested by the client and necessary for interpretation of the test results
- Test results are reported with the analyte, result, units, PQL, batch, method, date, analyst and notes
- Reports include the sample information, client information, NELAC certification and authorization by SATL management
- Reports are transmitted electronically to the client
- Amended test reports include a new date and time and comment describing the reason for the revision

# Appendices

- Appendix A – Analytical Methods, Sample Preparation and Holding Times
- Appendix B – Sample Receipt Checklist
- Appendix C – Final Report Review Checklist
- Appendix D – Laboratory Qualifiers
- Appendix E – Laboratory Ethics Policy
- Appendix F – Conflict of Interest Form
- Appendix G – Client Confidentiality

# STANDARD OPERATING PROCEDURE

*Title*

**Analysis of Total Metals By ICP – AES**

*Method No.:*

**EPA 200.7 and EPA 6010B**

*Matrix/Matrices:*

**Liquid/Solid**

*Document Control Number/Revision Number*

**SOP003B/Revision 5.1**

| | |
|---|---|
| Charles R. Morrow | 09/14/21 |
| ***Approved By: Quality Assurance Manager*** | ***Date*** |
| Richard Hauk | 09/14/21 |
| ***Approved By: General Manager*** | ***Date*** |
| | 09/14/21 |
| ***Approved By: Laboratory Director*** | ***Date*** |

Standard Operating Procedures shall be reviewed at least once in two years or as needed to determine their continued suitability, compliance with applicable requirements, and to ensure that they reflect actual procedures being performed.



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**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF TOTAL METALS BY ICP – AES [EPA 200.7 & 6010B]**

## 1.0 SCOPE AND APPLICATION

**1.1** This Standard Operating Procedure describes the analysis and determination of metals by ICP – AES.

**1.2** This method is applicable to most matrices including ground water, liquids, and digestate of TCLP, waste, soil, sludge, sediment, and other solid wastes.

## 2.0 REPORTING LIMIT

**2.1** This procedure yields reporting limits for various elements; typical limits are as shown in the following table.

**2.2** Lower limits of quantitation may be possible when a lower calibration point is included as part of the calibration curve.

**TABLE – A**

| Elements | CAS No. | Water (mg/L) | Soil (mg/Kg) |
|---|---|---|---|
| Aluminum | 7429-90-5 | 0.05 | 5.0 |
| Antimony | 7440-36-0 | 0.01 | 1.0 |
| Arsenic | 7440-38-2 | 0.01 | 1.0 |
| Barium | 7440-39-3 | 0.01 | 1.0 |
| Beryllium | 7440-41-7 | 0.004 | 0.5 |
| Boron | 7440-42-8 | 0.01 | 1.0 |
| Cadmium | 7440-43-9 | 0.005 | 0.5 |
| Calcium | 7440-70-2 | 1.0 | 100 |
| Chromium | 7440-45-1 | 0.01 | 1.0 |
| Cobalt | 7440-47-3 | 0.01 | 1.0 |
| Copper | 7440-48-4 | 0.02 | 1.0 |
| Iron | 7440-50-8 | 0.05 | 5.0 |
| Lead | 7439-89-6 | 0.01 | 1.0 |
| Magnesium | 7439-92-1 | 0.05 | 5.0 |
| Phosphorus | 7723-14-0 | 0.01 | 1.0 |
| Manganese | 7439-96-5 | 0.01 | 1.0 |

| Elements | CAS No. | Water (mg/L) | Soil (mg/Kg) |
|---|---|---|---|
| Molybdenum | 7439-98-7 | 0.01 | 1.0 |
| Nickel | 7440-02-0 | 0.01 | 1.0 |
| Potassium | 7440-09-7 | 1.0 | 100 |
| Selenium | 7782-49-2 | 0.01 | 1.0 |
| Silicon | 7440-21-3 | 0.05 | 5 |
| Silver | 7440-22-4 | 0.01 | 0.45 |
| Sodium | 7440-23-5 | 1.0 | 100 |
| Strontium | 7440-24-6 | 0.01 | 1.0 |
| Thallium | 7440-28-0 | 0.01 | 1.0 |
| Titanium | 7440-32-6 | 0.01 | 1.0 |
| Tin | 7440-31-5 | 0.01 | 1.0 |
| Vanadium | 7440-62-2 | 0.01 | 1.0 |
| Zinc | 7440-66-6 | 0.01 | 1.0 |

**2.3** A linear dynamic range has been established for each element and shall be verified annually.

**2.4** Lower limits of quantitation may be possible when a lower calibration point is included as part of the calibration curve

## 3.0 SUMMARY

**3.1** Prior to analysis, samples are prepared and digested; refer to SATL#SOP004B for preparation and digestion of samples.

**3.1.1** When samples have been properly preserved with acid and the turbidity is <1 NTU, the sample can be analyzed directly for certain metal and metalloid contaminants; with the exception of silver.



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**3.2** Digested samples in solution are introduced into the instrument through a nebulizer as an aerosol and are transported to the Plasma.

**3.3** Element specific emission spectra are generated by a radio frequency Inductively Coupled Plasma [ICP].

**3.4** Spectral line intensities are monitored by a photosensitive device, such as a camera, and are converted into digital signals and further into elemental concentrations.

**3.5** Due to the nature of the technique, background noise is corrected by measuring the background levels on either side of the elemental lines during sample analysis.

## 4.0 DEFINITIONS

**4.1** **ICP–AES** – Inductively Coupled Plasma – Atomic Emission Spectrometer.

**4.2** **Calibration Blank –** A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to auto-zero the instrument.

**4.3** **Initial Calibration Blank (ICB)** – Analyzed immediately following instrument calibration. This blank monitors instrument baseline drift as well as any contamination that may be introduced from the laboratory environment.

**4.4** **Interference Check Standard–A (ICS-A) –** High purity Standard, commercially obtained with known concentrations of Calcium, Magnesium, Iron, and Silver [See Table II, Appendix – B].

**4.5** **Interference Check Standard–AB (ICS-AB) –** High purity Standard commercially obtained with known concentrations of various elements [See Table II, Appendix – B].

**4.6** **Initial Calibration Verification (ICV)** – Analyzed immediately following instrument calibration. This verification confirms the accuracy of the instrument calibration and to monitor instrument drift and overall instrument performance.

**4.7** **Continuing Calibration Blank (CCB)** – Analyzed at prescribed intervals throughout the entire run of samples. This blank monitors instrument baseline drift as well as any contamination that may be introduced from the laboratory environment.

**4.8** **Continuing Calibration Verification (CCV)** – Analyzed at prescribed intervals throughout the entire run of samples. This verification confirms the continued accuracy of the instrument calibration and to monitor instrument drift and overall instrument performance.

**4.9** **Laboratory Reagent Blank [LRB] –** For this method, the LRB is synonymous to a method blank. An aliquot of reagent water or other blank matrix [such as analyte-free solid reagent, for soils] treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The LRB is used to determine if any method analytes or other interferences are present in the laboratory environment, reagents, or apparatus. An analyte-free reagent must be used [spiked] that mimics the matrix of the associated environmental samples.

**4.10** **Blank Spike [BS] –** Although the laboratory uses the term Blank Spike, this quality control measure is synonymous with the industry term "Laboratory Fortified Blank/Laboratory Control Sample". The BS is an aliquot of LRB spiked with a known concentration of one or more of method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. An aliquot of reagent water may be used for aqueous samples, while analyte-free solid reagent, must be used for soils.



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**4.11** **Blank Spike Duplicate [BSD] –** Although the laboratory uses the term Blank Spike/Blank Spike Duplicate, this quality control measure is synonymous with the industry terms "Laboratory Fortified Blank Duplicate/Laboratory Control Sample Duplicate." The BSD is a second aliquot or sample that is treated the same as the original sample in order to determine the precision of the analytical method.

**4.12** **Laboratory Duplicates –** Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of duplicates indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

**4.13** **Laboratory Fortified Matrix [LFM] –** The LFM is synonymous with a matrix spike**.** An aliquot of an environmental sample to which a known quantity of the method analyte is added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

**4.14** **Laboratory Fortified Matrix Duplicate [LFMD] –** A second aliquot or sample that is treated the same as the original sample in order to determine the precision of the analytical method.

**4.15** **Linear Dynamic Range [LDR] –** The concentration range over which the instrument response to an analyte is linear.

**4.16** **Method Detection Limit [MDL] –** The method detection limit (MDL) is defined as the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results. For purposes of this method, the MDL is equivalent to NELAC's Limit of Detection [LOD]. See Section 19.0 METHOD PERFORMANCE for more information regarding LOD.

**4.17** **Limit of Detection [LOD]** – See Method Detection Limit.

**4.18** **Practical Quantitation Limit [PQL]** – The lowest concentration that can be reliably measured within specified limits of precision and accuracy for a specific laboratory analytical method during routine laboratory operating conditions. The laboratory uses the NELAC term of Limit of Quantitation [LOQ] to establish the lowest Reporting Limit [RL] that a concentration of an analyte can be reported without qualification.

**4.19** **Limit of Quantitation [LOQ]** – For purposes of this method, the LOQ is equal to the low standard used for initial calibration for an analytical method, and is equal to the Reporting Limit [RL], which is the lowest limit an analyte's concentration can be reported without qualification.

## 5.0 INTERFERENCES

**5.1** Spectral interferences are caused by background emission, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

**5.1.1** Utilizing a computer correction of the raw data, which requires the monitoring and measurement of the interfering elements, can compensate for the overlap of spectral lines of elements. Any unresolved overlap of molecular band spectra may require selection of an alternate wavelength. Interferences caused by background emission, stray light from the line emission of high concentration elements can usually be compensated by a background correction adjacent to the analyte line.



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**5.2** Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump, by using an internal standard, or by using a high solids nebulizer.

**5.3** Buildup of salt from high dissolved solids at the tip of the nebulizer can affect aerosol flow rate and causing instrumental drift. This problem can be controlled by wetting the argon prior to nebulization, or by using a high solids nebulizer, or by diluting the sample.

**5.4** Fluctuations in Argon flow it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance. This may be accomplished with the use of mass flow controllers.

**5.5** Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

**5.6** The ICP is extremely sensitive to temperature fluctuations. It is important to ensure that the instrument is not in contact with direct sunlight and that the temperature in the laboratory does not fluctuate drastically during the day.

**5.7** Once the plasma is lit, it is imperative that there is always a solution flowing through the plasma. If the torch is allowed to run dry, severe damage may occur to the nebulizer/torch assembly. If the instrument is left unattended, ensure that an adequate amount of solution is available for nebulization.

## 6.0 SAFETY

**6.1** Safety glasses and laboratory coats must be worn at all times while in the laboratory. In addition gloves and a face shield or goggles must be worn when dealing with toxic, caustic, and/or flammable chemicals.

**6.2** A partial facemask should be worn when working with samples suspected to contain high levels of volatile organics, such solvents, and samples contaminated with gasoline, etc.

**6.3** All chemical compounds should be treated as potential health hazards.

**6.4** The toxicity and/or carcinogenicity of each sample will most likely not be known. Therefore, it is imperative that each sample be handled as a potential health hazard.

**6.5** The analyst should familiarize themselves with all Safety Data Sheets [SDS], safety facilities, and equipment prior to beginning this procedure.

**6.6** Please address any and all health and safety concerns to management before beginning this procedure.

## 7.0 EQUIPMENT AND SUPPLIES

**7.1** ICP-AES Thermo-Scientific, iCAP 6500, or equivalent

**7.2** Volumetric Flask, 100mL, Fisher Scientific, Catalog No. 10-209H, or Equivalent.

**7.3** Pipetter, 10-100 µL, Fisher Scientific, Catalog No. NC9929298, or Equivalent.



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**7.4** Pipetter, 100-1000 µL, Fisher Scientific, Catalog No. NC9929299, or Equivalent.
**7.5** Pipetter, 1000-5000 µL, Fisher Scientific, Catalog No. NC9012869, or Equivalent.
**7.1** Pipette Tip, 1-200µL, BVA Scientific, Catalog No. P38K-3YB, or Equivalent.
**7.2** Pipette Tip, 200-1000µL, BVA Scientific, Catalog No. P38K-15BB, or Equivalent.
**7.3** Pipette Tip, 1000-5000µL, BVA Scientific, Catalog No. P38-MPT5, or Equivalent.
**7.4** Spoonula, Fisher Scientific, Catalog No. 14-375-10, or Equivalent.
**7.5** Filter Paper, 15cm, BVA Scientific, Catalog No. F8J-150, or Equivalent.
**7.6** Pall Magnetic Filter Holder and Funnel, Hach Product No. 1352900, or Equivalent.
**7.7** Balance, Top Loading, Accurate to 0.01g, Denver Instruments, or Equivalent.
**7.8** Digestion Tubes, 68mL Capacity, Environmental Express, Catalog No. SC475.
**7.9** pH Paper, Fisher Scientific, Catalog No. 14-850-11B, or Equivalent.

## 8.0 REAGENTS AND STANDARDS

**8.1** Ultra-Pure Water, San Antonio Testing Laboratory, Wet Chemistry
**8.2** Hydrochloric Acid [HCl], 2.5L, Concentrated Trace Metal Grade, Fisher Scientific, Catalog No. A508SK212, or Equivalent
**8.3** Nitric Acid [$HNO_3$], 2.5L, Concentrated Trace Metal Grade, Fisher Scientific, Catalog No. A509SK212, or Equivalent
**8.4** Hydrogen Peroxide, 30% [$H_2O_2$], Trace Metal Grade, Fisher Scientific, Catalog No.524004, or Equivalent
**8.5** Quality Control Standard #1, AccuStandard Reference Standard, ICP Multi-Element Standard, 500mL, Catalog No. QCS-01-5, or Equivalent
**8.6** Quality Control Standard #2, AccuStandard Reference Standard, ICP Multi-Element Standard, 500mL, Catalog No. QCS-02-5, or Equivalent
**8.7** Quality Control Standard, Second Source #1, AccuStandard Reference Standard, ICP Multi-Element Standard, 500mL, Catalog No. QCS-ASL-21-5, or Equivalent
**8.8** Quality Control Standard, Second Source #2, AccuStandard Reference Standard, ICP Multi-Element Standard, 500mL, Catalog No. QCS-ASL-7-5, or Equivalent
**8.9** **Standard Stock Solutions:** Commercial stock solutions containing the compounds of interest are purchased from approved vendors at concentrations ranging from 100µg/mL to 1000µg/mL.
**8.10** **Spike Solutions:** Known amounts of the reference standards are directly spiked into the samples prior to digestion procedure. All metals except Silicon, Silver and Potassium, have a final concentration of 2µg/mL. The latter have 10µg/mL, 1µg/mL, and 20µg/mL respectively. The following table lists the amounts of spiking standards to be used in this SOP. The spiking amounts may vary based upon client project requirements.

**TABLE – B**

| Spike Solution | | Stock Conc. [µg/mL] | Final Vol. [mL] | Stock Vol. [mL] | Final Conc. [µg/mL] |
|---|---|---|---|---|---|
| Liquid Matrix | BS | 50 / 100 / 500 / 1000 | 50 | 1.0 | 1 / 2 / 10 / 20 |
| | BSD | 50 / 100 / 500 / 1000 | 50 | 1.0 | 1 / 2 / 10 / 20 |
| | LFM | 50 / 100 / 500 / 1000 | 50 | 1.0 | 1 / 2 / 10 / 20 |
| | LFMD | 50 / 100 / 500 / 1000 | 50 | 1.0 | 1 / 2 / 10 / 20 |

| | | | | | |
|---|---|---|---|---|---|
| Solid Matrix | BS | 50 / 100 / 500 / 1000 | 50 | 1.0 | 1 / 2 / 10 / 20 |
| | BSD | 50 / 100 / 500 / 1000 | 50 | 1.0 | 1 / 2 / 10 / 20 |
| | LFM | 50 / 100 / 500 / 1000 | 50 | 1.0 | 1 / 2 / 10 / 20 |
| | LFMD | 50 / 100 / 500 / 1000 | 50 | 1.0 | 1 / 2 / 10 / 20 |

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

### 9.1 Sample Collection

#### 9.1.1 Aqueous Samples

**9.1.1.1** Aqueous/liquid samples are collected in plastic or glass bottles. At least 250mL of sample is required for digestion and analysis.

#### 9.1.2 Solid Samples

**9.1.2.1** Solid [soils and sediment] samples are collected in 4oz wide mouth borosilicate glass jars or plastic. A minimum of 200 grams of sample is required for digestion & analysis of metals.

**9.1.2.2** Solid samples must be homogenized in the field and further homogenized in the laboratory prior to digestion and analysis.

#### 9.1.3 Waste Characterization Samples

**9.1.3.1** Waste characterization samples are collected similar to the solid samples and transported to the laboratory for analysis.

### 9.2 Preservation

#### 9.2.1 Aqueous Samples

**9.2.1.1** Samples for dissolved metals need to be filtered in the field and pH adjusted to <2.

**9.2.1.1.1** Client may request that the laboratory perform the filtration; sample is filtered using filter paper (7.5) with Pall magnetic filter holder and funnel (7.6).

**9.2.1.2** Aqueous samples are preserved with approximately 2mL of 1:1–$HNO_3$: $H_2O$ to a pH of 2.0 or less and it is recommended that the samples be kept on ice during transport and refrigerated until digestion and analysis.

#### 9.2.2 Solid Samples

**9.2.2.1** Solid samples do not required preservation with acid but recommended to be kept on ice after collection to prevent loss of extremely volatile organics.

### 9.3 Holding Times

#### 9.3.1 Aqueous Samples

**9.3.1.1** Aqueous samples preserved with acid as described in 9.2.1.1 have a holding time of 180 days from the time of collection until the time of analysis.

#### 9.3.2 Solid Samples

**9.3.2.1** Solid and Waste samples also have a holding time of 180 days from the time of collection until the time of analysis.

## 10.0 STORAGE

**10.1** Aqueous samples are stored until the time of analysis in a refrigerator at >0°C but ≤ 6°C.

**10.2** Solid samples are stored at >0°C but ≤ 6°C in a refrigerator until the time of digestion and analysis.



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## 11.0 SAMPLE IDENTIFICATION

**11.1** Samples are received from Sample Receiving with an In-House Chain of Custody form generated from the Laboratory Information Management System [LIMS]. This includes client identification, sample number, and test to be performed.

**11.2** Each sample is assigned a unique number and a container number if more than one container is received.

## 12.0 CALIBRATION AND STANDARDIZATION

**12.1** **BALANCES:** All balances used for this procedure must be checked with S-Class weights and monitored to be within tolerance limits on each day of use.

**12.2** **PIPETTES:** All mechanical pipettes must be checked using a calibrated analytical balance. Checks are to be performed quarterly. Pipette checks are to be noted in the Pipette Calibration electronic spreadsheet.

**12.3** The calibration of the instrument (iCAP) includes the analysis of a Calibration Blank, a Low Standard, a Mid Standard, and a High Standard, followed by the analysis of additional QC standards.

**12.4** The typical analytical sequence includes the calibration, ICV, ICB, ICS-AB, ICS-A, Rinse Blank, Samples 1-10, Rinse Blank, CCV, CCB, and Samples 11-20, Rinse Blank CCV, CCB, any additional samples, and finally an ending CCV and CCB.

**12.5** Prior to the analysis of samples the ICP instrument must be calibrated each day of use. The calibration curve consists of: Calibration Blank, Low Standard (0.01 ppm), Mid Standard (0.5 ppm), and High Standard (2 ppm). Such calibration standards are prepared according to table III, found in Appendix B (at the end of this SOP).

**12.6** The calibration is verified by using a standard spiked at 0.5 ppm from a second source (ICV) and interferences are monitored by running an ICS-A and ICA-AB, which are spiked with known interferants and analytes.

**12.7** Correlation coefficient of each of the elements of interest must be $\geq 0.995$. All other standards follow the acceptance criteria cited in Table IV.

**12.8** Calculate the Relative Standard Error (%RSE) of the calibration curve for analytes with linear or quadratic fits. Determine the %RSE using the equation below.

$$\%\,RSE = 100 \times \sqrt{\sum_{i=1}^{n}\left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where,

$x_i$ = True value for the calibration standard
$x'_i$ = Measured concentration of the calibration standard
n = Number of calibration points
p = Number of terms in the fitting equation
(Average = 1, Linear = 2, Quadratic = 3)



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**12.9** Coefficient of determination must be >0.920 (which approximately corresponds to the 15% RSD limit set forth in the reference method). If this cannot be achieved, the calibration is unacceptable and recalibration is necessary after remedial action to correct the problem.

**12.10** Calculate the Relative Error (%RE) for those analytes are calibrated using linear or quadratic curve fits and determine the coefficient of determination using the following equation.

$$\%Relative\ Error = \frac{x'_i - x_i}{x_i} \times 100$$

Where,

$x_i$ = True value for the calibration standard
$x'_i$ = Measured concentration of the calibration standard

**12.11** The relative error percent must be calculated for two of the calibration levels, i.e., the low calibration standard and the mid-point calibration standard. The acceptance criteria for low standard is 30% and the mid-point standard is 15%.

**12.12 Initial/Continuing calibration verification**

**12.12.1** All initial instrument calibrations must be verified with an ICV. The ICV must be prepared from a standard obtained from a second manufacturer or lot if the lot can be demonstrated from the manufacturer as prepared independently from other lots. The ICV recovery must be within ±5% and ±10% of the stated concentration (for EPA 200.7 and for EPA 6010B respectively).

**12.12.2** Calibration of the ICP-AES system is verified by analyzing a continuing calibration verification standard [CCV]. If the CCV standard meets acceptance criteria of ≤10% Difference [%D] for elements of interest, then the initial calibration is deemed valid.

**12.12.3** If the CCV fails to meet the acceptance criteria, refer to Appendix B, Table-IV for recommended corrective actions.

**12.12.4** If the ICV and CCV fail to meet the criteria in the above-mentioned tables, system check/ maintenance may be required as described in the next section.

**12.13 Recommended system maintenance**

**12.13.1** In cases where the initial calibration does not meet the acceptance criteria or the CCV does not meet the %D criteria, system maintenance is required. A short list of the remedial actions is given below:

a. Check the Argon gas flow to the ICP-AES system.
b. Clean and/or replace the nebulizer.
c. Check all pressure gauges and bulk gas supply.
d. Clean and/or replace the Plasma Torch.
e. Check and replace all pump tubing once a week or as necessary.
f. Flush all tubing including the auto-sampler tubing.
g. Analyze reagent water blanks containing 2% $HNO_3$.
h. If none of these maintenance tasks resolve the problems, contact the manufacturer for either technical help or service call.



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## 13.0 PROCEDURE

### 13.1 Instrument preparation

**13.1.1** Prior to any analysis check Argon and Nitrogen bulk tank levels and pressure. The gauges located in the metals room should read about 80-85psi.

**13.1.2** Be sure that waste collection containers are not near capacity. If so, dispose of the waste before proceeding.

**13.1.3** Check that all pump tubing is attached and in good condition. It may be necessary to replace the tubing.

### 13.2 Instrument Operation

**13.2.1** From the computer desktop, click the iTEVA icon to open instrument software. This will initiate the instrument settings.

**13.2.2** In the iTEVA Software window, click on the plasma icon [candle like] at the bottom of the screen to open the Plasma Control Panel window. Allow the instrument to warm up for 15 minutes prior to the start of calibration.

**13.2.3** Once the instrument has been started up, ignite the plasma by clicking on the 'Plasma on' located at the bottom of the plasma status screen and allow the instrument to warm up for at least 15 minutes prior to the start of calibration.

**13.2.4** On the Plasma Control Panel, the instrument parameters should be set as below (may be adjusted as needed for optimal performance of the instrument:

| | |
|---|---|
| RF Power | 1150 W |
| Pump Rate | 50 RPM |
| Auxiliary Gas Flow | 0.5 L/min |
| Nebulizer Gas Flow | 0.95 L/min |
| Coolant Gas Flow | 12 L/min |
| Purge Gas Flow | Normal |

### 13.3 Sample preparation

**13.3.1** All samples except those that are analyzed only for dissolved metals or direct analysis are digested prior to analysis. Refer to SATL#SOP004B for sample preparation. For drinking water samples, check the turbidity of the preserved sample and record results in the logbook as either >1.0 or <1.0. If turbidity is <1.0 NTU, direct analysis can be performed. If turbidity is >1.0 NTU, samples will be digested prior to analysis.

**13.3.1.1** ***Soils:*** Must be centrifuged for 10 minutes, filtered, or allowed to settle overnight.

**13.3.1.2** ***Liquids:*** Allow settling overnight if suspended solids are present

**13.3.1.3** Samples may be filtered if necessary, with a Whatman 42 filter or equivalent.

### 13.4 Auto-Sampler and Sample Sequence

**13.4.1** From the iTEVA control center, click on the 'Analyst' icon. Choose a desired method by clicking on the method name for the Sequence, click 'OK'.

**13.4.2** Click on the 'Sequence' tab located on the lower left-hand side. Then, go to the upper left-hand side and click on 'Auto-session'. From the drop-down menu click on 'New Autosampler'.

**13.4.3** Once the New Automation Session opens up, click on the 'New' button, this will prompt the new Sequence screen. Once there, enter the number of samples to be added to the sequence and



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click 'OK'. Click 'OK" on the previous screen (New Automation Sequence) so as to close it as well.

**13.4.4** Click on the grid-like button located at the upper-center of the screen, the workspace will now be in 'List-view'. On this screen, analyst may name the samples to be run by simply clicking in each sample line and typing in the sample identification with any other pertinent information.

**13.4.5** After entering the sample run sequence, click on the 'Auto-session' tab and save the sequence (usually the date of the run).

**13.4.6** Right-click on the newly created sequence located on the left side of the screen and click 'Auto-locate all'. This will allow the autosampler to find each sample location.

**13.4.7** Once all Standards, QC standards, and Samples have been loaded onto the appropriate racks on the autosampler, the sequence can be started by simply clicking 'Play' button (yellow-side ways triangle) found on the upper portion of the workspace.

**13.4.8** The instrument begins by performing the calibration, followed by running the QC check standards, and the samples as well as running a CCV and CCB after the analysis of every ten samples and the ending CCV and CCB.

**13.4.9** Click the "Auto Sampler Rack" icon to open the sample setup diagram.

**13.4.10** If the instrument continues to run after work hours, then select "Shut down Plasma" option to shut the instrument down at the end of sample analysis.

### 13.5 Editing a Sequence

**13.5.1** The analyst may edit the sequence by going to 'List View' and using the 'test-tube+' for adding and the 'test-tube-' for deleting samples. Delete the sample by highlighting the sample row and click 'test-tube-'.

**13.5.2** The analyst may also opt to set runtime and actions such as: sound an audible alarm once the sequence has been run or to set the instrument to shut-down by extinguishing the plasma following the completion of a sequence.

**13.5.3** To set the shutdown, right-click on the sequence on the left portion of the screen and click 'Modify'. Under the conclusions heading click on the desired action and save the changes.

### 13.6 Pause and Stop Actions During a Sequence Run

**13.6.1** To pause the sequence, such as when more samples need to be added or the order of the run is to be altered, click on the 'Pause' tab (two yellow bars) in the upper center of the list-view screen. If the instrument is running a sample at that moment, the analysis of that sample will be completed and the auto-sampler will go into pause mode immediately after that.

**13.6.2** Once the changes have been made and saved, click on the 'pause' button once more to continue running the sequence.

**13.6.3** In order to stop a run, locate the 'halt autosession' button located in the upper center (Yellow Square) of the screen and click on it. This will stop the analysis and return the sipper to the home position. To abort a sequence, click on the abort autosession button (red square).

**13.6.4** To resume the analysis click on '+' button to the left of the sequence name, then click on the '+' button to the left of the Method name, click on the '+' button to the left of the samples. Once, the list of samples is displayed, right-click on the sample at which you wish to start running the sequence.



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**13.6.5** Verify that the instrument calibration is valid and subsequent QC samples meet the acceptance criteria. Sample data may be released with qualification on the analytical report for any QC failures observed.

**13.6.6** Review the instrument data and export into LIMS system for reporting.

## 14.0 DATA ANALYSIS AND CALCULATIONS

### 14.1 *Data Analysis:*

**14.1.1** Percent Relative Standard Deviation:

$$\%\text{RSD} = \frac{\text{SD}}{\overline{\text{RF}}} \times 100$$

Where:

SD = Standard Deviation; $\overline{\text{RF}}$ = Average Response Factor

### 14.2 *Calculation of the Unknowns:*

**14.2.1** Concentration of each element in a **Water** Sample:

$$\text{Concentration (mg/L)} = \left[(I_R) \times (\text{DF})\right]$$

Where:

DF = Dilution Factor
$I_R$ = Result from Instrument analysis in [µg/mL]

**14.2.2** Concentration of each analyte in a **Soil/Waste** Sample:
(Sediment and Soil Sludge Based On Dry Wt.; Waste Based On Wet Wt.)

$$\text{Concentration (mg/kg)} = \frac{(I_R) \times (\text{FV}) \times (\text{DF})}{\text{Ws} \times \text{D}}$$

Where:

$I_R$ = Result from Instrument analysis in [µg/mL]
FV = Final digestate volume [mL]
$W_s$ = Weight Of Sample Extracted [g]
D = (% Dry Weight of Sample/100) or 1 For Wet Weight Basis

**14.2.3** LFM (Matrix Spike) Recovery

$$\text{Matrix Spike Recovery} = \frac{\text{MSR} - \text{SR}}{\text{SA}} \times 100$$

Where:

MSR = Element Spike from Sample Result
SR = Element from Sample Result
SA = Spike Added to the Sample

**14.2.4** Relative Percent Difference:



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$$RPD = \left| \frac{MSR - MSDR}{\left( \frac{MSR + MSDR}{2} \right)} \right| \times 100$$

Where:

RPD = Relative Percent Difference.
MSR = LFM [Matrix Spike] Recovery.
MSDR = LFMD [Matrix Spike Duplicate] Recovery.

## 15.0 QUALITY CONTROL

**15.1** Acceptance limits for quality control measures are listed in Table IV.

**15.2** Initial calibration is verified initially with a second source ICV and after every 10 samples and at the end of the run using the primary source CCV versus acceptance criteria provided in Table IV.

***Note:*** *A second source ICV may also be used after every ten samples to verify calibration.*

**15.2.1** The instrument's Linear Dynamic Range must be established as per the manufacturer recommendations.

**15.3** Each batch of samples requires the analysis of a LRB, BS, BSD, LFM and LFMD. LFM/LFMD (however named) must be performed with each batch of samples regardless of matrix type, at a frequency of 10% for aqueous samples, and 5% for soils. Sample duplicates are optional based on client requests.

**15.4** Recommended matrix interference checks for LFM/LFMD and Sample and Sample Dilution

**15.4.1** ***Liquids***

**15.4.1.1** ***Dilution test:*** If an analyte concentration is above the high standard for the calibration curve but within the LDR, the analyte may be reported with data qualifier or diluted and re-analyzed. If the analyte concentration is sufficiently high (by a factor of 50 above the instrument detection limit in the original solution but <90% of the linear limit), an analysis of a 1:5 dilution should agree (after correction for the fivefold dilution) within ±10% of the original determination. If not, a chemical or physical interference effect should be suspected and the associated data flagged accordingly.

***Example:*** *If the concentration of Arsenic in a sample is 0.5mg/L at the instrument level [this is equal to a factor of 50 above the IDL (0.01mg/L, for example)], in the dilution analysis the concentration of Arsenic should fall between 0.45mg/L and 0.55mg/L [after taking into account a dilution factor or 5×] at the instrument level. If not an interference effect, either physical or chemical is suspected.*

**15.4.1.2** ***Post Digestion Spike:*** An analyte(s) standard of known concentration added to a portion of a digested and prepared sample, or its dilution, should be recovered to within 85% to 115% of the known value.

**15.4.1.3** The analyte(s) addition should produce a minimum level of 20 times and a maximum of 100 times the instrument detection limit. If recovery of the analyte(s) is not within the



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specified limits, a matrix effect should be suspected, and the associated data flagged accordingly.

> ***Example:*** *The concentration of Lead in a sample is 0.5mg/L at the instrument level [this equals a factor of 50 above the IDL (0.01mg/L)], and a spike of 2mg/L is added to the sample. The recovery of in the post digestion spike should fall between 2.125mg/L and 2.875mg/L at the instrument level. If not a matrix effect is suspected and the data is flagged accordingly on the report.*

**15.4.2** ***Solids***

**15.4.2.1** ***Dilution test:*** If an analyte concentration is above the high standard for the calibration curve but within the LDR, the analyte may be diluted and re-analyzed or reported with qualification. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

> ***Example:*** *The concentration of Arsenic in a sample is 0.5mg/L at the instrument level [this equals to a factor of 25 above the IDL (0.02mg/L for example)], in the dilution analysis the concentration of Arsenic should fall between 0.45mg/L and 0.55mg/L [after taking into account a dilution factor or 5×] at the instrument level. If not an interference effect, either physical or chemical is suspected.*

**15.4.3** ***Post Digestion Spike***: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike added should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. For instance, if the instrument detection limit for Lead is 0.02mg/L then the spike added should be between 0.2mg/L to 2mg/L. If the spike recovered is not within the specified limits of 75% – 125%, a matrix effect is suspected.

> ***Example:*** *The concentration of Lead in a sample is 0.2mg/L at the instrument level [this equals a factor of 50 above the IDL (0.02mg/L for example)], and a spike of 2mg/L is added to the sample. The recovery of the post digestion spike should fall between 1.65mg/L and 2.75mg/L at the instrument level. If not a matrix effect is suspected and the data is flagged accordingly on the report.*

**15.4.4** All pertinent information such as: calibration standards/equipment identification numbers, unique identification numbers for stock and working standards/solutions, and balance/thermometer serial numbers must be recorded in log books/bench sheets.

> *Note: All working calibration standards and solutions prepared daily must also be assigned a unique identification number in Element.*

## 16.0 ACCEPTANCE CRITERIA

**16.1** Refer to Appendix B, Table IV for acceptance criteria.



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**16.2** The acceptance limits for Demonstration of Capability (DOC) by this method are %RSD <15 (precision) of 4 QC replicates, and an average recovery range of 85-115% (accuracy) of the true concentration, for water. For soil, %RSD <20, and an average recovery range of 80-120%. DOCs must take into account all sample preparation steps, must be performed per analyst, per matrix, and must be prepared from a secondary source.

## 17.0 CORRECTIVE ACTIONS FOR NON-CONFORMANCE DATA

**17.1** Refer to Appendix B, Table IV for corrective actions.

**17.2** When QC does not fall within the acceptable range, the QC must be reanalyzed, along with the associated samples. If the QC continues to fail, identify the root of the problem and correct. A Corrective Action Form may be required per the determination of the Quality Assurance Manager.

## 18.0 HANDLING NON-CONFORMANCE DATA

**18.1** Non-conformance is monitored and is resolved by classifying into categories such as system based, method based, preparative method based, etc., and are resolved once the problem areas are identified.

## 19.0 METHOD PERFORMANCE

**19.1** The minimum level of quantitation is equivalent to NELAC's Limit of Quantitation [LOQ] and must be verified at least annually with a second source material as compared to the initial calibration.

**19.1.1** The LOQ is equal to the low standard used for initial calibration.

**19.2** A method detection limit study is performed, initially and verified quarterly thereafter for analyte that is listed in this method.

**19.3** During the beginning of each quarter, two replicate samples of organic free reagent water are spiked with a known amount of target analytes at the concentration used in the initial determination of the MDL and analyzed on the ICP/AES.

**19.4** If any analytes are repeatedly not detected in the quarterly spiked sample analyses, or do not meet the qualitative identification criteria of the method, then this is an indication that the spiking level is not high enough and should be adjusted.

**19.5** Prepare and analyze seven spike replicates and seven method blanks on at least three different days carried out through sample preparation steps. Existing routine method blanks can be used for this study.

**19.6** A minimum of seven MDL replicate samples and seven method blanks are used to calculate the MDL values. For purposes of this method, the MDL is equivalent to TNI's Limit of Detection (LOD).

Calculate the $MDL_S$ (MDL spiked samples) value using the following formula:

$$MDL_s = t_{[n-1,\ 1-\infty = 0.99]}\ S_s$$

Where,

$t_{[n-1,\ 1-\infty = 0.99]}$ = Student's t value for the 99% confidence level with n-1 degrees of freedom,

n = number of replicates.



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$S_s$ = the standard deviation of the replicate analyses.

Calculate the $MDL_B$ (MDL blank samples) values using the following formula:

$$MDL = t_{[n-1,\ 1\text{-alpha} = 0.99]}\ S_b$$

Where,

$t_{[n-1,\ 1\text{-alpha} = 0.99]}$ = Student's t value for the 99% confidence level with n-1 degrees of freedom,
n = number of replicates.
$S_b$ = the standard deviation of the replicate method blank sample analyses.

| Number of Replicates | Degrees (degrees of freedom) | $t_{(n-1,\ 0.99)}$ |
|---|---|---|
| 7 | 6 | 3.143 |
| 8 | 7 | 2.998 |
| 9 | 8 | 2.896 |
| 10 | 9 | 2.821 |
| 11 | 10 | 2.764 |

**19.7** Current MDL values for method analytes in this SOP can be found in the SATLMDL.xls spreadsheet.

## 20.0 POLLUTION PREVENTION

**20.1** No solvents are utilized in this method. However, various acids are used throughout the method and are disposed of by diluting with Di-ionized water.

**20.2** Solutions used to prepare calibration standards are purchased only at levels required to prepare dilute working standards and at the smallest possible amounts possible.

**20.3** Only the amount of chemical that is actually needed is purchased, to eliminate the pollution and cost of disposal later.

## 21.0 WASTE MANAGEMENT

**21.1** Toxic waste must never be disposed of down the drain.

**21.2** Waste generated from sample analysis must be segregated if the process knowledge indicates the presence of any of the hazardous components listed in Table–1, 40 CFR 261.24 and exceed the limits set in the table.

**21.3** When disposing samples the analyst must follow current revision of the "Laboratory Waste Handling and Disposal" SOP (SATL#007G) for detailed disposal procedures.

**21.4** All chemicals and containers must be properly identified and labeled at all times to eliminate ambiguity and cost of disposal of unknowns. If an unknown chemical or container is discovered, label it as 'unknown' and attach a note detailing any information about what the chemical may be, what test it may have been used for, and where it was found. If you find an unlabeled chemical that



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has crystallized or there is any other indication that it may be unstable, notify management immediately.

**21.5** Generally, empty chemical containers are not considered hazardous waste. Check with management if one such container is found and in doubt. To dispose of the container in the regular trash the container must be completely empty and triple.

**21.6** The waste drums are picked up upon notification and a copy of the report is submitted to the waste management company.

## 22.0 REFERENCES

**22.1** "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma – Atomic Emission Spectrometry," Method 200.7, Revision 4.4, May 1994. U.S. Environmental Protection Agency.

**22.2** "Inductively Coupled Plasma Atomic Emission Spectroscopy," Method 6010B, Revision 2, December 1996, SW-846 Test Methods for Evaluating Solid Waste, U.S. Environmental Protection Agency.

**22.3** Operational Manual, TJA ICP–AES, Model # ICAP. ThermoElectron Corporation.

**22.4** The TNI Standard, 2016.

## 23.0 REVISION HISTORY

**23.1** New revision of the method.

**23.2** Revision 2 from Revision 1: changes stemming from an annual review, and the most recent TCEQ on-site assessment.

**23.3** Revised section 12.0 and 13.0.

**23.4** Annual revision 2012, Rev 2.0.2 – No changes made

**23.5** Annual Revision 2014, Rev 2.1.0 – Updated Tables in Appendix B (calibration curve standard preparation), revised sections: 4.0, 6.0, 12.0, 13.0, and 15.0.



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SAN ANTONIO TESTING LABORATORY, INC.

**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF TOTAL METALS BY ICP – AES [EPA 200.7 & 6010B]**

# APPENDIX A

**SOP History and Version Control**

| Version | Date of Reviewed/Revision | Review/Revision Approved by | Brief Description |
|---|---|---|---|
| 2.2 | 11/23/2015 | M. Bernard | Addition of Appendix A to reflect SOP history and version control. Revised to clarify ICS QC requirements per reference method. Change Appendix I to Appendix B. |
| 2.3 | 07/08/16 | M. Bernard | Revision of title page and clarification on procedure for direct analysis (13.3.1). |
| 2.4 | 2/27/17 | M. Bernard | Revision of procedure prior to assessment. |
| 3.0 | 06/15/2017 | M. Bernard | Biennial review; revision of waste management protocol. |
| 3.1 | 10/31/2018 | M. Bernard | Revised to update internal standard protocol and Appendix B QC acceptance criteria. |
| 4.0 | 04/15/2019 | M. Bernard | Biennial review; general grammatical corrections. |
| 5.0 | 03/05/2021 | A.Rosecrance | Biennial review; update title page; change MSDS to SDS. |
| 5.1 | 09/13/2021 | C. Morrow | Revised the following:<br>Update title page.<br>Section 2 – Update quantitation limit requirements.<br>Section 4 – Update definitions.<br>Section 19 – Update MDL/LOD procedure. |



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# APPENDIX – B

## 1. List of Tables

a. **Table I – Typical Wavelengths** used [nm] (other wavelengths may be used in order to optimize response and adhere to required quality control acceptance limits).

| Element | λ | Element | λ | Element | λ | Element | λ |
|---|---|---|---|---|---|---|---|
| Ag | 328.068 | Co | 228.616 | Mo | 202.030 | Sn | 189.989 |
| Al | 308.215 | Cr | 267.716 | Na | 588.995 | Sr | 421.552 |
| As | 189.042 | Cu | 324.754 | Ni | 231.604 | Ti | 334.941 |
| Ba | 493.409 | Fe | 259.940 | Pb | 220.353 | Tl | 190.864 |
| Be | 313.042 | K | 766.490 | Sb | 206.833 | V | 292.402 |
| Ca | 315.887 | Mg | 279.079 | Se | 196.090 | Zn | 213.856 |
| Cd | 226.502 | Mn | 257.610 | Si | 251.612 | Y* | 224.306 |
| B | 249.678 | P | 177.4 | | | Y* | 371.030 |
| | | | | | | In* | 224.606 |

* Yittrium – Internal Standard. *Indium – Internal Standard.

### b. Table II – ICS-A and ICS-AB Solution Elements and Concentrations

| Elements | ICS-A (PPM) | ICS-AB (mg/L) | Elements | ICS-A (PPM) | ICS-AB (mg/L) |
|---|---|---|---|---|---|
| Aluminum | 250 | 250 | Manganese | 0 | 0.05 |
| Antimony | 0 | 0 | Molybdenum | 0 | 0 |
| Arsenic | 0 | 0 | Nickel | 0 | 0.10 |
| Barium | 0 | 0.05 | Potassium | 0 | 0 |
| Beryllium | 0 | 0.05 | Selenium | 0 | 0 |
| Cadmium | 0 | 0.10 | Silver | 0 | 0.10 |
| Calcium | 250 | 250 | Sodium | 0 | 0 |
| Chromium | 0 | 0.05 | Strontium | 0 | 0 |
| Cobalt | 0 | 0.05 | Thallium | 0 | 0 |
| Copper | 0 | 0.05 | Tin | 0 | 0 |
| Iron | 100 | 100 | Vanadium | 0 | 0.05 |
| Lead | 0 | 0.10 | Zinc | 0 | 0.10 |
| Magnesium | 250 | 250 | B | 250 | - |



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### c. Table III: Calibration Curve Standards Preparation

| | |
|---|---|
| Calibration Blank | 50 mL 4%/2% ($HNO_3$/HCl) Rinse water |
| Standard 1 (Low Standard) | 250 µL Standard 3 + 49.75 mL 4%/2% ($HNO_3$/HCl) Rinse water |
| Standard 2 (Mid Standard) | 250 µL of each ICP stock standards + 49.5 mL 4%/2% ($HNO_3$/HCl) Rinse water |
| Standard 3 (High Standard) | 1 mL of each ICP stock standards + 48 mL 4%/2% ($HNO_3$/HCl) Rinse water |
| ICV | 1 mL of each ICP (second source) stock standards + 48 mL 4%/2% ($HNO_3$/HCl) Rinse water |
| CCB | 50 mL 4%/2% ($HNO_3$/HCl) Rinse water |
| ICS-A | 2.5 mL of Primary Interferants Standard + 47.5 mL 50 mL 4%/2% ($HNO_3$/HCl) Rinse water |
| ICS-AB | 2.5 mL of Primary Interferants Standard + 50 µL Primary Analytes Standard + 47.45 mL 4%/2% ($HNO_3$/HCl) Rinse water |
| CCV | 250 µL of each ICP stock standards + 49.5 mL 4%/2% ($HNO_3$/HCl) Rinse water |

### d. Table IV – Quality Control Acceptance Criteria

#### i. Liquids (EPA 200.7)

| QC Standard | Acceptance Limits | Corrective Action |
|---|---|---|
| Initial Calibration prior to sample analysis: High Std., Low Std. and Calibration Blk. | Correlation coefficient of ≥ 0.995. | Determine root cause of problem and re-calibrate. |
| Calibration Blank | ≤ IDL and > lower 3 Sigma of Calibration blank data | Cross Contamination – Check for possible reagent contamination and replace and re-analyze the batch of samples. |
| LRB | ≤ 10% the analyte's conc. in associated samples, or ≤ 2.2 × the MDL | If not met, re-digest and re-analyze. |
| ICB & CCB | ≤ IDL and > lower 3 Sigma of Calibration blank data | Cross Contamination – Check for possible reagent contamination and replace and re-analyze the batch of samples. |
| ICV<br>CCV | (ICV) 95-105%<br>(CCV) 90-110% | Re-analyze ICV/CCV. If still fail to meet the acceptance criteria, then prepare fresh standards and re-analyze. |
| ICS-A, ICS-AB | ± 10% of actual conc. | Identify issue and correct, then recalibrate, and reanalyze all associated samples. |
| BS, BSD | 85-115%; ≤ 20% RPD | Re-analyze sample batch; Further failure warrants re-digestion and reanalysis. |



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| | | |
|---|---|---|
| LFM, LFMD | 75-125%; ≤ 20% RPD every 10 samples | Analyze post-digestion spike as per section 15.4 of this SOP. |
| Post Digestion Spike | 85-115% | Flag data accordingly, If section 15.4 is indicative of matrix problems. |
| Serial Dilution | 90-110% | Flag data accordingly, If section 15.4 is indicative of matrix problems. |

### ii. Solids/TCLP/SPLP (EPA 6010B)

| QC Standard | Acceptance Limits | Corrective Action |
|---|---|---|
| Initial Calibration prior to sample analysis: High Std., Low Std. and Calibration Blk. | Correlation coefficient of ≥ 0.995. | Determine root cause of problem and re-calibrate. |
| Calibration Blank & ICB | ≤ IDL and > lower 3 Sigma of Calibration blank data | Cross Contamination – Check for possible reagent contamination and replace and re-analyze the batch of samples. |
| LRB | ≤ 10% the analyte's conc. in associated samples, or ≤ 2.2 × the MDL | If not met, re-digest and re-analyze. |
| CCB | ≤ ½ R.L | Cross Contamination – Check for possible reagent contamination and replace and re-analyze the batch of samples. |
| ICV<br>CCV | (ICV) 90-110%<br>(CCV) 90-110% | Re-analyze ICV/CCV. If still fail to meet the acceptance criteria, then prepare fresh standards and re-analyze. |
| ICS-A, ICS-AB | ± 10% of actual conc. | Identify issue and correct, then recalibrate, and reanalyze all associated samples. |
| BS, BSD | 85-115%; ≤ 20% RPD | Re-analyze sample batch; Further failure warrants re-digestion and reanalysis. |
| LFM, LFMD | 75-125%; ≤ 20% RPD, every 20 samples | Analyze post-digestion spike as per section 15.4 of this SOP. |
| Post Digestion Spike | 75-125% | Flag data accordingly, If section 15.4 is indicative of matrix problems. |
| Serial Dilution | 90-110% | Flag data accordingly, If section 15.4 is indicative of matrix problems. |



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### e. Example Analysis Sequence

* Calibration Blank
* Low Standard (*equal to the concentration of the LOQ and*
* Mid Standard
* High Standard
ICV
ICB
ICS-AB
ICS-A
Rinse Blank
LRB
BS
BSD
Sample 1
Sample Duplicate [for liquid samples]
Sample 1 LFM
Sample 1 LFMD [for solid and TCLP/SPLP samples]
Sample 1 A (Post Digestion Spike)
Sample 1 DL (Serial Dilution)
Sample 2 Sample 10
Rinse Blank
CCV
CCB
Next 10 Samples Rinse Blank
CCV – End of Analysis
CCB – End of Analysis

### f. Table V – Inter-Elemental Spectral Interferences

| Interferants at 100mg/L level. | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | WL (nm) | Interferant* | | | | | | | Analyte | WL (nm) | Interferant* | | | | | | | |
| Ag | 328.068 | Ce, | Ti, | Mn | | | | | Mg | 279.079 | Ce | | | | | | | |
| Al | 308.215 | V, | Mo, | Ce, | Mn | | | | Mn | 257.610 | Ce | | | | | | | |
| As | 193.759 | V, | Al, | Co, | Fe, | Ni | | | Mo | 203.844 | Ce | | | | | | | |
| B | 249.678 | None | | | | | | | Na | 588.995 | None | | | | | | | |
| Ba | 493.409 | None | | | | | | | Ni | 231.604 | Co, | Tl | | | | | | |
| Be | 313.042 | V, | Ce | | | | | | P | 214.914 | Cu, | Mo | | | | | | |
| Ca | 315.887 | Co, | Mo, | Ce | | | | | Pb | 220.353 | Co, | Al, | Ce, | Cu, | Ni, | Ti, | Fe | |
| Cd | 226.502 | Ni, | Ti, | Fe, | Ce | | | | Sb | 206.833 | Cr, | Mo, | Sn, | Ti, | Ce, | Fe | | |



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**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF TOTAL METALS BY ICP – AES [EPA 200.7 & 6010B]**

| | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | 413.765 | None | | | | | | | Se | 196.099 | Fe | | | | | | |
| Co | 228.616 | Ti, | Ba, | Cd, | Ni, | Cr, | Mo, | Ce | SiO | 251.611 | None | 2 | | | | | |
| Cr | 205.552 | Be, | Mo, | Ni | | | | | Sn | 189.980 | Mo | Ti | Fe | Mn | Si | | |
| Cu | 324.754 | Mo, | Ti | | | | | | Sr | 421.552 | None | | | | | | |
| Fe | 259.940 | None | | | | | | | Tl | 190.864 | Ti, | Mo, | Co, | Ce, | Al, | V, | Mn |
| Hg | 194.227 | V, | Mo | | | | | | Ti | 334.941 | None | | | | | | |
| K | 766.491 | None | | | | | | | V | 292.402 | Mo, | Ti, | Cr, | Fe, | Ce | | |
| Li | 670.784 | None | | | | | | | Zn | 213.856 | Ni, | Cu, | Fe | | | | |



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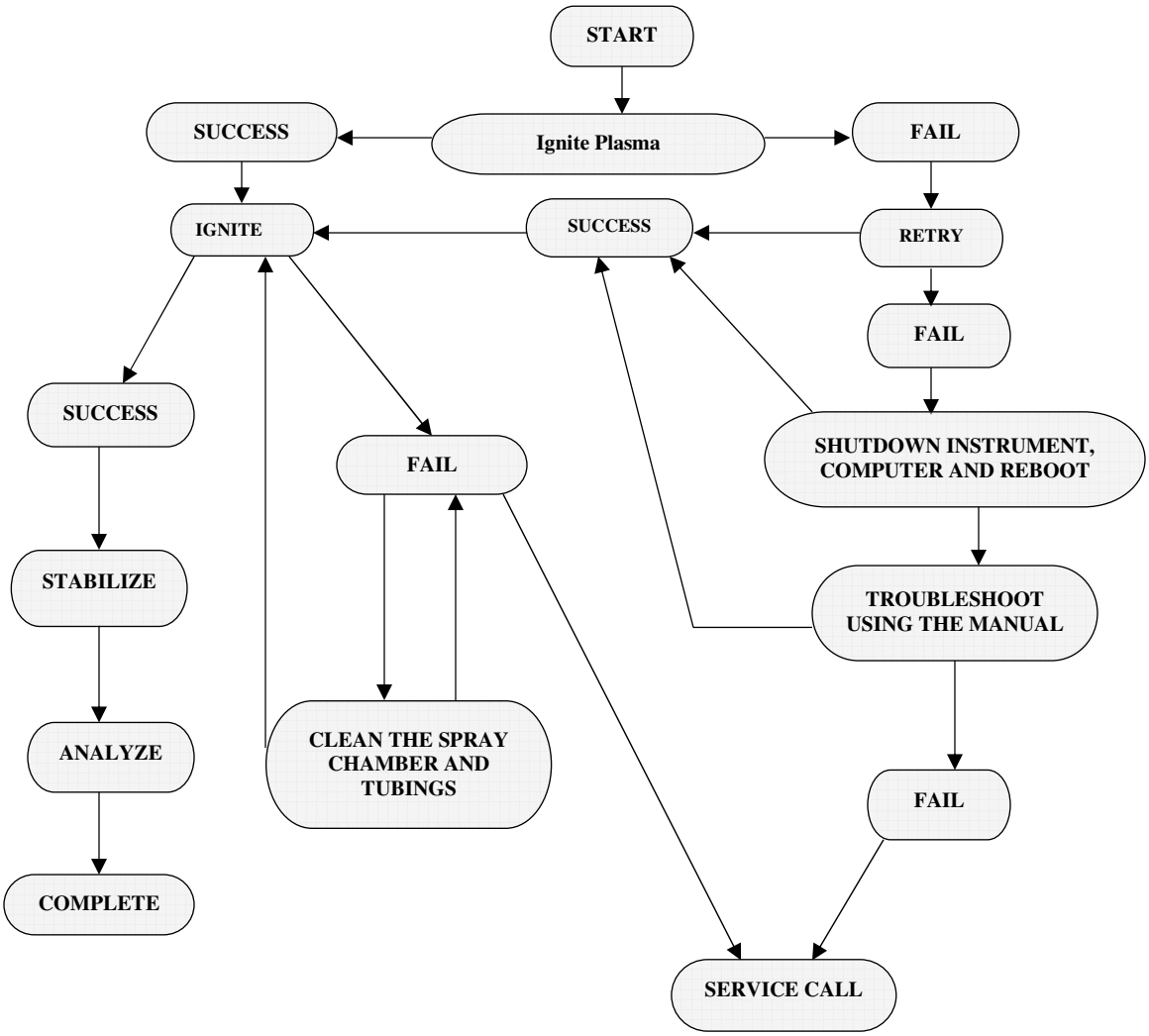
**g. Flow Chart – I**



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# STANDARD OPERATING PROCEDURE

*Title*

## Analysis of Ammonia [Nitrogen]

*Method No.:*

**SM4500-$NH_3$–N [B & C] (23rd Edition, 2017) & EPA 350.2**

*Matrix/Matrices:*

**Liquid/Solid**

*Document Control Number/Revision Number*

**SOP006A/Revision 5.1**

| | |
|---|---|
| Charles R. Morrow | 09/14/21 |
| ***Approved By: Quality Assurance Officer*** | ***Date*** |
| Richard Hauk | 09/14/21 |
| ***Approved By: General Manager*** | ***Date*** |
| | 09/14/21 |
| ***Approved By: Laboratory Director*** | ***Date*** |

Standard Operating Procedures shall be reviewed at least once in two years or as needed to determine their continued suitability, compliance with applicable requirements, and to ensure that they reflect actual procedures being performed.



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**STANDARD OPERATING PROCEDURE**
**ANALYSIS OF AMMONIA-N IN WATER/WASTEWATER/LIQUID/SOLID MATRICES**

## 1.0 SCOPE AND APPLICATION

**1.1** This Standard Operating Procedure describes the determination of ammonia-nitrogen exclusive of total kjeldahl nitrogen, in drinking, surface and saline waters, solids/sediments, domestic and industrial wastes.

**1.2** This SOP is applicable to liquid/solid matrices by distillation and subsequent determination of ammonia-N by titrimetry.

## 2.0 REPORTING LIMIT

**2.1** The method covers the range from 1.0 to 25 mg/L for the titrimetric procedure with a practical quantitation limit of 1.0mg/L and 10mg/L for solids.

## 3.0 SUMMARY

**3.1** A representative sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid.

**3.2** The ammonia in the distillate is then determined by titration with standard sulfuric acid in the presence of an indicator.

## 4.0 DEFINITIONS

**4.1** **Method Blank –** Reagent water that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

**4.2** **Duplicate (DUP) –** A separate aliquot of the same sample from the same sample container.

**4.3** **Laboratory Fortified Blank/Laboratory Control Sample (LFB/LCS)** – Reagent water matrix, spiked with a solution containing method analyte[s] of known concentration. It is used to check analytical technique and sample preparation and method performance.

**4.4** **Laboratory Fortified Blank Duplicate/Laboratory Control Sample Duplicate (LFBD/LCSD)** – LCSD is same as LCS and is used to check instrument performance as well as to determine the precision of the analytical method.

**4.5** **Laboratory Fortified Matrix (LFM)** – An aliquot of a sample from the analytical batch spiked with a solution containing a mixture of anions of interest at known concentration. An LFM is used to check the effect of matrix on the analytes of interest.

**4.6** **Practical Quantitation Limit/Reporting Limit (PQL/RL)** – The lowest concentration that can be reliably measured within specified limits of precision and accuracy for a specific laboratory analytical method during routine laboratory operating conditions.

**4.7** **Working Standard Solution (WSS)** – A working standard solution is one that is an intermediate standard prepared by diluting the commercially purchased stock solution. A WSS is used to prepare standard solutions to calibrate the instrument.

## 5.0 INTERFERENCES

**5.1** Residual chlorine, Cyanates, Urea, etc., may cause interferences with the sample analysis. Residual chlorine may be removed by treatment with a solution of sodium thiosulfate prior to distillation and titration if the sample is known to contain residual chlorine.



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## 6.0 SAFETY

**6.1** Care should be exercised when handling the distillation equipment due to heat and possible pressure build up.

**6.2** Safety glasses and laboratory coats must be worn at all times while in the laboratory. In addition gloves and a face shield or goggles must be worn when dealing with toxic, caustic, and/or flammable chemicals.

**6.3** A partial facemask should be worn when working with samples suspected to contain high levels of volatile organics, such solvents, and samples contaminated with gasoline, etc.

**6.4** All chemical compounds should be treated as potential health hazards.

**6.5** The toxicity and/or carcinogenicity of each sample will most likely not be known. Therefore, it is imperative that each sample be handled as a potential health hazard.

**6.6** The analyst should familiarize themselves with all Safety Data Sheets (SDS), safety facilities, and equipment prior to beginning this procedure.

**6.7** Please address any and all health and safety concerns to management before beginning this procedure.

## 7.0 EQUIPMENT AND SUPPLIES

**7.1** Distillation apparatus such as RAPID STILL II, Labconco or equivalent.

**7.2** Distillation flasks, collection vessels, etc.

**7.3** Balance, Top Loading, Accurate to 0.001g, Denver, Sartorius, American Scientific, or Equivalent.

**7.4** Beakers/Erlenmeyer flasks, 125-250mL, Fisher Scientific, or Equivalent.

**7.5** Graduated Cylinders, 100mL Fisher Scientific, or Equivalent.

**7.6** Pipetter, 100–1000–5000µL, Fisher Scientific, or Equivalent.

**7.7** Pipette Tips, 200–1000–5000µL, BVA Scientific, or Equivalent.

**7.8** Volumetric flasks, 100, 1000mL, with ground-glass stoppers.

**7.9** Spatulas – Stainless steel.

**7.10** Whatman No. 42 filter papers.

**7.11** Aluminum dishes.

**7.12** Reciprocating shaker.

**7.13** Clean Ottawa sand.

**7.14** Teflon boiling chips.

## 8.0 REAGENTS AND STANDARDS

**8.1** Ultra-Pure Water, San Antonio Testing Laboratory, or equivalent.

**8.2** Ammonium chloride, stock solution – commercially purchased at 1000mg/L of $NH_3$-N.

**8.3** When commercial stock is unavailable then prepare in the laboratory a stock solution as below

**8.3.1** Dissolve 3.819 g $NH_4Cl$ in distilled water and bring to volume in a 1-liter volumetric flask. The concentration of this solution is 1000mg/L of $NH_3$-N [or 1,216mg/L of $NH_3$].

**8.4** Mixed indicator solution: Prepare as described below and combine solutions. Prepare monthly.

**8.4.1** Methyl Red indicator: Dissolve 200mg methyl red indicator in 100mL of 95% ethyl or isopropyl alcohol.



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**8.4.2** Methylene blue indicator: Dissolve 100mg of methylene blue in 50mL of 95% ethyl or isopropyl alcohol. Alternatively measure 10mL of a 1% aqueous solution of Methylene Blue and mix with 40mL of 95% ethyl or isopropyl alcohol.

**8.5** Indicating Boric acid solution: Dissolve 20 g of $H_3BO_3$ in distilled water, add 10mL of the mixed indicator solution [8.4] and dilute to 1 liter. Prepare monthly.

**8.6** Absorbent Boric acid [plain] solution: Dissolve 20g of $H_3BO_3$ in distilled water and dilute to 1 liter. DO NOT add the mixed indicator solution, and prepare fresh monthly.

**8.7** Borate buffer solution: Add 88 mL of 0.1 N NaOH solution to 500 mL of approximately 0.025 M sodium tetraborate solution (5.0 g anhydrous $Na_2B_4O_7$, or 9.5 g $Na_2B_4O_7$ $10H_2O$ per liter) and dilute to 1 liter.

**8.8** Sulfuric acid:

**8.8.1** 1N: Prepare by adding 28 mL of Conc. $H_2SO_4$ [18N] to 1 liter with reagent water.

**8.8.2** 0.1N: Prepare by diluting 2.8mL of Conc. $H_2SO_4$ [18N] to 1 liter with reagent water.

**8.8.3** 0.02N: Purchase commercially available solution or prepare by diluting 0.56mL of Conc. $H_2SO_4$ [18N] to 1L with laboratory reagent water.

**8.9** Sodium hydroxide:

**8.9.1** 10N: Dissolve 400 g NaOH in laboratory reagent water and dilute to 1 liter.

**8.9.2** 6N: Dissolve 240g NaOH in laboratory reagent water and dilute to 1 liter.

**8.9.3** 1N: Dissolve 40g NaOH in laboratory reagent water and dilute to 1 liter.

**8.9.4** 0.1N: Dissolve 4g NaOH in laboratory reagent water and dilute to 1 liter.

**8.10** Sodium Carbonate Solution [0.05N]: Dissolve 2.5g ±0.2g of $Na_2CO_3$ 100mL of water, transfer into a 1L volumetric flask and dilute to the mark with reagent water. Do not use after 1 week and prepare weekly or as needed.

**8.11** De-chlorinating reagent: Dissolve 3.5 g $Na_2S_2O_3.5H_2O$ in water and dilute to 1 liter or 1.75g in 500mL of reagent water. One mL of this solution will remove 1 mg/L of residual chlorine in 500 mL of sample.

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

### 9.1 Solid Sample Collection

**9.1.1** Solid [soil, sludge, and sediment] samples are collected in 4oz wide mouth borosilicate glass jars with PTFE lined lids.

**9.1.2** Solid samples do not require preservation with $H_2SO_4$, but must be kept on ice after collection and during transport to the lab to preserve sample integrity.

### 9.2 Liquid Sample Collection

**9.2.1** Representative [grab or composite] samples may be collected in 500mL–1000mL plastic or glass containers with screw cap lids.

**9.2.2** Preserve sample collection bottles with 2mL/500mL of sample with 1:1 $H_2SO_4$ prior to sampling, to adjust pH to <2. Samples may be collected unpreserved, however, in such cases un-acidified samples must be refrigerated ≤6°C and analyzed within 24 hours of sample collection. If samples are not going to be analyzed within 24 hours, pH must be adjusted to <2.0

**9.2.3** When dealing with samples subjected to or suspected of chlorination, add 1-2mL of dechlorinating solution to the sample container to remove 1-2mg/L of residual chlorine.



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**9.2.4** Transport of samples to the laboratory for ammonia-N analysis on wet ice to maintain the temperature >0°C and ≤6°C is recommended.

**9.3 Holding Time**

**9.3.1** The holding time for Ammonia-N analysis is 28 days for preserved samples and 24 hours for unpreserved samples, from the time of collection until the time of analysis.

## 10.0 STORAGE

**10.1** Samples must be stored until the time of analysis in a refrigerator at >0°C but ≤ 6°C to preserve sample integrity.

## 11.0 SAMPLE IDENTIFICATION

**11.1** Samples are received from Sample Receiving with a work order form generated from the Laboratory Information Management System (LIMS). This includes client identification, sample number, and tests to be performed under each department.

**11.2** Each sample is assigned a unique number and a container number if more than one container is received.

## 12.0 CALIBRATION AND STANDARDIZATION

**12.1** Balance must be checked using S Class weights on each day of use.

**12.2** *Standardization of sulfuric acid is only necessary when commercially purchased stock is not available.* Standardize sulfuric acid titrant if prepared in the laboratory with standard $Na_2CO_3$ solution prepared as in section 8.10.

**12.2.1** Transfer 40mL of standard $Na_2CO_3$ solution [0.025N] into a beaker or other suitable container and mix with 60mL of reagent water.

**12.2.2** Insert pH electrode into the beaker and titrate with sulfuric acid titrant [8.8.3] until a pH of approximately 5. Stop the titration at this point.

**12.2.3** Remove and rinse electrodes into the same beaker and gently heat the solution, covered with a watch glass over the beaker for 3–5 minutes, cool to room temperature. Rinse the watch glass into the beaker.

**12.2.4** Continue the titration very slowly to a pH of 4.5. Repeat two more times and calculate the normality of the standard sulfuric acid solution using the following formula:

$$Normality\ of\ H_2SO_4, N = \frac{A \times B}{53.00 \times C}$$

Where:

A = g of $Na_2CO_3$ weighed into 1L volumetric flask
B = mL of $Na_2CO_3$ solution taken for titration
C = mL of sulfuric acid used.

**12.2.5** Use the true normality of the acid titrant thus prepared in the calculations for $NH_3$-N, when laboratory prepared acid titrant is used in the analysis.

**12.2.6** Prepare a working standard solution using dilute $NH_4Cl$ stock prepared as in section 8.3.1 or use purchased ready to use stock solution.



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## 13.0 PROCEDURE

### 13.1 Solid Sample Extraction

**13.1.1** Weigh 5.00g field-moist soil sample into a kjeldahl (distillation) flask.

**13.1.2** Measure 50mL laboratory reagent water using graduated cylinder and pour it into the kjeldahl flask containing the soil sample. (If the sample is limited, it can be reduced to a minimum of 1.00g).

**13.1.3** Rinse the sides of kjeldahl flask with small amounts water to wash the soil down to the bottom of the flask.

**13.1.4** Prepare a laboratory control sample (LCS) using clean sand or boiling chips and spike with 1mL of a 1000 mg/L $NH_3$-N stock solution, this will yield a final concentration of 20mg/L of Ammonia-N.

**13.1.5** Prepare a matrix spike (MS) sample using a field sample as described in above section (13.1.1–13.1.3) and spike with 1mL of a 1000 mg/L $NH_3$-N stock solution, this will yield a final concentration of 20mg/L of Ammonia-N.

**13.1.6** Carry a method blank through the procedure using clean sand or boiling chips.

**13.1.7** Proceed as described in the sections below (13.3 and 13.4).

### 13.2 Liquid Sample Preparation

**13.2.1** Remove samples from the refrigerator holding area and allow to come to room temperature. Mix the contents of the sample container to obtain a representative sample for analysis and confirm that pH is <2.0 with pH strip. Record the pH results, pH paper Element ID and acid Element ID used to adjust pH in logbook.

**13.2.2** Prepare a sample duplicate by transferring an additional aliquot of a well-mixed representative field sample from the sample batch.

**13.2.3** Treat the field samples known to be subjected to or suspected of chlorination using the dechlorinating solution prior to distillation and reagent addition.

**13.2.4** Prepare a method blank, and LCS [Duplicate if necessary] using laboratory reagent water. Spike the LCS sample with 1mL of a 1000mg/L $NH_3$-N stock solution, this will yield a final concentration of 20mg/L of Ammonia-N.

### 13.3 Sample Distillation

**13.3.1** Set the Rapid Still II distillation unit according the manufacturer's instructions and follow all safety protocols described. Check the water level of the steam generation flask located at the back of the distillation unit and fill if necessary prior to initiating the distillation step.

**13.3.2** Using a graduated cylinder measure a 50mL portion of the well-mixed sample into the ammonia kjeldahl distillation flask. Neutralize the samples if necessary to approximately pH 7 with dilute base or acid.

**13.3.3** Add 2.5mL of borate buffer solution to all the samples and adjust the pH to 9.5 with 2 mL 6N NaOH and mix the contents. Add 1 mL de-chlorinating reagent. Attach the distillation flask to the unit.

**13.3.4** Add 25mL of Boric acid with 2 drops mixed indicator solution into a 250mL Erlenmeyer collection flask or a beaker or other suitable collection container.

**13.3.5** Start the unit and distill the samples at a rate of about 6–8mL per minute making sure that the tip of the delivery tube is below the liquid surface of the collection beaker.



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**13.3.6** Collect ~75mL of the distillate into an Erlenmeyer flask or a collection beaker containing indicating boric acid solution.

**13.3.7** Lower the distillation receiver so that the end of the delivery tube is free of contact with the liquid and continue distillation for two more minutes to clean the condenser and delivery tube.

**13.3.8** Collect all distilled samples and set aside for analysis of ammonia by titration as described below.

**13.4 Analysis of Ammonia and Ammonia-N by Titration**

**13.4.1** Determination by titration is done only after the samples have been distilled as in section 13.3. The presence of ammonia and ammonia-N thereof is indicated by a pale green color in the distillate.

**13.4.2** Titrate the distilled samples from section 13.3 with standard $H_2SO_4$ [0.02N or thereof] titrant prepared or purchased as in section 8.9.3 and 12.2.

**13.4.3** Titrate slowly as the end point is approached and continue until the pale green color turns into a pale lavender color.

**13.4.4** Record the initial and final burette reading and calculate the volume of titrant used to reach end point (in titration logbook).

**13.4.5** Carry a method blank, LCS [Dup], samples, sample duplicate, etc., through all steps of the procedure from preliminary distillation through titration. Apply any corrections derived from blank analysis to the results.

## 14.0 DATA ANALYSIS AND CALCULATIONS

**14.1** Sample Calculations

Calculate the concentration of Ammonia-N in liquid sample as follows:

$$NH_3\text{-}N\ mg/L = \frac{[A - B] \times [N] \times 14.007 \times 1000}{Sample\ Vol.\ [mL]}$$

Calculate the concentration of Ammonia in liquid sample as follows:

$$NH_3 mg/L = \frac{[A - B] \times [N] \times 17.031 \times 1000}{Sample\ Vol.\ [mL]}$$

Calculate the concentration of Ammonia-N in solid sample as follows:

$$NH3\text{-}N\ mg/Kg = \frac{[A - B] \times [N] \times 14.007 \times 1000}{Sample\ Wt.\ (g)}$$

Where:

A = Volume of $H_2SO_4$ [mL] titrated for sample.

B = Volume of $H_2SO_4$ [mL] titrated for Blank.

N = Normality of $H_2SO_4$ used for titration.

**14.2** Laboratory Control Sample [Dup] Recovery



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$$LCS\ \%\ Recovery = \frac{LCSR}{LCSA} \times 100$$

Where:

LCSR = LCS Spike Result
LCSA = Spike Added

**14.3** Relative Percent Difference

$$RPD = \left| \frac{SR - SDR}{\left( \frac{SR + SDR}{2} \right)} \right| \times 100$$

Where:

RPD = Relative Percent Difference
SR = Spike Recovery [or Sample Result]
SDR = Spike Duplicate Recovery [or Sample Duplicate Result]

## 15.0 QUALITY CONTROL

**15.1** The Practical Quantitation Limit/Reporting Limit (PQL/RL) for this method is 1mg/L for liquids and 10mg/kg for solids.

**15.2** At a minimum, a method blank, LCS/LCS-Duplicate, field sample duplicate, and a matrix spike if volume permits, should be analyzed of a batch of 20 samples or less for liquid.

**15.3** For solid/sediment samples, at a minimum a method blank, LCS/LCS-Duplicate, field sample duplicate of a batch of 20 samples or less. A Matrix Spike and matrix spike duplicate should be analyzed when sample amount permits the use of such MS/MSD sample.

**15.4** Chemicals and standards must be entered upon receipt into the LIMS and assigned a number. The containers must be dated when first opened and discarded by the expiration date. Any chemical or standard that fails to meet Quality Control requirements should be returned to the manufacturer for replacement.

**15.5** Working standards must be entered and assigned a number from the Chemical and Standards Database when prepared. All working standards must be discarded by the expiration date. Any working standard that fails to meet Quality Control requirements must be discarded and re-prepared. If the working standard continues to fail, contact the manufacturer of the chemicals, and if necessary order new supplies.

## 16.0 ACCEPTANCE CRITERIA

**16.1** Determine the blank concentration; the acceptance limit for the blank is ≤ 0.56

**16.2** Calculate the LCS recovery. The acceptable range for the LCS is 80-120%.

**16.3** Determine the RPD for the sample and sample duplicate or LCS/LCS-Duplicate. The acceptance limit for the RPD is <20.

## 17.0 CORRECTIVE ACTIONS FOR NON-CONFORMANCE DATA



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**17.1** Should a sample become contaminated or compromised, the preparation shall be terminated and repeated with a fresh sample aliquot. A Corrective Action must be completed to document the actions taken.

**17.2** When Quality Control measures fail, and the client's results are affected, the client will be advised that the results may not be reliable. It may be necessary based on client's needs to recollect the sample and submit at a later time. If the client is unable to recollect a sample, the data will be released with the appropriate documentation. The laboratory staff will complete a Corrective Action form to document this occurrence.

**17.3** When QC samples do not fall within the acceptable range, the analyst shall review the data for obvious errors such as calculations, preparation errors, or inadvertent spiking errors or other such causes that are not resultant of a systemic failure. The data may be released with a qualifying statement after concurring with the quality manager. A Corrective Action must be completed documenting the actions taken when the root cause identified is deemed detrimental to the analysis.

## 18.0 HANDLING NON-CONFORMANCE DATA

**18.1** Non-conformance data are monitored and resolved by identifying categories such as system based, methods based, preparative method based, etc., and are resolved once the problematic areas are identified.

## 19.0 METHOD PERFORMANCE

**19.1** One-hundred and two laboratory reagent water samples [analyzed between January 2020 and December 2020] spiked with 20mg/L of ammonia-N standard had an average recovery of 99.8% with a standard deviation of 5.16.

## 20.0 POLLUTION PREVENTION

**20.1** Each method is evaluated prior to use in order to minimize waste volume and toxicity.

**20.2** A non-hazardous or less toxic substitute may be used whenever possible.

**20.3** Purchase only the amount of chemical that is actually needed or that will be used to eliminate the cost of disposal later.

## 21.0 WASTE MANAGEMENT

**21.1** Toxic waste must never be disposed of down the drain.

**21.2** Waste generated from sample analysis must be segregated if the process knowledge indicates the presence of any of the hazardous components listed in Table–1, 40 CFR 261.24 and exceed the limits set in the table.

**21.3** When disposing samples the analyst must follow current revision of the "Laboratory Waste Handling and Disposal" SOP (SATL#007G) for detailed disposal procedures.

**21.4** All chemicals and containers must be properly identified and labeled at all times to eliminate ambiguity and cost of disposal of unknowns. If an unknown chemical or container is discovered, label it as 'unknown' and attach a note detailing any information about what the chemical may be, what test it may have been used for, and where it was found. If you find an unlabeled chemical



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that has crystallized or there is any other indication that it may be unstable, notify management immediately.

**21.5** Generally, empty chemical containers are not considered hazardous waste. Check with management if one such container is found and in doubt. To dispose of the container in the regular trash the container must be completely empty and triple rinsed several times.

**21.6** The waste drums are picked up upon notification and a copy of the report is submitted to the waste management company.

## 22.0 REFERENCES

**22.1** "Determination of Ammonia-Nitrogen SM4500-$NH_3$ [B&C]", Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

**22.2** "Determination of Ammonia-Nitrogen SM4500-$NH_3$ [B&C]", Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005.

**22.3** "Determination of Ammonia-Nitrogen SM4500-$NH_3$ [B&C]", Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2011.

**22.4** "Determination of Ammonia-Nitrogen SM4500-$NH_3$ [B&C]", Standard Methods for the Examination of Water and Wastewater, 23rd Edition, 2017.

**22.5** "Determination of Nitrogen, Ammonia, [Distillation, and Titration]", Method 350.2, US Environmental Protection Agency, 1974.

**22.6** Carter, 1993. Soil Sampling and Methods of Analysis, Florida: Lewis Publishers.

## 23.0 REVISION HISTORY

**23.1** New revision of the method.

**23.2** Annual revision of the method.

**23.3** Annual revision 2012, Rev 2.0.0 – revised for language, redundancy and formatting.

**23.4** Annual revision 2014. Revised sections: 8.0, 9.0, 12.2.6, 13.0, 13.2, 13.3 15.0, and 19.0.

**23.5** Post assessment revision to provide reference method edition on title page.

**23.6** Annual revision 2019, Revised sections: 7.0, 8.0, 13.1, 14.0, and 19.0.



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**STANDARD OPERATING PROCEDURE**
**ANALYSIS OF AMMONIA-N IN WATER/WASTEWATER/LIQUID/SOLID MATRICES**

## APPENDIX A
## SOP History and Version Control

| Version | Date of Review/Revision | Review/Revision Approved by | Brief Description |
|---|---|---|---|
| 2.3 | 05/17/2016 | M. Bernard | Revised title page, update method performance data, clarification of reagents and amounts used for distillation. Addition of Appendix A to reflect SOP history and version control. |
| 3.0 | 06/19/2017 | M. Bernard | Biennial review; confirm pH adjustments, method performance update and waste disposal protocol. |
| 4.0 | 03/05/2019 | S. Abburu | Biennial review; Procedural change for solids; calculations updated; method performance data updated. |
| 5.0 | 02/05/2021 | A.Rosecrance | Biennial review; update cover page; add dechlorination reagent to sample distillation procedure; change color from distinct yellow to pale green |
| 5.1 | 09/13/2021 | C. Morrow | Revised the following:<br>Section 2 – Quantitation limit.<br>Section 4 – Update definitions.<br>Section 15 – Update QC requirements.<br>Section 19 – Update method performance data.<br>Section 22 – Update reference method information.<br>Added Appenix B – Quality acceptance criteria. |



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# APPENDIX B
## Quality Acceptance Criteria

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Method Blank (MB) | Every batch of 20 samples or less | If MB > ½ PQL but < PQL and sample results are > PQL, then qualify results to indicate that analyte was detected in the reagent blank.<br>If reagent blank is > PQL, then further action and qualification is required | Take remedial action(s) as defined in 17, repeat measurement and/or qualify data. |
| Laboratory-fortified blank (LFB)/Laboratory-fortified blank duplicate (LFBD) | Daily, before sample analysis. | Within control limits. If outside control limits, take corrective action. | Take remedial action(s) as defined in 17, repeat measurement and/or qualify data. |
| Laboratory-fortified matrix (LFM)/Laboratory-fortified matrix duplicate (LFMD) | If a LFM is feasible, one LFB every batch of 20 samples or less. | Within control limits. If outside control limits, qualify data. | Qualify data. |



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# STANDARD OPERATING PROCEDURE

*Title*

**Total Dissolved Solids (TDS)**

Filterable Residue

*Reference Method No.:*

**EPA 160.1/SM2540C (23rd Edition, 2017)**

*Matrix/Matrices:*

**Liquid/Drinking Water**

*Document Control Number/Revision Number*

**SOP007A/Revision 5.1**

| | |
|---|---|
| Charles R. Morrow | 09/14/2021 |
| ***Approved By: Quality Assurance Manager*** | ***Date*** |
| Richard Hauk | 09/14/2021 |
| ***Approved By: General Manager*** | ***Date*** |
| | 09/14/2021 |
| ***Approved By: Laboratory Director*** | ***Date*** |

Standard Operating Procedures shall be reviewed at least once in two years or as needed to determine their continued suitability, compliance with applicable requirements, and to ensure that they reflect actual procedures being performed.



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**STANDARD OPERATING PROCEDURE**
**ANALYSIS OF TOTAL DISSOLVED SOLIDS (TDS) FILTERABLE RESIDUE**

## 1.0 SCOPE AND APPLICATION

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

## 2.0 REPORTING LIMIT

**2.1** The practical range of the determination is 2.5 mg/L to 20,000 mg/L, however the working range is 2.5 to 200 mg of residue.

## 3.0 SUMMARY

**3.1** A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C and dissolved solids are calculated by the gravimetry.

## 4.0 DEFINITIONS

**4.1** **Filterable Residue** – Solids capable of passing through a glass fiber filter and dried to constant weight at 180°C.

**4.2** **Batch** –The batch is a set of up of the same matrix processed using the same procedures and reagents within the same time period. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible.

**4.3** **Method Blank –** Reagent water, which are carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

**4.4** **Laboratory Fortified Blank/Laboratory Control Sample (LRB/LCS)** – Reagent water spiked with a solution containing a known concentration of total dissolved solids. LCS sample is optional in this method and can be analyzed when suitable standard is available from external vendors. LCS data may be used to generate precision and method performance.

**4.5** **Laboratory Fortified Blank Duplicate/Laboratory Control Sample Duplicate (LFBD/LCSD)** – LCSD is same as LCS and is used to check instrument performance as well as to determine the precision of the analytical method.

**4.6** **Duplicate (DUP) –** A separate aliquot of the same sample from the same sample container.

**4.7** **Practical Quantitation Limit/ Reporting Limit (PQL/RL)** – The lowest concentration that can be reliably measured within specified limits of precision and accuracy for a specific laboratory analytical method during routine laboratory operating conditions. The laboratory uses the NELAC term of Limit of Quantitation (LOQ) to establish the lowest Minimum Reporting Limit (MRL) that a concentration of an analyte can be reported without qualification.

**4.8** **Limit of Quantitation (LOQ)** – For purposes of this method, the LOQ is equal to the Reporting Limit (RL), which is the lowest limit an analyte's concentration can be reported without qualification.

## 5.0 INTERFERENCES



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**5.1** Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

**5.2** Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

**5.3** If process knowledge is known or historical data suggest high TDS values, a smaller amount of sample volume may be used.

**5.4** Drying time and temperature should be monitored.

## 6.0 SAFETY

**6.1** Safety glasses and laboratory coats must be worn at all times while in the laboratory. In addition gloves and a face shield or goggles must be worn when dealing with toxic, caustic, and/or flammable chemicals.

**6.2** A partial facemask should be worn when working with samples suspected to contain high levels of volatile organics, such solvents, and samples contaminated with gasoline, etc.

**6.3** All chemical compounds should be treated as potential health hazards.

**6.4** The toxicity and/or carcinogenicity of each sample will most likely not be known. Therefore, it is imperative that each sample be handled as a potential health hazard.

**6.5** The analyst should familiarize themselves with all Safety Data Sheets (SDS), safety facilities, and equipment prior to beginning this procedure.

**6.6** Please address any and all health and safety concerns to management before beginning this procedure.

## 7.0 EQUIPMENT AND SUPPLIES

**7.1** Balance, Top Loading, Accurate to 0.0001g, Denver Instruments, or Equivalent

**7.2** Mechanical Convection Drying Oven, Precision, or Equivalent

**7.3** Vacuum Pump, With Safety Trap Flask, Filter Manifold, and Waste Flask

**7.4** Buchner Funnel with Fixed Perforated Plate, Fisher Scientific, Catalog No. 10-356C, or Equivalent

**7.5** Glass Fiber Filters, Fisher Scientific, Catalog No. 09-790-46J, or Equivalent

**7.6** Porcelain Evaporation Dish, Fisher Scientific, Catalog No. S33705, or Equivalent

**7.7** Desiccators, Fisher Scientific, Catalog No.08-615B, or Equivalent

**7.8** Graduated Cylinder, 100mL, Fisher Scientific, Catalog No. 08-549-11C, or Equivalent

## 8.0 REAGENTS AND STANDARDS

**8.1** Ultra-Pure Water, [<1µmho/cm conductivity] San Antonio Testing Laboratory or equivalent.

**8.2** Solids Standard, AccuStandard, Catalog No. WC-SOL, or Equivalent.

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

**9.1** Samples must be collected in plastic or glass bottles. At least 100mL is required to complete the analysis. Analysis should begin as soon as practically possible.

**9.2** The maximum holding time for Total Dissolved Solids is 7 days from collection to analysis.



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## 10.0 STORAGE

**10.1** Samples are stored until the time of analysis in a refrigerator at >0°C but ≤ 6°C to preserve sample integrity.

## 11.0 SAMPLE IDENTIFICATION

**11.1** Samples are received from Sample Receiving with a work order form generated from the Laboratory Information Management System (LIMS). This includes client identification, sample number, and tests to be performed under each department.

**11.2** Each sample is assigned a unique number and a container number if more than one container is received.

## 12.0 CALIBRATION AND STANDARDIZATION

**12.1** Balance must be QC checked using S-Class weights prior to each day of use.

**12.2** Oven and refrigerator temperatures are verified and recorded each day in the Daily Laboratory QC Log book located in the laboratory.

## 13.0 PROCEDURE

**13.1** Preparation of Evaporation Dish For Filterable Residue

**13.1.1** Heat a clean porcelain-evaporating dish to 180 ± 2°C for approximately 1 hour.

**13.1.2** Remove and store in a desiccator until needed.

**13.1.3** Weigh evaporation dish immediately before use. Record initial weight as tare weight.

**13.2** Preparation of Glass Fiber Filter.

**13.2.1** Place a glass fiber filter on the Buchner funnel. Turn the vacuum on.

**13.2.2** Wash the filter three times with approximately 20mL portions of ultra pure water.

**13.2.3** Continue to vacuum until all traces of water have passed through.

**13.2.4** Leave glass fiber filter in place and discard washings.

**13.3** Remove field samples from refrigerator and warm to room temperature.

**13.4** Shake the sample container well to mix and measure a 100mL representative sample using a graduated cylinder. However, the sample amount used may need to be adjusted in order to yield a dried residue between 2.5 and 200 mg as per reference method.

*Note: Samples can be screened for either conductivity or TDS using a conductivity/TDS probe to aid in estimating the sample volume required for analysis. However, this may not be enough to judge the proper sample volume required but may provide a rough estimate. Use caution while screening samples containing high TDS values as probes may give lower than actual value. Upon the completion of the gravimetric analysis, it may be required to use higher sample volume to obtain a residue between 20-200mg.*

**13.5** Turn on the vacuum and slowly pour the sample over the glass fiber filter in the Buchner funnel.



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**13.6** Filter the sample through the glass fiber filter. Rinse the graduate cylinder with three successive 10mL portions of reagent water and pour into the Buchner funnel while allowing each 10mL volume to completely drain.

**13.7** Continue to apply vacuum for approximately 3 minutes after filtration is complete to remove as much water as possible.

**13.8** Transfer the total filtrate from the flask to a pre-weighed evaporation dish prepared in 13.1.

*Note: In rare cases the filtrate volume may exceed the holding capacity of the dish. In such cases do not pour the excess into another evaporation dish. Dry the sample dish to evaporate the filtrate and add the filtrate to the same evaporation dish to accommodate the remaining filtrate volume.*

**13.9** Evaporate sample in a drying oven at 104°C ± 1°C until all the sample has evaporated to dryness. This may take 6-8 hours. Alternately, dry the samples in the drying oven at 104°C ± 1°overnight to dryness.

**13.10** Adjust temperature of the oven to 180 ± 2°C or transfer dish to oven set at 180 ± 2°C and dry the evaporating dish for at least 1 hour. After drying for at least 1 hour, remove the dish and cool in a desiccator.

**13.11** Weigh the evaporating dish. The drying cycle must be repeated at least once and further if necessary until a constant weight is obtained or until the weight loss between two successive measurements is less than 0.5 mg.

**13.12** Record the initial and final weights of the dish in the electronic spreadsheet.

## 14.0 DATA ANALYSIS AND CALCULATIONS

**14.1** Filterable Residue

$$Filterable\ Residue\ [TDS]\ (mg/L) = \frac{(A - B) \times 1000}{Vs}$$

Where:

A = Weight of dried residue [g] + Weight of dish [g]
B = Weight of dish [g]
$V_s$ = Sample volume [mL]

**14.2** Laboratory Control Sample Recovery

$$Spike\ Recovery\ [\%] = \frac{LCSR}{LCSA} \times 100$$

Where:

LCSR = LCS Spike Result
LCSA = Spike Added

**14.3** Relative Percent Difference

$$RPD = \left| \frac{SR - SDR}{\left( \frac{SR + SDR}{2} \right)} \right| \times 100$$



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Where:

RPD = Relative Percent Difference
SR = Spike Recovery
SDR = Spike Duplicate Recovery

## 15.0 QUALITY CONTROL

**15.1** The Practical Quantitation Limit/Reporting Limit (PQL/RL) for this method is 2.5mg/L from a 1 liter sample volume.

**15.2** Perform a minimum of one method blank, one fortified reagent blank, and one sample duplicate for every 20 field samples or less. Duplicate sample results should agree within 5%.

**15.3** Chemicals and standards must be entered upon receipt into the LIMS and assigned a number. The containers must be dated when first opened and discarded by the expiration date. Any chemical or standard that fails to meet Quality Control requirements should be returned to the manufacturer for replacement.

**15.4** Working standards must be entered and assigned a number from the Chemical and Standards Database when prepared. All working standards must be discarded by the expiration date. Any working standard that fails to meet Quality Control requirements must be discarded and re-prepared. If the working standard continues to fail, contact the manufacturer of the chemicals, and if necessary order new supplies.

**15.5** All Certificates of Analysis should be retained.

## 16.0 ACCEPTANCE CRITERIA

**16.1** Method blanks must yield a value below the established reporting limit.

**16.2** Duplicate determinations should agree within 5% of their average weight. When samples containing high dissolved solids are analyzed as field duplicates, the RPD values may exceed the 5% requirement. In such cases the data shall be flagged on the analytical report.

**16.3** The acceptance limits for spike standard recovery (LCS/D) are 80-120% (accuracy) of the true concentration.

## 17.0 CORRECTIVE ACTIONS FOR NON-CONFORMANCE DATA

**17.1** When QC samples do not fall within the acceptable range, the analyst shall review the data for obvious errors such as calculations, preparation errors, or inadvertent spiking errors or other such causes that are not resultant of a systemic failure. The data may be released with a qualifying statement after concurring with the quality manager A Corrective Action must be completed documenting the actions taken when the root cause identified is deemed detrimental to the analysis.

**17.2** Should a sample become contaminated or compromised, the preparation shall be terminated and repeated with a fresh sample aliquot. A Corrective Action must be completed to document the actions taken.

## 18.0 HANDLING NON-CONFORMANCE DATA



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**18.1** Non-conformance data are monitored and resolved by identifying categories such as system based, methods based, preparative method based, etc., and are resolved once the problematic areas are identified.

## 19.0 METHOD PERFORMANCE

**19.1** Two hundred and ten reagent water samples spiked with 100mg/L of TDS standard analyzed January 2020 – December 2020, had an average recovery of 99.1% with a standard deviation of 9.9. Method Detection Limit studies, or NELAC's Limit of Detection (LOD), are not applicable to this gravimetric procedure.

**19.2** The Reporting Limit (RL) of quantitation is equivalent to NELAC's Limit of Quantitation (LOQ).

## 20.0 POLLUTION PREVENTION

**20.1** Each method is evaluated prior to use in order to minimize waste volume and toxicity.

**20.2** A non-hazardous or less toxic substitute may be used whenever possible.

**20.3** Purchase only the amount of chemical that is actually needed or that will be used to eliminate the cost of disposal later.

## 21.0 WASTE MANAGEMENT

**21.1** Toxic waste must never be disposed of down the drain.

**21.2** Waste generated from sample analysis must be segregated if the process knowledge indicates the presence of any of the hazardous components listed in Table–1, 40 CFR 261.24 and exceed the limits set in the table.

**21.3** When disposing samples the analyst must follow current revision of the "Laboratory Waste Handling and Disposal" SOP (SATL#007G) for detailed disposal procedures.

**21.4** All chemicals and containers must be properly identified and labeled at all times to eliminate ambiguity and cost of disposal of unknowns. If an unknown chemical or container is discovered, label it as 'unknown' and attach a note detailing any information about what the chemical may be, what test it may have been used for, and where it was found. If you find an unlabeled chemical that has crystallized or there is any other indication that it may be unstable, notify management immediately.

**21.5** Generally, empty chemical containers are not considered hazardous waste. Check with management if one such container is found and in doubt. To dispose of the container in the regular trash the container must be completely empty and tripled rinsed.

**21.6** The waste drums are picked up upon notification and a copy of the report is submitted to the waste management company.

## 22.0 REFERENCES

**22.1** "Filterable Residue," Storet No. 70300, EPA Method 160.1, 1971

**22.2** Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2011

**22.3** Standard Methods for the Examination of Water and Wastewater, 23rd Edition, 2017

**22.4** The TNI Standard, 2016



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**22.5** EPA/600/R-04/003, March 2012

## 23.0 REVISION HISTORY

**23.1** The following sections of this SOP were revised for Revision 1.9.0, as a result of an annual review and the last TCEQ on-site assessment: sections 13.4, 15.2, 16.1, and 16.2

**23.2** Annual revision 2.0, added Drinking Water matrix to this SOP.

**23.3** Annual revision 2.0.0 – Revised for general language and removed redundancies in section 17.0, and updated method performance data in 19.0

**23.4** Annual revision 2014. Revised sections: 5.0, 6.0, 12.0, 13.0, and 14.0, 16.0 and 19.0

**23.5** Post assessment revision to provide reference method edition on the title page.



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**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF TOTAL DISSOLVED SOLIDS (TDS) FILTERABLE RESIDUE**

## APPENDIX A
## SOP History and Version Control

| Version | Date of Review/Revision | Review/Revision Approved by | Brief Description |
|---|---|---|---|
| 2.3 | 07/08/2016 | M. Bernard | Revision of cover page, update of method performance data and addition of Appendix A to reflect SOP history and version control. |
| 3.0 | 06/19/2017 | M. Bernard | Biennial review; method performance update and waste disposal protocol. |
| 4.0 | 02/18/2019 | M. Bernard | Biennial review; revised cover page, (2.1) clarify PQL, (13.9, 13.11) clarify drying protocol and recording of weights, (15.1) clarify QC range, (19.1) method performance update and (22.0) reference update. |
| 5.0 | 04/16/2021 | A. Rosecrance | Biennial review; update cover page; change MSDS to SDS. |
| 5.1 | 09/13/2021 | C. Morrow | Revised the following:<br>Section 15 – Update QC requirements.<br>Section 19 – Update method performance.<br>Section 22 – Update reference information.<br>Add Appendix B – QC acceptance criteria. |



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**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF TOTAL DISSOLVED SOLIDS (TDS) FILTERABLE RESIDUE**

## APPENDIX B
## Quality Control Acceptance Criteria

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Method Blank (MB) | Every batch of 20 samples or less | If MB > ½ PQL but < PQL and sample results are > PQL, then qualify results to indicate that analyte was detected in the reagent blank.<br>If reagent blank is > PQL, then further action and qualification is required | Take remedial action(s) as defined in Section 17, repeat measurement and/or qualify data. |
| Laboratory-fortified blank (LFB)/Laboratory-fortified blank duplicate (LFBD) | Daily, before sample analysis. | Within control limits. If outside control limits, take corrective action. | Take remedial action(s) as defined in Section 17, repeat measurement and/or qualify data. |



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# STANDARD OPERATING PROCEDURE

*Title*

**Analysis of Specific Conductance**

*Reference Method No.:*

**EPA 120.1/SM2510B (23rd Edition, 2017)**

*Matrix/Matrices:*

**Liquid/Drinking Water/Solids**

*Document Control Number/Revision Number*

**SOP008A/Revision 5.1**

| | |
|---|---|
| Charles R. Morrow | 09/14/2021 |
| ***Approved By: Quality Assurance Manager*** | ***Date*** |
| Richard Hauk | 09/14/2021 |
| ***Approved By: General Manager*** | ***Date*** |
| | 09/14/2021 |
| ***Approved By: Laboratory Director*** | ***Date*** |

Standard Operating Procedures shall be reviewed at least once in two years or as needed to determine their continued suitability, compliance with applicable requirements, and to ensure that they reflect actual procedures being performed.



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**STANDARD OPERATING PROCEDURE**
**ANALYSIS OF SPECIFIC CONDUCTANCE IN WATER/WASTEWATER/LIQUIDS**

## 1.0 SCOPE AND APPLICATION

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

## 2.0 REPORTING LIMIT

**2.1** Bench top meter has an accuracy of 1 µmhos/cm.

## 3.0 SUMMARY

**3.1** The specific conductance of a sample is measured by use of a self-contained conductivity meter.
**3.2** A representative sample is collected in a digestion cup and the specific conductance is measured directly from the conductivity meter, and reported as µmhos/cm.

## 4.0 DEFINITIONS

**4.1** **Conductivity:** is a measure of the ability of an aqueous solution to carry an electric current, which depends on the presence of ions, their total concentration, mobility and valence, and on temperature of measurement.
**4.2** **Batch** –The batch is a set of samples of the same matrix processed using the same procedures and reagents within the same time period. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible.
**4.3** **Duplicate (DUP) –** A separate aliquot of the same sample from the same sample container.
**4.4** **Laboratory Fortified Blank/Laboratory Control Sample (LFB/LCS)** – A solution, such as 0.01M KCl, having a known specific conductance value.
**4.5** **Practical Quantitation Limit/Reporting Limit (PQL/RL)** – The lowest concentration that can be reliably measured within specified limits of precision and accuracy for a specific laboratory analytical method during routine laboratory operations.

## 5.0 INTERFERENCES

**5.1** Electrode fouling and inadequate sample circulation are the most common reasons for inaccurate data.
**5.2** Temperature variations also represent a large source of potential error; meter equipped with ATC [automatic temperature compensation] probe is recommended to reduce errors.
**5.3** Dissolved carbon dioxide in liquid matrices interferes with conductivity measurements.

## 6.0 SAFETY

**6.1** Safety glasses and laboratory coats must be worn at all times while in the laboratory. In addition gloves and a face shield or goggles must be worn when dealing with toxic, caustic, and/or flammable chemicals.
**6.2** A partial face mask should be worn when working with samples suspected to contain high levels of volatile organics, such solvents, and samples contaminated with gasoline, etc.
**6.3** All chemical compounds should be treated as potential health hazards.



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**6.4** The toxicity and/or carcinogenicity of each sample will most likely not be known. Therefore, it is imperative that each sample be handled as a potential health hazard.

**6.5** The analysts should familiarize themselves with all Safety Data Sheets (SDS), safety facilities, and equipment prior to beginning this procedure.

**6.6** Please address any and all health and safety concerns to management before beginning this procedure.

## 7.0 EQUIPMENT AND SUPPLIES

**7.1** Orion Five Star Benchtop meter, Thermo-Electron Corporation, or Equivalent

**7.2** Conductivity Probe, Thermo-Electron , Fisher Scientific, or Equivalent

**7.3** Digestion Cups, ~50mL Capacity, Environmental Express, Catalog No. SC475 or Equivalent.

**7.4** Graduated Cylinder, 100mL, Class A, Fisher Scientific, Catalog No. 08-549-11C, or Equivalent

## 8.0 REAGENTS AND STANDARDS

**8.1** Ultra-Pure Water [<1µmho/cm], San Antonio Testing Laboratory, or Equivalent.

**8.2** Conductivity Standards (1409 µmhos/com, 12,856 µmho/cm, 1000 µmhos/com) LabChem Catalog No. LC187802, LC187792, LC187712, or Equivalent.

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

**9.1** Samples can be collected in plastic, Teflon, or glass containers and refrigerated upon collection.

**9.2** Sample bottles must be filled as full as possible and kept tightly closed.

**9.3** No chemical preservation is required for specific conductance.

**9.4** Properly preserved samples stored under conditions described below have a holding time of 28 days from the time of collection.

## 10.0 STORAGE

**10.1** Analysis should begin as soon as practically possible once the samples are received at the laboratory.

**10.2** If analysis cannot be started immediately, samples must be stored until the time of analysis in a refrigerator at >0°C but ≤ 6°C to preserve sample integrity.

## 11.0 SAMPLE IDENTIFICATION

**11.1** Samples are received from Sample Receiving with a work order form generated from the Laboratory Information Management System (LIMS). This includes client identification, sample number, and tests to be performed under each department.

**11.2** Each sample is assigned a unique number and a container number if more than one container is received.

## 12.0 CALIBRATION AND STANDARDIZATION

**12.1** Follow instrument manufacturer's recommended calibration procedure.



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## 13.0 PROCEDURE

### 13.1 Liquid Samples

**13.1.1** Allow samples to reach room temperature prior to analysis.

**13.1.2** Ultra-pure water serves as a method blank. Place the electrode in a digestion cup with ultra-pure water and press the measure button on the conductivity meter. If reading is less than the reporting limit of 1 µmhos/cm, proceed to read the LCS.

**13.1.3** Prepare the LCS by adding 25 ± 1mL of Conductivity Standard to a digestion cup. Concentration of Conductivity Standard is 1000µmhos/cm.

**13.1.4** Place the electrode in the sample in the digestion cup and press the measure button on the conductivity meter.

*Note: Conductivity meter is capable of automatically switching between units depending on the conductivity of the sample. Typical units are µS/cm, mS/cm, µmhos/cm and/or mmhos/cm. Ensure that correct units are recorded in the laboratory logbook and entered into the Element system.*

**13.1.5** While the meter is in the measuring mode, the "Read" symbol will blink. Wait until the meter shows a constant reading and record the value in the logbook.

**13.1.6** Repeat steps 13.1.4 and 13.1.5 for each sample, using 25 ± 1mL of a representative sample in a digestion cup.

**13.1.7** If samples are saturated with dissolved salts are being measured, dilute the sample appropriately and measure. Record dilution used in the logbook. A dilution factor of 10–20 is recommended to minimize errors due to high dilutions.

**13.1.8** Report final results from dilution analysis, by multiplying the dilution factor with meter reading.

### 13.2 Solid/Soil Samples

**13.2.1** Allow samples to reach room temperature prior to analysis.

**13.2.2** For solid samples, prepare a 5 g sample in 25 mL deionized water in a digestion cup and shake for 2 minutes. For soil samples, prepare a 5 g sample in 5 mL deionized water in a digestion cup and shake for a few seconds.

**13.2.3** Analyze samples as in steps 13.1.4 and 13.1.5.

**13.2.4** Report results in µmhos/cm without a dilution factor.

## 14.0 DATA ANALYSIS AND CALCULATIONS

**14.1.1** Laboratory Control Sample [Dup] Recovery

$$Spike\ Recovery = \frac{LCSR}{LCSA} \times 100$$

Where:

LCSR = LCS Spike Result
LCSA = Spike Added

**14.1.2** Relative Percent Difference

$$RPD = \left| \frac{SR - SDR}{\left( \frac{SR + SDR}{2} \right)} \right| \times 100$$

Where:

RPD = Relative Percent Difference
SR = Spike Recovery
SDR = Spike Duplicate Recovery

## 15.0 QUALITY CONTROL

**15.1** The Practical Quantitation Limit (PQL)/Reporting Limit (RL) for this method is 1umho/cm or 1µS/cm.

**15.2** A minimum of a method blank, a sample duplicate every 20 samples or less, and one Laboratory Control Sample (LCS) must be analyzed for a batch of 20 samples or less.

**15.3** Chemicals and standards must be entered upon receipt into the LIMS and assigned a number. The containers must be dated when first opened and discarded by the expiration date. Any chemical or standard that fails to meet Quality Control requirements should be returned to the manufacturer for replacement.

**15.4** Working standards must be entered and assigned a number from the Chemical and Standards Database when prepared. All working standards must be discarded by the expiration date. Any working standard that fails to meet Quality Control requirements must be discarded and re-prepared. If the working standard continues to fail, contact the manufacturer of the chemicals, and if necessary order new supplies.

**15.5** All Certificates of Analysis should be retained.

## 16.0 ACCEPTANCE CRITERIA

**16.1** Calculate the LCS recovery. The acceptable range for the LCS is 80-120%.

**16.2** Analyze a sample duplicate for every 10 samples.

**16.3** Determine the RPD for the sample and sample duplicate. The acceptable range for the RPD is <20%.

## 17.0 CORRECTIVE ACTIONS FOR NON-CONFORMANCE DATA

**17.1** When QC samples do not fall within the acceptable range, the analyst shall review the data for obvious errors such as calculations, preparation errors, or inadvertent spiking errors or other such causes that are not resultant of a systemic failure. The data may be released with a qualifying statement after concurring with the quality manager A Corrective Action must be completed documenting the actions taken when the root cause identified is deemed detrimental to the analysis.

**17.2** Should a sample become contaminated or compromised, the preparation shall be terminated and repeated with a fresh sample aliquot. A Corrective Action must be completed to document the actions taken.



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## 18.0 HANDLING NON-CONFORMANCE DATA

**18.1** Non-conformance data are monitored and resolved by identifying categories such as system based, methods based, preparative method based, etc., and are resolved once the problematic areas are identified.

## 19.0 METHOD PERFORMANCE

**19.1** Precision of the method is dependent on the instrument and conductivity probe recommended by the manufacturer.

**19.2** One hundred sixty-five reagent water samples with 1000µmho/cm of Conductivity standard analyzed form January 2020 -December 2020, had an average recovery of 102% with a standard deviation of 4.9.

## 20.0 POLLUTION PREVENTION

**20.1** Each method is evaluated prior to use in order to minimize waste volume and toxicity.

**20.2** A non-hazardous or less toxic substitute may be used whenever possible.

**20.3** Purchase only the amount of chemical that is actually needed or that will be used to eliminate the cost of disposal later.

## 21.0 WASTE MANAGEMENT

**21.1** Toxic waste must never be disposed of down the drain.

**21.2** Waste generated from sample analysis must be segregated if the process knowledge indicates the presence of any of the hazardous components listed in Table–1, 40 CFR 261.24 and exceed the limits set in the table.

**21.3** When disposing samples the analyst must follow current revision of the "Laboratory Waste Handling and Disposal" SOP (SATL#007G) for detailed disposal procedures.

**21.4** All chemicals and containers must be properly identified and labeled at all times to eliminate ambiguity and cost of disposal of unknowns. If an unknown chemical or container is discovered, label it as 'unknown' and attach a note detailing any information about what the chemical may be, what test it may have been used for, and where it was found. If you find an unlabeled chemical that has crystallized or there is any other indication that it may be unstable, notify management immediately.

**21.5** Generally, empty chemical containers are not considered hazardous waste. Check with management if one such container is found and in doubt. To dispose of the container in the regular trash the container must be completely empty and triple rinsed.

**21.6** The waste drums are picked up upon notification and a copy of the report is submitted to the waste management company.

## 22.0 REFERENCES

**22.1** EPA 120.1, Conductance (Specific Conductance µmhos at 25°C)

**22.2** Standard Methods for the Examination of Water and Wastewater, 21st Edition 2005.

**22.3** Standard Methods for the Examination of Water and Wastewater, 22nd Edition 2011.



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**22.4** Standard Method for the Examination of Water and Wastewater, 23rd Edition, 2017.

## 23.0 REVISION HISTORY

**23.1** New revision (# 1.0.0) of the method.
**23.2** Annual revision # 1.0.1, added Drinking Water matrix to the SOP.
**23.3** Annual revision - 2.0.0 – Revised for language and redundancy.
**23.4** Annual revision 2014. revised sections:4.0, 5.0, 13.0, and 19.0
**23.5** Post assessment revision to provide performance criteria for method blank and to include reference method edition on the title page.



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**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF SPECIFIC CONDUCTANCE IN WATER/WASTEWATER/LIQUIDS**

## APPENDIX A
## SOP History and Version Control

| Version | Date of Review/Revision | Review/Revision Approved by | Brief Description |
|---|---|---|---|
| 2.3 | 07/11/2016 | M. Bernard | Revision of cover page, update of method performance data and addition of Appendix A to reflect SOP history and version control. |
| 3.0 | 06/19/2017 | M. Bernard | Biennial review; method performance update and waste disposal protocol. |
| 4.0 | 08/02/2019 | M. Bernard | Biennial review; revision of title page, method performance update and reference update. |
| 5.0 | 04/16/2021 | A.Rosecrance | Biennial review; revision of title page; change MSDS to SDS; added procedure for solid/soil samples |
| 5.1 | 09/13/2021 | C. Morrow | Revised the following:<br>Section 2 – Update quantitation limit.<br>Section 15 – Update QC requirements.<br>Section 19 – Update method performance.<br>Section 22 – Update reference information.<br>Add Appendix B – QC acceptance criteria. |



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**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF SPECIFIC CONDUCTANCE IN WATER/WASTEWATER/LIQUIDS**

## APPENDIX B
## Quality Control Acceptance Criteria

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Method Blank (MB) | Every batch of 20 samples or less | If MB > ½ PQL but < PQL and sample results are > PQL, then qualify results to indicate that analyte was detected in the reagent blank.<br>If reagent blank is > PQL, then further action and qualification is required | Take remedial action(s) as defined in Section 17, repeat measurement and/or qualify data. |
| Laboratory-fortified blank (LFB)/Laboratory-fortified blank duplicate (LFBD) | Daily, before sample analysis. | Within control limits. If outside control limits, take corrective action. | Take remedial action(s) as defined in Section 17, repeat measurement and/or qualify data. |



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# STANDARD OPERATING PROCEDURE

*Title*

## Analysis of Anions By Ion Chromatography

*Reference Method No.:*

**EPA 300.0/EPA 300.0 B/SM 4110 B (23rd Edition, 2017)**

*Matrix/Matrices:*

**Liquid/Drinking Water/Solid**

*Document Control Number/Revision Number*

**SOP012A/Revision 5.1**

| | |
|---|---|
| Charles R. Morrow | 09/14/2021 |
| ***Approved By: Quality Assurance Manager*** | ***Date*** |
| Richard Hauk | 09/14/2021 |
| ***Approved By: General Manager*** | ***Date*** |
| A Sanders | 09/14/21 |
| ***Approved By: Laboratory Director*** | ***Date*** |

Standard Operating Procedures shall be reviewed at least once in two years or as needed to determine their continued suitability, compliance with applicable requirements, and to ensure that they reflect actual procedures being performed.



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**STANDARD OPERATING PROCEDURE**
**ANALYSIS OF ANIONS BY ION CHROMATOGRAPHY**

## 1.0 SCOPE AND APPLICATION

**1.1** This SOP describes the procedure for the determination of anions by Ion Chromatography in drinking water, solids (after extraction), leachates (when no acetic acid is used), ground, surface and saline waters as well as industrial and domestic aqueous wastes.

## 2.0 REPORTING LIMIT

**2.1** The Reporting Limit (RL) varies for individual anions and ranges from 0.25 mg/L to 1.0 mg/L in liquid as shown in the following table.

**2.2** Lower RLs may be achieved by utilizing a larger sample loop size for analytes that require a lower reporting limit for compliance purposes.

**Table – I**

| Anion | Liquid RL [mg/L] | Soil RL [mg/Kg] |
|---|---|---|
| Fluoride | 0.25 | 2.5 |
| Chlorate | 1.00 | 10.0 |
| Chloride | 1.00 | 10.0 |
| Chlorite | 1.00 | 10.0 |
| Nitrite as Nitrogen | 0.50 | 5.0 |
| Bromide | 0.50 | 5.0 |
| Nitrate as Nitrogen | 0.50 | 5.0 |
| ortho-Phosphate as P | 1.00 | 10.0 |
| Sulfate | 0.50 | 5.0 |

## 3.0 SUMMARY

**3.1** A well-mixed homogeneous sample is filtered through a 0.45µm membrane filter and introduced into the Ion Chromatograph.

**3.2** A fixed volume of the filtered sample is then carried by a Carbonate–Bicarbonate eluent through an analytical column into a conductivity detector. The resulting analyte peaks are quantified using a calibration curve.

**3.3** Solid samples are extracted using laboratory reagent water and the extract is filtered and analyzed by Ion Chromatography.

## 4.0 DEFINITIONS

**4.1** **Laboratory Reagent Blank/Method Blank (LRB/MBLK) –**An aliquot of reagent water that is treated exactly as a sample. The blank is exposed to all glassware, equipment, and reagents, etc. The method blank is used to define the level of laboratory background and reagent contamination.

**4.2** **Duplicate (DUP) –** A separate aliquot of the same sample from the same sample container.

**4.3** **Laboratory Fortified Blank/Laboratory Control Sample (LFB/LCS)** – A clean matrix spiked with a solution containing a mixture of seven anions of known concentration. An LFB is used to check extraction and/or method performance. For this test procedure, the LFB is equivalent to a Laboratory Control Sample (LCS).



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**4.4** **Laboratory Fortified Blank Duplicate/ Laboratory Control Sample Duplicate (LFBD/LCSD)** – LFBD/LCSD is the same as LFB/LCS and is used to check precision of the analytical method.

**4.5** **Laboratory Fortified Matrix (LFM)** – An aliquot of a sample from the analytical batch spiked with a solution containing a mixture of anions of interest at known concentration. An LFM is used to check the effect of matrix on the analytes of interest.

**4.6** **Limit of Detection (LOD)** – An estimate of the minimum amount of a substance that an analytical process can reliably detect (qualitatively). LOD is analyte and matrix specific. For purposes of this test procedure, the LOD is equivalent to the MDL.

**4.7** **Method Detection Limit (MDL) –** The method detection limit (MDL) is defined as the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results. For purposes of this method, the MDL is equivalent to NELAC's Limit of Detection [LOD]. See Section 19.0 METHOD PERFORMANCE for more information regarding LOD.

**4.8** **Practical Quantitation Limit/Reporting Limit (PQL)/RL** – The lowest concentration that can be reliably measured within specified limits of precision and accuracy for a specific laboratory analytical method during routine laboratory operating conditions.

**4.9** **Limit of Quantitation (LOQ)** – For purposes of this method, the LOQ is equal to the low standard used for initial calibration for an analytical method, and is equal to the Reporting Limit (RL).

## 5.0 INTERFERENCES

**5.1** Interferences resulting from co-elution of analytes that elute closely to one another.

**5.2** Ionic overloading can result in the saturation of the analytical column and/or detector may result in retention shifting of the analytes of interest. A sample dilution may eliminate or mitigate this type of interference problem.

**5.3** Sample matrices with high mineral content or hardness may influence the separation efficiency of the analytical column.

**5.4** Contaminated reagent water, eluent, reagents, glassware and other sample processing equipment may yield artifacts in the chromatogram resulting in elevated baseline or false positives.

**5.5** Acetate elutes early and may interfere with the analytes of interest. Disinfection byproducts can also be problematic in certain situations. These should be evaluated on a case-by-case basis when detected.

**5.6** Presence of chlorine dioxide in the sample may result in the formation of Chlorite and may pose interference problems in identifying the anions. Sample should be purged with an inert gas such as Argon or Helium, for about 5 minutes or longer if necessary, if prior knowledge of the process generating the sample is available.

**5.7** Proper glassware washing is essential to ensure reliable results. Refer to SATL#SOP003G for glassware washing, especially when making eluent and/or calibration standards.

**5.8** Samples consisting of complex matrices containing substances such as particulates and detergents, which may interfere with the sample analysis, may require a smaller volume to be analyzed.



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**5.9** Very late eluting ions, from chlorinated & ozonated matrices may carry over into the subsequent analytical run in the sequence. This should be monitored when obvious abnormal chromatographic responses are observed.

## 6.0 SAFETY

**6.1** Safety glasses and laboratory coats must be worn at all times while in the laboratory. In addition gloves and a face shield or goggles must be worn when dealing with toxic, caustic, and/or flammable chemicals.

**6.2** All chemical compounds should be treated as potential health hazards. The toxicity and/or carcinogenicity of each sample will most likely not be known. Therefore, it is imperative that each sample be handled as a potential health hazard.

**6.3** The analyst should familiarize themselves with all Safety Data Sheets (SDS), safety facilities, and equipment prior to beginning this procedure.

**6.4** Address any and all health and safety concerns to management before beginning this procedure.

## 7.0 EQUIPMENT AND SUPPLIES

**7.1** Ion Chromatograph equipped with anion separator analytical and guard columns, and conductivity detector – Dionex Corporation, or equivalent.

**7.2** Anion Suppressor device – Dionex Corp. or equivalent. Suppressor device to minimize the background noise.

**7.3** Poly Vial Sample cups – 5mL capacity to hold samples, QC standards, etc. Dionex, or Environmental Express, or equivalent.

**7.4** Poly Vial Filter caps with 0.2µm filters – Dionex, Environmental Express, or equivalent.

**7.5** Automated Sampler Cassettes – 5 mL capacity poly vial holder, Dionex, Environmental Express, or equivalent.

**7.6** Nylon filters 0.45µm syringe filters – Environmental express, BVA scientific or equivalent.

**7.7** Sample bottles – Glass or plastic 500 mL or 1000 mL capacity to hold sufficient volume to allow replicate sample analyses – BVA Scientific or equivalent.

**7.8** Disposable Pasteur pipettes – Fisher Scientific, or equivalent.

**7.9** Digestion tubes – Environmental Express, or Equivalent (for use in centrifuge).

**7.10** Filter Paper – Whatman No. 40 – Fisher Scientific, or Equivalent.

**7.11** Argon or Nitrogen gas, Industrial Grade- Matheson Tri-Gas, or Equivalent.

**7.12** 100 mL and 1 L Graduated Cylinder – Fisher Scientific, or Equivalent.

**7.13** 5 mL, 10 mL Class–A pipettes.

**7.14** Balance, Top Loading, Accurate to 0.0001g, Denver Instruments, or Equivalent.

## 8.0 REAGENTS AND STANDARDS

**8.1** Ultra-Pure Water, San Antonio Testing Laboratory, or Equivalent.

**8.2** Ion Chromatography Eluent solution – Eluent with Carbonate–Bicarbonate at 4.5mM and 1.4mM mixture respectively, Dionex or equivalent.

**8.2.1** Prepare working standard eluent according to manufacturer's instructions if purchased as a concentrate from a commercial supply vendor.

**8.2.1.1** Dilute 20 mL of the concentrated eluent to 2000 mL of ultrapure reagent water to obtain the working eluent concentration.



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**8.2.2** When eluent is not commercially available, prepare eluent as ***concentrate*** in the laboratory by mixing Sodium Carbonate and Sodium Bicarbonate salts as follows:

**8.2.2.1** Accurately weigh 0.954 g of Sodium Carbonate, and 0.235 g of Sodium Bicarbonate into reagent water and dilute to 2000 mL.

**8.3** Stock solutions such as those shown below may be prepared as described in section 12.3:

**8.3.1** Fluoride [$F^-$] 1000 mg/L
**8.3.2** Chlorate [$ClO_3^-$] 1000 mg/L
**8.3.3** Chloride [$Cl^-$] 1000 mg/L
**8.3.4** Chlorite [$ClO_2^-$] 1000 mg/L
**8.3.5** Nitrite as Nitrogen [$NO_2^-$–N] 1000 mg/L
**8.3.6** Bromide [$Br^-$] 1000 mg/L
**8.3.7** Nitrate as Nitrogen [$NO_3^-$–N] 1000 mg/L
**8.3.8** Phosphate [$PO_4^{\equiv}$–P] 1000 mg/L
**8.3.9** Sulfate [$SO_4^=$] 1000 mg/L

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

**9.1** Collect a representative sample in a clean 1-Liter, plastic or glass container for liquid sample and 4oz jar for solids.

**9.2** Preservation and holding time requirements for the anions being analyzed by this procedure are shown in the table below.

**Table – II**

| Anion | Preservation | Holding Time |
|---|---|---|
| Fluoride | None required * | 28 days |
| Chlorate | 50 mg/L EDA | 28 days |
| Chloride | None required * | 28 days |
| Chlorite | 50 mg/L EDA Cool to ≥0° C ≤6°C | 14 days |
| Bromide | None required * | 28 days |
| Nitrite-N | Cool to ≥0° C ≤6°C | 48 hours |
| Nitrate-N | Cool to ≤6°C | 48 hours |
| Combined [Nitrate–N/Nitrite–N] | to a pH < 2 [Conc. $H_2SO_4$] | 28 days |
| ortho-Phosphate-P | Cool to ≥0° C ≤6°C | 48 hours; Filter <15mins of collection** |
| Sulfate | Cool to ≥0° C ≤6°C | 28 days |

* It is recommended that all samples be cooled to ≤6°C and analyzed as soon as possible.

** qualify data if not filtered within 15mins of collection.

## 10.0 STORAGE

**10.1** Store samples until the time of analysis in a refrigerator at >0°C and ≤ 6°C to preserve sample integrity.

**10.2** Preserved samples have a maximum holding time of 28 days from the time of collection until analysis unless otherwise stated for specific analytes.

## 11.0 SAMPLE IDENTIFICATION



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**11.1** Samples are received from Sample Receiving with an In-House Chain of Custody form generated from the Laboratory Information Management System (LIMS). This includes client identification, sample number, and test to be performed.

**11.2** Each sample is assigned a unique number and a container number if more than one container is received.

## 12.0 CALIBRATION AND STANDARDIZATION

**12.1** Balance must be QC checked using S Class weights on each day of use.

**12.2** Ion Chromatograph must be calibrated prior to sample analysis either on the day of analysis or calibration verified on the day of analysis prior to sample analysis.

**12.3** Calibration Standards may be purchased where commercially available or prepared in the laboratory using Sodium and/or potassium salts as described below. Use two separate lots to prepare stock standards. Use one set to calibrate the instrument and use second to verify the instrument calibration.

**12.3.1** Bromide [$Br^-$] 1000 mg/L: Dissolve 0.1288 g Sodium Bromide [NaBr, CAS No. 7647-15-6] in reagent water and dilute to 100 mL in a volumetric flask.

**12.3.2** Chlorate [$ClO_3^-$] 1000 mg/L: Dissolve 0.1275 g Sodium Chlorate [$NaClO_3^-$, CAS No. 7775-09-9] in reagent water and dilute to 100 mL in a volumetric flask.

**12.3.3** Chloride [$Cl^-$] 1000 mg/L: Dissolve 0.1649 g Sodium Chloride [NaCl, CAS No. 7647-14-5] in reagent water and dilute to 100 mL in a volumetric flask.

**12.3.4** Chlorite [$ClO_2^-$] 1000 mg/L: Dissolve 0.1676 g Sodium Chlorite [Na $ClO_2^-$, CAS No. 7758-19-2] in reagent water and dilute to 100 mL in a volumetric flask.

**12.3.5** Fluoride [$F^-$] 1000 mg/L: Dissolve 0.2210 g Sodium Fluoride [NaF, CAS No. 7681-49-4] in reagent water and dilute to 100 mL in a volumetric flask.

**12.3.6** Nitrate [$NO_3^-$ –N] 1000 mg/L: Dissolve 0.6068 g Sodium Nitrate [$NaNO_3$, CAS No. 7631-99-4] in reagent water and dilute to 100 mL in a volumetric flask.

**12.3.7** Nitrite [$NO_2^-$ –N] 1000 mg/L: Dissolve 0.4926 g Sodium Nitrite [$NaNO_2$, CAS No. 7632-00-0] in reagent water and dilute to 100 mL in a volumetric flask.

**12.3.8** Phosphate [$PO_4^{\equiv}$–P] 1000 mg/L: Dissolve 0.4394 g Potassium Dihydrogenphosphate [$KH_2PO_4$, CAS No. 7778-77-0] in reagent water and dilute to 100 mL in a volumetric flask.

**12.3.9** Sulfate [$SO_4^=$] 1000 mg/L: Dissolve 0.1814 g Potassium Sulfate [$K_2SO_4$, CAS No. 7778-80-5] in reagent water and dilute to 100 mL in a volumetric flask.

**12.3.10** To prepare a mix of seven anions in a single working stock – add standard volumes of each of the stock solutions [12.3.1 – 12.3.7] as shown in Table – III, into a CLEAN 100mL volumetric flask and bring up to volume with reagent water.

***Note:*** *Stability of the standards – Stock standards stable for a minimum of 1 month and up to 3 months when stored at ≥0°C ≤6°C. Diluted working standards should be prepared weekly.*

**Table – III**

| WORKING STANDARDS PREPARATION FROM STOCK SOLNs. | | | | |
|---|---|---|---|---|
| **Anion** | **Initial Conc.** | **Initial Vol.** | **Working Std. Final Vol.** | **Cal. Std. Conc.** |
| Fluoride | 1000 mg/L | 2.0 mL | 100 mL | 20 mg/L |
| Chlorate | 1000 mg/L | 10.0 mL | 100 mL | 100 mg/L |



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| | | | | |
|---|---|---|---|---|
| Chloride | 1000 mg/L | 10.0 mL | 100 mL | 100 mg/L |
| Chlorite | 1000 mg/L | 10.0 mL | 100 mL | 100 mg/L |
| Nitrite-N | 1000 mg/L | 10.0 mL | 100 mL | 100 mg/L |
| Bromide | 1000 mg/L | 10.0 mL | 100 mL | 100 mg/L |
| Nitrate-N | 1000 mg/L | 10.0 mL | 100 mL | 100 mg/L |
| ortho-Phosphate-P | 1000 mg/L | 20.0 mL | 100 mL | 200 mg/L |
| Sulfate | 1000 mg/L | 10.0 mL | 100 mL | 100 mg/L |

**12.4** Refer to Table–A, Appendix B of this SOP for instructions on the preparation of the calibration curve with varied concentrations of individual anions.

**12.5** Prior to sample analysis on the Ion Chromatograph, a set of calibration standards is analyzed following the guidelines in Table–A, Appendix B.

**12.5.1** Refer to section 13.1 for IC operating conditions and eluent concentration.

**12.6** Calculate the Relative Standard Error (%RSE) of the calibration curve for analytes with linear or quadratic fits. Determine the %RSE using the equation below.

$$\%\ RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where,

$x_i$ = True value for the calibration standard
$x'_i$ = Measured concentration of the calibration standard
n = Number of calibration points
p = Number of terms in the fitting equation
(Average = 1, Linear = 2, Quadratic = 3)

**12.7** Coefficient of determination must be >0.920 (which approximately corresponds to the 35% RSD limit set forth in the reference method). If this cannot be achieved, the calibration is unacceptable and recalibration is necessary after remedial action to correct the problem.

**12.8** Calculate the Relative Error (%RE) for those analytes are calibrated using linear or quadratic curve fits and determine the coefficient of determination using the following equation.

$$\%Relative\ Error = \frac{x'_i - x_i}{x_i} \times 100$$

Where,

$x_i$ = True value for the calibration standard
$x'_i$ = Measured concentration of the calibration standard



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**12.9** The relative error percent must be calculated for two of the calibration levels, i.e., the low calibration standard and the mid-point calibration standard. The acceptance criteria for low standard is 50% and the mid-point standard is 35%.

**12.10 Retention Time windows**

**12.10.1** Retention time windows are established by analyzing a mid-point calibration standard [5 mg/L] initially. Retention time is inversely proportional to concentration, use caution when establishing RT windows.

**12.10.2** A suggested method of establishing RT windows is to calculate three times the standard deviation of the actual retention time of the anion of interest, measured over the course of a day.

**12.10.3** Retention time windows should be re-assessed every time a new IC column is installed and/or new eluent is prepared, or after high concentration samples have been analyzed and integration parameters adjusted to reflect the correct RT windows.

**12.10.4** Analyte elution order in an IC run is shown in the table below with approximate retention times corresponding to IC conditions described in section 13.1. Retention time may shift with aging column or other conditions described in the above sections and should be updated as needed.

**Table IV**

| Peak No. | Anion | Retention Time [min] |
|---|---|---|
| 1 | Fluoride | 3.120 |
| 2 | Chlorite | 3.624 |
| 3 | Chloride | 4.374 |
| 4 | Nitrite-N | 5.190 |
| 5 | Chlorate | 5.244 |
| 6 | Bromide | 6.054 |
| 7 | Nitrate-N | 6.820 |
| 8 | ortho-Phosphate-P | 9.0637 |
| 9 | Sulfate | 11.070 |

**12.11 Initial Calibration**

**12.11.1** Prior to sample analysis, the IC system is calibrated using multiple calibration points. The standards may be prepared as described in the appendix of this SOP or are purchased from approved vendors.

**12.11.2** Refer to Table A, Appendix B for initial calibration curve points for varied concentrations of individual anions of interest. Standards typically range from 0.25 mg/L to 40 mg/L for water and solid matrices.

**12.11.3** Analyze all calibration standards as type “Standards” and save the results file on the computer.

**12.11.4** A calibration curve with a correlation coefficient of 0.995 or greater for individual anions of interest is deemed valid and sample analysis may begin.

**12.11.4.1** When using a non-linear curve, a linear calibration range is not applicable.

**12.12 Calibration Verification – Initial and Continuing [ICV/CCV]**



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**12.12.1** The initial calibration is verified at the beginning of each working day or a batch of 10 samples using a second source calibration verification standard.

**12.12.2** Initial calibration of the IC system is verified by analyzing a single point calibration standard [CCV] at a mid-calibration level.

**12.12.2.1** Prepare 50 mL of CCV standard fresh on the day of analysis. Dilute 2.5 mL of the working stock standard (section 12.3.8) to 50 mL in ultrapure water to obtain a 5 mg/L concentration standard. The source of this standard may be the same as the initial calibration stock solution.

**12.12.2.2** Prepare 50 mL of ICV standard on the day of initial calibration using working stock standard prepared from a source (section 12.3) other than that used for preparing the initial calibration curve. Dilute 2.5 mL of the second source working stock to 50mL in ultrapure water. The concentration of this verification standard is 5 mg/L.

**12.12.3** In this SOP, the mid-point standard used is 5mg/L [except 1.0 mg/L for Fluoride]. The acceptance criteria for the ICV [or CCV] standard is ±10% of true value.

**12.12.4** If the ICV standard meets acceptance criteria of ±10% deviation [%D] then the initial calibration is deemed valid and the calibration factor can be used to quantitate the field samples.

**12.12.4.1** If the ICV exceeds ±10% the expected value, then evaluate for possible spiking errors, calculation errors, injection malfunction, etc. If no obvious problems are identified, the stock solution may be suspect. Prepare fresh stock solution, re-analyze and verify calibration. Recalibration of the instrument is necessary if the second attempt of ICV still exceeds expected range.

**12.12.4.2** Failure of the ICV to meet the ±10% expected value requires instrument recalibration.

**12.12.5** A continuing calibration verification standard must be analyzed every 10 samples and at the end of the analytical sequence (ending CCV) and must meet the acceptance criteria of ±10% deviation. An instrument blank must be run before the ending CCV.

**12.12.6** If the CCV fails to meet the acceptance criteria, reanalyze the CCV one more time after performing routine maintenance on the analytical system before recalibrating the instrument. If the CCV fails the second time, then the initial calibration is deemed invalid and system must be recalibrated as in section 12.7.

**12.12.6.1** Further corrective actions such as cleaning the IC system, preparing new eluent, conditioning the analytical column, and/or suppressor, etc. may be performed. However, after major maintenance is done on the system, two consecutive CCV standards must be analyzed and both must meet the acceptance criteria. If both consecutive CCV standards meet the acceptance criteria then samples can be analyzed on the system without recalibrating the system as in section 12.7.

**12.12.6.2** If any one of the two CCV standards fail to meet the acceptance criteria then a new initial calibration curve must be analyzed and processed prior to sample analysis.

**12.13** Column overloading [separation capacity] may result in non–linear response. In such cases system maintenance may be required after the evaluation is determined to be column related and not related to calibration standard solution.

**12.14** When capacity [i.e., column overloading] of analytical [separator] column is exceeded non-linear responses may result at which time the analytical column should be replaced.



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### 12.15 Recommended system maintenance

**12.15.1** In cases where the initial calibration does not meet the acceptable correlation coefficient criteria of 0.995 or better or the CCV is not within ±10% deviation criteria, system maintenance is required. A short list of the remedial actions is given below:

a. Check system backpressure for clogging and air bubbles and clean as necessary.
b. Check the sample and system tubing.
c. Prime the pump and check pump valves and clean if necessary.
d. Check the concentration of the eluent and fill the reservoir if necessary.
e. Refer to the operational manual for other maintenance and suggested troubleshooting techniques.
f. If none of these maintenance tasks resolve the problems, replace the column, or contact the manufacturer for either technical help or service call.

## 13.0 PROCEDURE

### 13.1 Analytical System

**13.1.1** Analytical system is comprised of an Ion Chromatograph equipped with a suppressor device and a conductivity cell. The IC system has an analytical column for anion separation and a guard column to protect the analytical column and extend the life of the analytical column.

**13.1.2** Software for data acquisition and data processing: refer to and follow manufacturer's instructions on the operation of the IC system and software.

**13.1.2.1** The following are typical settings in IC program. These values may be adjusted to achieve better resolution and/or sensitivity toward the instrument.

| Parameter | Value |
|---|---|
| Analytical Column | IonPac AS 22 [4x250mm] |
| Guard Column | IonPac AG 22 [4x50mm] |
| Suppressor Type | ASRS 4mm |
| Suppressor Current | 31–34 [mA] |
| Pressure Lower Limit | 0 [PSI] |
| Pressure Upper Limit | 3000 [PSI] |
| Pump Inject Valve State | Load Position |
| Data Collection Rate | 5.0 [Hz] |
| Cell Temperature Nominal | 35.0 [°C] |
| Column Temperature Nominal | 40.0 [°C] |
| Pump ECD Carbonate [Eluent] | 4.5 mM |
| Pump ECD Bicarbonate [Eluent] | 1.4 mM |
| Pump ECD Recommended Current | 31–34 [mA] |
| Pump Flow | 1.20 [ml/min] |
| Sample Loop Size | 10 µL |
| Expected background Conductivity | 20 – 23µS |

### 13.2 Sample Preparation and Equipment

**13.2.1** Samples are collected and preserved as per sections 9.0 and 10.0.

**13.2.2** Allow samples to equilibrate to room temperature before starting the analysis. Do not allow samples to sit at room temperature for more than 6 cumulative hours.



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**13.2.3** Samples must be filtered through a 0.45µm anion free filter prior to taking a sample aliquot, to prevent clogging of the analytical system.

**13.2.4 Solid Sample Extraction**

**13.2.4.1** Weigh a 5.0 +/- 0.1g of a well homogenized sample into a suitable container such as a 50 mL digestion cup. Add 50 mL of ultrapure reagent to the digestion cup and shake the container with hand for one minute.

**13.2.4.2** Place the sample containers on a mechanical shaker with lids closed tightly and shake for 15 minutes at high speed.

**13.2.4.3** Remove samples from the shaker and allow samples to settle or centrifuge to settle suspended material. Filter the slurry using 0.45µm membrane syringe filter.

**13.2.5** Transfer 5 mL of the filtered sample into an auto-sampler sample cup equipped with a filter cap for analysis. Follow manufacturer's instructions for setting up the auto-sampler and loading sample cassettes.

**13.2.6** Power on the IC and allow the system to equilibrate by priming the pump and allowing the eluent to pump through the system for 30 minutes.

**13.2.7** Set-up a sample sequence on the Chromeleon software and perform "Ready Checks" built into the Dionex software prior to initiating sample run.

**13.2.8** Analyze a reagent water blank, calibration verification standard at the beginning of each sequence on each day of use. Instrument [re] calibration may be required depending on the CCV standard result. If CCV fails to meet the acceptance criteria, follow the procedure described in section 12.0.

**13.2.9** Analyze calibration standards from low concentration to high concentration to avoid carry over issues between standards.

**13.2.10** Field samples may be analyzed immediately following calibration standards and after the calibration curve has been established and verified to meet acceptance criteria.

**13.2.11** Prepare a laboratory reagent blank [LRB], laboratory fortified blank [LFB/LCS], laboratory fortified matrix [LFM/MS], duplicate [Dup], etc., along with field samples in a batch.

**13.2.12** Using peak areas of the analytes of interest, sample concentration is calculated via initial calibration responses as in section 14.0.

**13.2.13** When sample concentration exceeds the calibration range of a particular anion, sample must be diluted appropriately so that the concentration will fall within the calibration range.

**13.2.14** When doubt exists over the identification of a peak in the chromatogram, then sample dilution and fortification may be used for confirmation.

**13.3 Data review and Data processing**

**13.3.1** All raw data must be reviewed for integration errors by the software to ensure that peaks are correctly assigned.

**13.3.2** Use peak area responses of the detected analytes of interest to compute the concentration of the field and QC samples.

**13.3.3** Report values that fall within the lowest and highest calibration points. Sample concentrations that fall beyond the highest calibration point must be diluted and re-analyzed.

**13.3.4** Report all results in mg/L for liquid and mg/Kg for solid matrices.

**13.3.5** Report results for Nitrate, Nitrite, and Phosphate as Nitrate as Nitrogen, Nitrite as Nitrogen, and ortho-Phosphate as Phosphorus respectively in the analytical report.



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**13.3.6** Refer to section 14.0 for the calculation of sample concentrations and LFB, LFM, etc., recoveries.

## 14.0 DATA ANALYSIS AND CALCULATIONS

**14.1** Report the concentrations of anions of interest in field samples directly from the instrument generated data in mg/L taking into account any sample factors such as dilutions, extraction factor, etc.

**14.1.1** Calculate the analyte concentration

Liquid/Water Samples:

$$Anion\ \ (mg/L)\ \ = Instrument\ \ \ Reading\ \ \ \times DF$$

Solid Samples:

$$Anion\ \ (mg/kg)\ \ = \frac{Instrument\ \ Reading\ \ \times DF\ \times FV}{Sample\ \ Wt\ \ (g)}$$

Where:

DF – Dilution factor
FV – Final extract volume

**14.2** Laboratory Fortified Blank/Laboratory Control Sample [LFB/LCS] Recovery

$$Spike\ \ Recovery\ \ = \frac{LFBR}{LFBA} \times 100$$

Where:

LFBR = LFB Spike Result
LFBA = Spike Added

**14.3** Laboratory Fortified Matrix/Matrix Spike [LFM/MS] Recovery

$$Spike\ \ Recovery\ \ = \frac{LFMR\ \ - SR}{LFM\ \ SA} \times 100$$

Where:

LFMR = LFM Result
SR = Sample Result [Un-spiked field sample]
LFMSA = LFM Spike Added

**14.4** Relative Percent Difference – Duplicate samples

$$RPD = \left| \frac{SR - SDR}{\left( \frac{SR + SDR}{2} \right)} \right| \times 100$$

Where:

RPD = Relative Percent Difference

SR = Sample Result
SDR = Sample Duplicate Result

## 15.0 QUALITY CONTROL

**15.1** Laboratory's initial demonstration of capability is documented by performing an MDL study and a Quality Control Check sample [purchased from a source external to the lab].

**15.2** The Limit of Quantitation (LOQ) for this method is 0.5 mg/L for all anions except Fluoride and orthophosphate, which are at 0.1 mg/L, and 1.0 mg/L respectively for liquid samples. LOQ for solids is 5mg/kg for all except fluoride and orthophosphate, which are at 1mg/kg and 10mg/kg respectively.

**15.3** A minimum of one method blank and one Laboratory Fortified Blank (LFB) and Laboratory Fortified Blank (LFB-DUP) must be analyzed for every batch of not more than 10 samples for liquid and solid samples

**15.4** Chemicals and standards must be entered upon receipt into the LIMS and assigned a number. The containers must be dated when first opened and discarded by the expiration date. Any chemical or standard that fails to meet Quality Control requirements should be returned to the manufacturer for replacement.

**15.5** Working standards including those prepared/used daily must be entered and assigned a number in LIMS when prepared. All working standards must be discarded by the expiration date.

**15.6** Any working standard that fails to meet Quality Control requirements must be discarded and re-prepared. If the working standard continues to fail, contact the manufacturer of the chemicals, and if necessary order new supplies.

**15.7** All Certificates of Analysis must be retained.

**15.8** All analytical records must be backed up monthly as a minimum.

## 16.0 ACCEPTANCE CRITERIA

**16.1** Laboratory Reagent Blank concentration of any anion of interest must be less than the corresponding MDL value.

**16.2** Calculate the LFB recovery. The acceptable range for the LFB is 90-110%.

**16.3** Calculate the LFM recovery. The acceptable range for the LFM is 90%-110%. If the concentration of un-spiked sample is ≥4 times the LFM spike concentration, the matrix spike recovery is not required to be calculated and reported.

**16.4** Determine the RPD for the sample and sample duplicate. The acceptable range for the RPD is <20.

**16.5** Refer to Table–C for QC acceptance criteria.

**16.6** The acceptance limits for Demonstration of Capability (DOC) by this method are %RSD <10 (precision) of 4 QC replicates, and an average recovery range of 90-110% (accuracy) of the true concentration. DOCs must take into account all sample preparation steps.

## 17.0 CORRECTIVE ACTIONS FOR NON-CONFORMANCE DATA

**17.1** Should a sample become contaminated or compromised, the preparation and analysis shall be terminated and repeated with a fresh sample aliquot. A Corrective Action must be completed to document the actions taken.



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**17.2** When Quality Control measures fail, and the clients' results are affected, the client will be advised that the results may not be reliable. It may be necessary based on clients' needs to recollect the sample and submit at a later time. If the client is unable to recollect a sample, the data will be released with the appropriate documentation. The laboratory staff will complete a Corrective Action form to document this occurrence.

**17.3** When QC samples do not fall within the acceptable range, the analyst shall review the data for obvious errors such as calculations, preparation errors, or inadvertent spiking errors or other such causes that are not resultant of a systemic failure. The data may be released with a qualifying statement after concurring with the quality manager. A Corrective Action must be completed documenting the actions taken when the root cause identified is deemed detrimental to the analysis.

## 18.0 HANDLING NON-CONFORMANCE DATA

**18.1** Non-conformance data are monitored and resolved by identifying categories such as system based, methods based, preparative method based, etc., and are resolved once the problematic areas are identified.

## 19.0 METHOD PERFORMANCE

**19.1** A method detection limit study is performed, initially and verified quarterly thereafter for all analytes that are listed in **TABLE I** of this method.

**19.2** During the beginning of each quarter, two replicate samples of organic free reagent water are spiked with a known amount of target analytes at the concentration used in the initial determination of the MDL and analyzed on the GC/FID analytical system.

**19.3** If any analytes are repeatedly not detected in the quarterly spiked sample analyses, or do not meet the qualitative identification criteria of the method, then this is an indication that the spiking level is not high enough and should be adjusted.

**19.4** Prepare and analyze seven spike replicates and seven method blanks on at least three different days carried out through sample preparation steps. Existing routine method blanks can be used for this study.

**19.5** The validity of the MDL shall be confirmed by qualitative identification of the analyte.

**19.6** A minimum of seven MDL replicate samples and seven method blanks are used to calculate the MDL values. For purposes of this method, the MDL is equivalent to TNI's Limit of Detection (LOD).

Calculate the $MDL_S$ (MDL spiked samples) value using the following formula:

$$MDL_s = t_{[n-1,\ 1-\infty = 0.99]}\ S_s$$

Where,

$t_{[n-1,\ 1-\infty = 0.99]}$ = Student's t value for the 99% confidence level with n-1 degrees of freedom,
n = number of replicates.
$S_s$ = the standard deviation of the replicate analyses.

Calculate the $MDL_B$ (MDL blank samples) values using the following formula:



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$$MDL = t_{[n-1,\ 1\text{-alpha} = 0.99]}\ S_b$$

Where,

$t_{[n-1,\ 1\text{-alpha} = 0.99]}$ = Student's t value for the 99% confidence level with n-1 degrees of freedom,
n = number of replicates.
$S_b$ = the standard deviation of the replicate method blank sample analyses.

| Number of Replicates | Degrees (degrees of freedom) | $t_{(n-1,\ 0.99)}$ |
|---|---|---|
| 7 | 6 | 3.143 |
| 8 | 7 | 2.998 |
| 9 | 8 | 2.896 |
| 10 | 9 | 2.821 |
| 11 | 10 | 2.764 |

**19.7** Current MDL values for method analytes in this SOP can be found in the SATLMDL.xls spreadsheet.

## 20.0 POLLUTION PREVENTION

**20.1** Each method is evaluated prior to use in order to minimize waste volume and toxicity.
**20.2** A non-hazardous or less toxic substitute may be used whenever possible.
**20.3** Purchase only the amount of chemical that is actually needed or that will be used to eliminate the cost of disposal later.

## 21.0 WASTE MANAGEMENT

**21.1** Toxic waste must never be disposed of down the drain.
**21.2** Waste generated from sample analysis must be segregated if the process knowledge indicates the presence of any of the hazardous components listed in Table–1, 40 CFR 261.24 and exceed the limits set in the table.
**21.3** When disposing samples the analyst must follow current revision of the "Laboratory Waste Handling and Disposal" SOP (SATL#007G) for detailed disposal procedures.
**21.4** All chemicals and containers must be properly identified and labeled at all times to eliminate ambiguity and cost of disposal of unknowns. If an unknown chemical or container is discovered, label it as 'unknown' and attach a note detailing any information about what the chemical may be, what test it may have been used for, and where it was found. If you find an unlabeled chemical that has crystallized or there is any other indication that it may be unstable, notify management immediately.
**21.5** Generally, empty chemical containers are not considered hazardous waste. Check with management if one such container is found and in doubt. To dispose of the container in the regular trash the container must be completely empty and tripled rinsed
**21.6** The waste drums are picked up upon notification and a copy of the report is submitted to the waste management company.



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## 22.0 REFERENCES

**22.1** Method 300.0, "Determination of Inorganic Anions by Ion Chromatography", Environmental Monitoring Systems Laboratory, Office of Research and Development, United States EPA, Revision 2.1, August 1993.

**22.2** "Anions by Ion Chromatography" Method 4110B, Standard Methods for the Examination of Water and Wastewater, 20th Edition, Standard Methods 1998.

**22.3** "Anions by Ion Chromatography" Method 4110B, Standard Methods for the Examination of Water and Wastewater, 23rd Edition, Standard Methods 2017.

**22.4** EPA 9056A, Determination of Inorganic Anions by Ion Chromatography, Revision 1, February 2007.

## 23.0 REVISION HISTORY

**23.1** New revision of the method.

**23.2** Revision 2 from Revision 1: changes stemming from an annual review, and the most recent TCEQ on-site assessment.

**23.3** Annual method revision # 2.1, added Drinking Water and Solid matrices in this SOP.

**23.4** Annual revision #2.1.0 – revised for language and redundancy. Deleted the preparation of intermediate range eluent concentrate from section 8.2. Added clarity on eluent preparation.

**23.5** Annual Revision 2014: revised section 6.0 and minor language changes throughout SOP. Included recommendations from NELAC and internal audits.



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**STANDARD OPERATING PROCEDURE**
**ANALYSIS OF ANIONS BY ION CHROMATOGRAPHY**

## APPENDIX A
## SOP History and Version Control

| Version | Date of Review/Revision | Review/Revision Approved by | Brief Description |
|---|---|---|---|
| 2.1.3 | 08/14/2015 | M. Bernard | Revised to reflect change in procedure for calibration curve (12.7.4.1). Addition of Appendix A to reflect SOP history and version control. Change Appendix 1 to Appendix B. |
| 2.5 | 07/25/2016 | M. Bernard | Revision of title page. |
| 3.0 | 06/19/2017 | M. Bernard | Biennial review and waste disposal protocol. |
| 3.1 | 10/13/2017 | M. Bernard | Addition of chlorate and chlorite. |
| 4.0 | 07/12/2019 | M. Bernard | Biennial review; revision of title page, reference method update. |
| 5.0 | 04/16/2021 | A.Rosecrance | Biennial review; update title page; change MSDS to SDS |
| 5.1 | 09/14/2021 | C. Morrow | Revised the following:<br>Section 4 – Definition for MDL<br>Section 12 – Add details to calibration process to include %RE.<br>Section 19 – Update MDL procedure.<br>Section 22 – Update reference method information. |



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**STANDARD OPERATING PROCEDURE**
**ANALYSIS OF ANIONS BY ION CHROMATOGRAPHY**

**APPENDIX – B**

## Table– A (a)

Example preparation of initial calibration curve for Ion Chromatograph: **Liquid/Solid**

| CAS No.: | 7681-49-4 | | Anion : | **Fluoride** | |
|---|---|---|---|---|---|
| ICAL Pnt | Final Conc. | Final vol | Init Conc | Init. Vol [mL] | Init Vol [uL] |
| 1 | 0.1 | 50 | 20 | | 250 |
| 2 | 0.2 | 50 | 20 | | 500 |
| 3 | 1 | 50 | 20 | 2.5 | |
| 4 | 2 | 50 | 20 | 5 | |
| 5 | 4 | 50 | 20 | 10 | |
| 6 | 8 | 50 | 20 | 20 | |

| CAS No.: | 7647-14-5 | | Anion : | **Chloride** | |
|---|---|---|---|---|---|
| ICAL Pnt | Final Conc. | Final vol | Init Conc | Init. Vol [mL] | Init Vol [uL] |
| 1 | 0.5 | 50 | 100 | | 250 |
| 2 | 1 | 50 | 100 | | 500 |
| 3 | 5 | 50 | 100 | 2.5 | |
| 4 | 10 | 50 | 100 | 5 | |
| 5 | 20 | 50 | 100 | 10 | |
| 6 | 40 | 50 | 100 | 20 | |

| CAS No.: | 7632-00-0 | | Anion : | **Nitrite-N** | |
|---|---|---|---|---|---|
| ICAL Pnt | Final Conc. | Final vol | Init Conc | Init. Vol [mL] | Init Vol [uL] |
| 1 | 0.5 | 50 | 100 | | 250 |
| 2 | 1 | 50 | 100 | | 500 |
| 3 | 5 | 50 | 100 | 2.5 | |
| 4 | 10 | 50 | 100 | 5 | |
| 5 | 20 | 50 | 100 | 10 | |
| 6 | 40 | 50 | 100 | 20 | |

| CAS No.: | 7647-15-6 | | Anion : | **Bromide** | |
|---|---|---|---|---|---|
| ICAL Pnt | Final Conc. | Final vol | Init Conc | Init. Vol [mL] | Init Vol [uL] |
| 1 | 0.5 | 50 | 100 | | 250 |
| 2 | 1 | 50 | 100 | | 500 |
| 3 | 5 | 50 | 100 | 2.5 | |
| 4 | 10 | 50 | 100 | 5 | |
| 5 | 20 | 50 | 100 | 10 | |
| 6 | 40 | 50 | 100 | 20 | |

| CAS No.: | 7631-99-4 | | Anion : | **Nitrate-N** | |
|---|---|---|---|---|---|
| ICAL Pnt | Final Conc. | Final vol | Init Conc | Init. Vol [mL] | Init Vol [uL] |
| 1 | 0.5 | 50 | 100 | | 250 |
| 2 | 1 | 50 | 100 | | 500 |
| 3 | 5 | 50 | 100 | 2.5 | |
| 4 | 10 | 50 | 100 | 5 | |
| 5 | 20 | 50 | 100 | 10 | |
| 6 | 40 | 50 | 100 | 20 | |

| CAS No.: | 7778-77-0 | | Anion : | **Phosphate-P** | |
|---|---|---|---|---|---|
| ICAL Pnt | Final Conc. | Final vol | Init Conc | Init. Vol [mL] | Init Vol [uL] |
| 1 | 1.0 | 50 | 200 | | 250 |
| 2 | 2.0 | 50 | 200 | | 500 |
| 3 | 10 | 50 | 200 | 2.5 | |
| 4 | 20 | 50 | 200 | 5 | |
| 5 | 40 | 50 | 200 | 10 | |
| 6 | 80 | 50 | 200 | 20 | |

| CAS No.: | 7778-80-5 | | Anion : | **Sulfate** | |
|---|---|---|---|---|---|
| ICAL Pnt | Final Conc. | Final vol | Init Conc | Init. Vol [mL] | Init Vol [uL] |
| 1 | 0.5 | 50 | 100 | | 250 |
| 2 | 1 | 50 | 100 | | 500 |
| 3 | 5 | 50 | 100 | 2.5 | |
| 4 | 10 | 50 | 100 | 5 | |
| 5 | 20 | 50 | 100 | 10 | |
| 6 | 40 | 50 | 100 | 20 | |

**STAGGERED CALIBRATION CURVE FOR 7 ANIONS [mg/L]**

| Anion | Ical-1 | Ical-2 | Ical-3 | Ical-4 | Ical-5 | Ical-6 |
|---|---|---|---|---|---|---|
| Fluoride | 0.10 | 0.2 | 1.0 | 2.0 | 4.0 | 8.0 |
| Chloride | 0.50 | 1.0 | 5.0 | 10 | 20 | 40 |
| Nitrate-N | 0.50 | 1.0 | 5.0 | 10 | 20 | 40 |
| Bromide | 0.50 | 1.0 | 5.0 | 10 | 20 | 40 |
| Nitrite-N | 0.50 | 1.0 | 5.0 | 10 | 20 | 40 |
| Phosphate-P | 1.00 | 2.0 | 10 | 20 | 40 | 80 |
| Sulfate | 0.50 | 1.0 | 5.0 | 10 | 20 | 40 |

Calibration Standard Solutions:

| **Calibration Curve Point** | **1** | **2** | **3** | **4** | **5** | **6** |
|---|---|---|---|---|---|---|
| **DI Water [mL]** | 49.75 | 49.50 | 47.50 | 45.00 | 40.00 | 30.00 |
| **Stock Standards Volume [mL]:** | 0.25 | 0.50 | 2.50 | 5.00 | 10.00 | 20.00 |
| **Final Calibration Standand Volume [mL]:** | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 |



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SAN ANTONIO TESTING LABORATORY, INC.

# STANDARD OPERATING PROCEDURE

*Title*

**Analysis of pH (Electrometric)**

*Method No.:*

**EPA 150.1/EPA 9045D/SM4500–$H^+$ B (23rd Edition, 2017)**

*Matrix/Matrices:*

**Liquid/Solid**

*Document Control Number/Revision Number*

**SOP014A/Revision 5.2**

| | |
|---|---|
| Charles R. Morrow | 09/07/2021 |
| ***Approved By: Quality Assurance Manager*** | ***Date*** |
| Richard Hauk | 09/07/2021 |
| ***Approved By: General Manager*** | ***Date*** |
| | 09/07/2021 |
| ***Approved By: Laboratory Director*** | ***Date*** |

Standard Operating Procedures shall be reviewed at least once in two years or as needed to determine their continued suitability, compliance with applicable requirements, and to ensure that they reflect actual procedures being performed.



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**STANDARD OPERATING PROCEDURE**
**ELECTROMETRIC MEASUREMENT OF PH IN LIQUID AND SOLID MATRICES**

## 1.0 SCOPE AND APPLICATION

**1.1** This SOP describes the measurement of pH of liquids, solids, and wastes from domestic and industrial sources.

**1.2** Method SM 4500–$H^+$ and 150.1 are used to measure the pH of drinking water, surface water, saline water, domestic and industrial wastewater.

**1.3** Method 9040C is used to measure the pH of aqueous wastes and multiphase wastes with at least 20% of the total volume being aqueous.

**1.4** Method 9045D is a procedure for measuring the pH in soil and waste samples. Waste samples may be solids, sludges, or non-aqueous liquids. When water is present, it must be less than 20% of the total volume of the sample.

## 2.0 REPORTING LIMIT

**2.1** The pH meter/electrode reads pH values from 0 – 14.

## 3.0 SUMMARY

**3.1** The pH of a sample is determined electrometrically using a combination electrode.

**3.2** The pH meter is calibrated using a series of standard buffer solutions of known pH.

**3.3** Solid and waste samples are mixed with reagent water and the pH of the resulting aqueous solution is measured.

## 4.0 DEFINITIONS

**4.1** **pH:** is a measure, at a given temperature, of the intensity of the acidic or basic character of a solution.

**4.2** **Batch –** The batch is a set of samples of the same matrix processed using the same procedures and reagents within the same time period. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible.

**4.3** **Duplicate (DUP) –** A separate aliquot of the same sample from the same sample container that is analyzed separately with identical procedures. Analyses of a duplicate indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

**4.4** **Laboratory Control Sample (LCS)/Verification Buffer Standard (VBS) –** A buffer solution of known pH with different lot number or different vendor is used as an LCS/VBS.

## 5.0 INTERFERENCES

**5.1** Generally, solution interferences from: color, turbidity, colloidal matter, oxidants, reductants, and/or high salinity are not a concern for the glass electrode.

**5.2** Samples with very low or very high pH levels may yield incorrect reading.

**5.3** Particulate matter or oily materials adhering to the electrode may also hinder electrode function. Usually, gentle wiping or detergent washing followed by rinsing with distilled water can remove such coatings. Additional treatment with 1.17 N hydrochloric acid (1:10 concentrated acid) may be needed to thoroughly clean the electrode.



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**5.4** Temperature fluctuations will cause measurement errors. The use of an instrument that has automatic temperature compensation is recommended.
**5.5** During calibration, pH buffers should be used only once.
**5.6** Never use a filling solution that contains silver with electrodes that require filling solutions.

## 6.0 SAFETY

**6.1** Safety glasses and laboratory coats must be worn at all times while in the laboratory. In addition gloves and a face shield or goggles must be worn when dealing with toxic, caustic, and/or flammable chemicals.
**6.2** A partial facemask should be worn when working with samples suspected to contain high levels of volatile organics, such solvents, and samples contaminated with gasoline, etc.
**6.3** All chemical compounds should be treated as potential health hazards.
**6.4** The toxicity and/or carcinogenicity of each sample will most likely not be known. Therefore, it is imperative that each sample be handled as a potential health hazard.
**6.5** The analyst should familiarize themselves with all Safety Data Sheets (SDS), safety facilities, and equipment prior to beginning this procedure.
**6.6** Please address any and all health and safety concerns to management before beginning this procedure.

## 7.0 EQUIPMENT AND SUPPLIES

**7.1** Orion Five Star pH/ISE meter, Thermo-Fisher, or equivalent.
**7.2** Digestion Cups, 50mL Capacity, Environmental Express, Catalog No. SC475
**7.3** Polystyrene Beakers, 5mL, Fisher Scientific, Catalog No. 08-732-119, or Equivalent
**7.4** Electrode Storage Solution, Fisher Scientific, Catalog No. SE40-1, or Equivalent
**7.5** Reference Electrode Filling Solution, Fisher Scientific, Catalog No. 13-641-755
**7.6** Beakers of various sizes from 50mL onwards.
**7.7** Balance, Top Loading, Accurate to 0.01g, Denver Instruments, or Equivalent.
**7.8** Stir plate – Fisher scientific or equivalent.

## 8.0 REAGENTS AND STANDARDS

**8.1** Ultra Pure Water, ASTM Type II, San Antonio Testing Laboratory.
**8.2** Buffer Solution, 4.00, Fisher Scientific, Catalog No. SB101-500, or Equivalent
**8.3** Buffer Solution, 7.00, Fisher Scientific, Catalog No. SB107-500, or Equivalent
**8.4** Buffer Solution, 10.00, Fisher Scientific, Catalog No. SB115-500, or Equivalent
**8.5** Buffer Solution, 7.00, Hach Company, Catalog No. 22834-49, or Equivalent

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

### 9.1 Sample Collection and Holding Times[1]

#### 9.1.1 Aqueous Samples

[1] pH measurements should be made in the field when collecting samples as it may vary with various environmental factors such as temperature, dissolved carbon dioxide, etc.



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**9.1.1.1** Sample may be collected in a plastic or glass containers with screw cap lids.

**9.1.1.2** No preservation is required for the pH analysis; however, samples must be analyzed as soon as practically possible.

**9.1.2 Solid Samples**

**9.1.2.1** Bulk Solid (soils and sediment) samples are collected in wide mouth borosilicate glass jars.

**9.1.2.2** No preservation is required for the pH analysis; however, samples must be analyzed as soon as practically possible.

## 10.0 STORAGE

**10.1** Aqueous and solid [soils and sediment] samples are stored until the time of analysis in a refrigerator at >0°C but ≤ 6°C to preserve sample integrity if analysis cannot begin soon after sample receipt.

## 11.0 SAMPLE IDENTIFICATION

**11.1** Samples are received from Sample Receiving with a work order form generated from the Laboratory Information Management System (LIMS). This includes client identification, sample number, and tests to be performed under each department

**11.2** Each sample is assigned a unique number and a container number if more than one container is received.

## 12.0 CALIBRATION AND STANDARDIZATION

**12.1** The pH meter must be calibrated each day of use.

**12.1.1** To calibrate the pH meter:

**12.1.1.1** Fill three disposable digestion tubes with approximately 10 mL of the appropriate buffer solutions that will bracket the pH of interest. Buffer Solutions with a pH of 4.00, 7.00, and 10.00 are used to calibrate the pH meter. All buffer solutions must be placed on a magnetic stir plate and gently stirred during the procedure.

*Note: the buffer solutions from different manufacturers may have a pH value of 4.01, 7.01, 10.01, these may also be used in this procedure.*

**12.1.1.2** Turn the meter on, and allow to equilibrate for at least 15 minutes.

**12.1.1.3** Press the "CAL" button on the meter to enter into calibration mode.

**12.1.1.4** Place the electrode in the buffer solution "4.00" and wait for the meter to read the value and record internally. Follow the on-screen instruction of the meter.

**12.1.1.5** Place the electrode in the buffer solution "7.00" and repeat the step above.

**12.1.1.6** Place the electrode in the third buffer solution "10.00" and wait for the meter to finish calibration using all three buffer solutions.

**12.1.1.7** Press "Enter" button to go to the next step to read the slope of the calibration and record the slope value in the logbook.

**12.1.1.8** Press the "Enter" button again to exit out of the calibration mode and enter into measuring mode.



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**12.1.1.9** After calibration is accepted, ensure that the meter is properly calibrated by measuring the pH of a buffer solution such as a buffer of pH 7.00su. This verification buffer solution must be from a different manufacturer or from a different lot number if from same manufacturer.

**12.1.1.10** Record the slope and the VBS value in the pH calibration Logbook. If the slope is not between the ranges of 98-102%, recalibrate the pH meter using fresh buffer solutions after a thorough cleaning of the electrode.

## 13.0 PROCEDURE

### 13.1 Liquids

**13.1.1** Remove the samples from the refrigerator and allow to reach room temperature.

**13.1.2** Gently invert the sample container and mix to homogenize the sample.

**13.1.3** Pour approximately 25 mL of a representative sample into a digestion cup and place a small magnetic stirring bar and place the cup on a magnetic stir plate and gently stir at a slow rate to avoid vortexing of the sample.

**13.1.4** Thoroughly rinse the pH electrode with ultrapure water and gently blot dry with a Kim-wipe to remove excess water.

**13.1.5** Insert the pH electrode into the sample solution in the digestion cup and press the measurement button on the meter.
Allow the meter to stabilize and wait for the 'pH' symbol to stop blinking. The pH meter is equipped with an automatic temperature compensation unit and will display the temperature at which the sample pH is measured.

**13.1.6** Record the pH and the temperature of the sample in the pH analysis logbook.

**13.1.7** Once a stable pH has been reached, remove the electrode from the sample and rinse the pH electrode with ultra-pure water before placing back in the electrode storage solution.

### 13.2 Sludges

**13.2.1** When sludge samples (mixture of solid and liquids) are to be measured for pH, gently mix the sample by inverting the container.

**13.2.2** Immediately obtain a representative portion of the sample and pour into a digestion cup.

**13.2.3** Thoroughly rinse the pH electrode with ultrapure water and gently blot dry with a Kim-wipe to remove excess water.

**13.2.4** Insert the pH electrode into the sample, being careful to position the electrode only in the liquid portion of the sample. Allow the instrument to stabilize and place a small magnetic stirring bar to gently stir at a slow rate so as to avoid vortexing the sample.

**13.2.5** Follow steps 13.1.4 through 13.1.6 above to measure sample pH.

### 13.3 Soils and Wastes

**13.3.1** Remove samples from the refrigerator and allow to reach room temperature. Weigh about 10g of a representative sample into a plastic digestion cup. Samples are analyzed using a 1:1 soil:water ratio. If adequate soil to solution ratio is not obtained, 1:2 or 1:5 soil to water ratio can be used.

**13.3.2** Add 10mL of ultrapure water and cover, if the solution is not enough add an additional 10mL of water to the soil. Cap the digestion cup and shake by hand for 2 minute at 10min intervals until the soil and liquid portions are mixed.



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**13.3.3** A mechanical shaker is suitable for mixing the soil and water to obtain a uniform slurry, if a mechanical shaker is used, shake the container for 15 minutes.

**13.3.4** Allow the sample to stand for 1 hour before proceeding to measure the pH.

*Note: Alternatively, the sample may be filtered or centrifuged to separate the solids from the aqueous phase. If the sample absorbs all the liquid, additional dilution is acceptable up to 1:5 ratio.*

**13.3.5** Thoroughly rinse the pH electrode with ultrapure water and blot dry with a Kim-wipe.

**13.3.6** Insert the pH electrode into the sample and adjust the electrode in the electrode holder, being careful to submerge the electrode into the liquid portion of the sample. Allow the electrode to stabilize. Follow the steps 13.1.3 through 13.1.6

**13.3.7** Once a stable pH has been reached, remove the electrode from the sample and rinse the pH electrode with ultra-pure water before placing back in the electrode storage solution. Record the pH and the temperature in the pH analysis logbook.

## 14.0 DATA ANALYSIS AND CALCULATIONS

**14.1** Report pH values to the nearest 0.1 units.

**14.2** Laboratory Control Sample [Dup] Recovery

$$Spike\ Recovery = \frac{LCSR}{LCSA} \times 100$$

Where:

LCSR = LCS Spike Result
LCSA = Spike Added

**14.3** Relative Percent Difference

$$RPD = \left| \frac{SR - SDR}{\left( \frac{SR + SDR}{2} \right)} \right| \times 100$$

Where:

RPD = Relative Percent Difference
SR = Spike Recovery
SDR = Spike Duplicate Recovery

## 15.0 QUALITY CONTROL

**15.1** Buffer solutions must be entered and assigned a number from the Chemical and Standards Database upon receipt. The containers must be dated when first opened. Buffers must be discarded by the expiration date.

**15.2** The instrument and electrode must be calibrated every day before any samples are processed.

**15.3** Thoroughly rinse the pH electrode between samples.

**15.4** A minimum of one sample duplicate must be analyzed for every batch of not more than 10 samples for liquid and 20 samples for solid (soil, waste and sludge).



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**15.5** On a quarterly basis, the temperature probe is to be calculated against a NIST calibrated thermometer.

## 16.0 ACCEPTANCE CRITERIA

**16.1** Duplicate samples must have a Relative Percent Difference (RPD) of <10%.

**16.2** Laboratory Control Sample/Verification Buffer Standard (LCS/VBS) recoveries must be within the stated pH values below.

**16.2.1**

**16.2.2** The acceptance criteria for 7.0 buffer range is between 6.83 and 7.17 pH units.

## 17.0 CORRECTIVE ACTIONS FOR NON-CONFORMANCE DATA

**17.1** When Quality Control measures fail, and the clients' results are affected, the client will be advised that the results may not be reliable. It may be necessary based on clients' needs to recollect the sample and submit at a later time. If the client is unable to recollect a sample, the data will be released with the appropriate documentation. The laboratory staff will complete a Corrective Action Report to document this occurrence.

**17.2** Should a sample become contaminated or compromised, the preparation shall be terminated and repeated with a fresh sample aliquot. A Corrective Action Report must be completed to document the actions taken.

**17.3** When QC samples do not fall within the acceptable range, the analyst shall review the data for obvious errors such as calculations, preparation errors, or inadvertent spiking errors or other such causes that are not resultant of a systemic failure. The data may be released with a qualifying statement after concurring with the quality manager. A Corrective Action Report must be completed documenting the actions taken when the root cause identified is deemed detrimental to the analysis.

## 18.0 HANDLING NON-CONFORMANCE DATA

**18.1** Non-conformance data are monitored and resolved by identifying categories such as system based, methods based, preparative method based, etc., and are resolved once the problematic areas are identified.

## 19.0 METHOD PERFORMANCE

**19.1** 41 samples were analyzed between May 1, 2021 and July 19, 2021. The mean recovery was 100% with a standard deviation of 0.027 SU.

## 20.0 POLLUTION PREVENTION

**20.1** No solvents are utilized in this method. Use of acids is very limited.

**20.2** Only the amount of chemical that is actually needed is purchased, to eliminate the pollution and cost of disposal.

## 21.0 WASTE MANAGEMENT

**21.1** Toxic waste must never be disposed of down the drain.



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**21.2** Waste generated from sample analysis must be segregated if the process knowledge indicates the presence of any of the hazardous components listed in Table–1, 40 CFR 261.24 and exceed the limits set in the table.

**21.3** When disposing samples the analyst must follow current revision of the "Laboratory Waste Handling and Disposal" SOP (SATL#007G) for detailed disposal procedures.

**21.4** All chemicals and containers must be properly identified and labeled at all times to eliminate ambiguity and cost of disposal of unknowns. If an unknown chemical or container is discovered, label it as 'unknown' and attach a note detailing any information about what the chemical may be, what test it may have been used for, and where it was found. If you find an unlabeled chemical that has crystallized or there is any other indication that it may be unstable, notify management immediately.

**21.5** Generally, empty chemical containers are not considered hazardous waste. Check with management if one such container is found and in doubt. To dispose of the container in the regular trash the container must be completely empty and triple rinsed.

**21.6** The waste drums are picked up upon notification and a copy of the report is submitted to the waste management company.

## 22.0 REFERENCES

**22.1**

**22.2** Standard Methods for the Examination of Water and Wastewater, 23rd Edition.

**22.3** EPA 150.1 – pH (Electrometric), Storet No. 00400 & 00403

**22.4** EPA SW-846, 9040C, November 2004

**22.5** EPA SW-846, 9045D, November 2004

## 23.0 REVISION HISTORY

**23.1** New revision of the method.

**23.2** Annual method revision # 1.0.1, added Solid matrix in this SOP.

**23.3** Annual revision, Rev 2.0.0 – Revised for language, redundancy and formatting.

**23.4** Annual revision 2014. Revised sections: 1.0, 4.0, 12.0, 13.0, and 16.0, 19.0. Removed the Appendix and incorporated the slope information within the procedure (section 12.0).

**23.5** Post assessment revision to provide reference method edition on title page.



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**STANDARD OPERATING PROCEDURE**
**ELECTROMETRIC MEASUREMENT OF PH IN LIQUID AND SOLID MATRICES**

## APPENDIX A
## SOP History and Version Control

| Version | Date of Review/Revision | Review/Revision Approved by | Brief Description |
|---|---|---|---|
| 2.3 | 07/13/2016 | M. Bernard | Revision of cover page, update of method performance data and addition of Appendix A to reflect SOP history and version control. |
| 3.0 | 06/19/2017 | M. Bernard | Biennial review; method performance update and waste disposal protocol. |
| 4.0 | 04/10/2019 | M. Bernard | Biennial review; clarity on acceptance criteria and method performance update. |
| 5.0 | 04/16/2021 | A.Rosecrance | Biennial review; update cover page; change MSDS to SDS. |
| 5.1 | 07/19/2021 | C. Morrow | Revised the following:<br>Section 7.0 – Added stir plate to equipment and supply list.<br>Section 12.1.1.1 – Include the requirement to gently stir calibration buffers.<br>Section 13.1.3 – Include the requirement to gently stir samples. |
| 5.2 | 09/07/2021 | C. Morrow | Revised the following:<br>Section 19 – Update method performance data.<br>Section 22 - Update reference for SM 23rd edition. |



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# STANDARD OPERATING PROCEDURE

*Title*

**Analysis of Total Organic Carbon**
***by* Heated–Persulfate Oxidation**

*Reference Method No.:*

**EPA 415.1 / SM 5310C (23rd Edition, 2017)**

*Matrix/Matrices:*

**Liquid/Drinking Water**

*Document Control Number/Revision Number*

**SOP030A/Revision 2.1**

| | |
|---|---|
| Charles R. Morrow | 09/14/21 |
| ***Approved By: Quality Assurance Manager*** | ***Date*** |
| Richard Hauk | 09/14/21 |
| ***Approved By: General Manager*** | ***Date*** |
| H Sanders | 09/14/21 |
| ***Approved By: Laboratory Director*** | ***Date*** |

Standard Operating Procedures shall be reviewed at least once in two years or as needed to determine their continued suitability, compliance with applicable requirements, and to ensure that they reflect actual procedures being performed.



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**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF TOTAL ORGANIC CARBON BY HEATED–PERSULFATE OXIDATION**

## 1.0 SCOPE AND APPLICATION

**1.1** This SOP describes the procedure for the determination of Total Organic Carbon (TOC) by heated-persulfate oxidation method in drinking water, ground, surface water as well as industrial and domestic aqueous wastes.

## 2.0 REPORTING LIMIT

**2.1** The Reporting Limit (RL) for TOC by heated-persulfate oxidation method is 0.05 mg/L.

## 3.0 SUMMARY

**3.1** Organic carbon is oxidized by persulfate in the presence of heat and the resulting carbon dioxide ($CO_2$) is purged, dried and transferred to and measured by nondispersive infrared detector (NDIR).

**3.1.1** Inorganic carbon present in the sample is removed by acidification (pH <2) and subsequent purging of the sample in the reaction vessel.

**3.1.2** Persulfate is added to the sample in the reaction vessel which is then heated to approximately 95ºC ± 2ºC and organic carbon is oxidized to carbon dioxide ($CO_2$).

**3.1.3** The $CO_2$ generated is transferred to the NDIR detector and is reported as mg/L of total organic carbon using calibration curve.

## 4.0 DEFINITIONS

**4.1** **Reagent Blank/Reagent Water Blank (RB/RWB) –** Reagent blank is the water used to prepare the reagents used in the analysis to determine the organic carbon contribution in the water source.

**4.2** **Total Organic Carbon (TOC) –** Total organic carbon is the derived from all carbon atoms from the organic components in a sample.

**4.3** **Total Inorganic Carbon (TIC)** – Total inorganic carbon is the fraction that is a result of inorganic components i.e., carbonate, bicarbonate, dissolved $CO_2$, etc.

**4.4** **Total Carbon (TC)** – Total carbon is a combination of all fractions of carbon in a sample

**4.5** **Dissolved Organic Carbon (DOC)** – Fraction of the organic carbon is sample that has been filtered through a 0.45µm pore diameter filter.

**4.6** **Purgeable Organic Carbon (POC)** – Fraction of organic carbon that can be measured by removing the carbon using an inert gas.

**4.7** **Non-Purgeable Organic Carbon (NPOC)** – Fraction of organic carbon that can measure by removing the carbon using an inert gas.

**4.8** **Method Blank (MBLK) –**An aliquot of reagent water that is treated exactly as a sample. The blank is exposed to all glassware, equipment, and reagents, etc. The method blank is used to define the level of laboratory background and reagent contamination.

**4.9** **Duplicate (DUP) –** A separate aliquot of the same sample from the same sample container.

**4.10** **Laboratory Fortified Blank/Laboratory Control Sample (LFB/LCS)** – A clean matrix spiked with a solution containing organic carbon at a known concentration. An LCS is used to check extraction and/or method performance.

**4.11** **Laboratory Fortified Blank Duplicate/Laboratory Control Sample Duplicate (LFBD/LCSD)** – LCSD is the same as LCS and is used to check precision of the analytical method.



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**4.12** **Laboratory Fortified Matrix/Matrix Spike (LFM/MS)** – An aliquot of a sample from the analytical batch spiked with a known amount of organic carbon standard. An MS is used to check the effect of matrix on the analyte of interest.

**4.13** **Limit of Detection (LOD)** – An estimate of the minimum amount of a substance that an analytical process can reliably detect (qualitatively). LOD is analyte and matrix specific. For purposes of this test procedure, the LOD is equivalent to the MDL.

**4.14** **Method Detection Limit (MDL) –** The method detection limit (MDL) is defined as the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results. For purposes of this method, the MDL is equivalent to NELAC's Limit of Detection [LOD]. See Section 19.0 METHOD PERFORMANCE for more information regarding LOD.

**4.15** **Practical Quantitation Limit (PQL)/Minimum Reporting Limit (MRL)** – The lowest concentration that can reliably be measured within specified limits of precision and accuracy for a specific laboratory analytical method during routine laboratory operating conditions.

**4.16** **Limit of Quantitation (LOQ)** – For purposes of this method, the LOQ is equal to the low standard used for initial calibration for the analytical method, and is equal to the minimum reporting limit (MRL or PQL).

## 5.0 INTERFERENCES

**5.1** Waters with high alkalinities or those that are laden with high carbonate and bicarbonates may interfere with TOC determination if acidification is incomplete.

**5.2** Highly saline waters and waters with high chloride content (typically >500 mg/L) may impede the oxidation of organic molecules due to preferential oxidation of chloride. Extended reaction time may minimize this interference to generate accurate results.

**5.3** Organic carbon due to volatiles present may be lost during sample preparation and/or acidification process.

**5.4** Large particulates present in the sample may interfere with sample delivery/injection.

**5.5** Large organic molecules such as lignins, tannins, humic acid, etc., oxidize slowly by persulfate and may not oxidize completely.

**5.6** Contamination of samples during handling and preparation is another likely source of interference, especially with reagent water used.

## 6.0 SAFETY

**6.1** Safety glasses and laboratory coats must be worn at all times while in the laboratory. In addition gloves and a face shield or goggles must be worn when dealing with toxic, caustic, and/or flammable chemicals.

**6.2** All chemical compounds should be treated as potential health hazards. The toxicity and/or carcinogenicity of each sample will most likely not be known. Therefore, it is imperative that each sample be handled as a potential health hazard.

**6.3** The analyst should familiarize themselves with all Safety Data Sheets (SDS), safety facilities, and equipment prior to beginning this procedure.

**6.4** Address any and all health and safety concerns to management before beginning this procedure.

## 7.0 EQUIPMENT AND SUPPLIES



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**7.1** Total Organic Carbon Analyzer (TOC) – OI Analytical, Aurora 1030.
**7.2** Nylon filters 0.45 µm syringe filters – BVA scientific or equivalent.
**7.3** Sample bottles – Glass or plastic 250 mL or 125 mL capacity to hold sufficient volume to allow replicate sample analyses – BVA Scientific or equivalent.
**7.4** Glass VOA vials 40 mL – CG Containers or equivalent.
**7.5** Disposable Pasteur pipettes – Fisher Scientific, or equivalent.
**7.6** Digestion tubes – Environmental Express, or Equivalent.
**7.7** 100 mL and 1 L Graduated Cylinder – Fisher Scientific, or Equivalent.
**7.8** Balance, Top Loading, Accurate to 0.0001 g, Denver Instruments, or Equivalent.

## 8.0 REAGENTS AND STANDARDS

**8.1** Reagent Water used in this method is also referred to as TOC reagent water – SATL Ultrapure, or Equivalent.
**8.1.1** Reagent water is generated from SATL's water generation system located in the main laboratory area. This reagent water is of Type II – Medium Reagent Water with Conductivity values ranging from 0.25-0.55 µmho/cm and Resistivity values ranging from 4 MΩ to 2 MΩ on freshly generated water.
**8.1.2** An aliquot of this water, when used for TOC analysis, must be analyzed as "Reagent Water Blank" part of the sequence to evaluate and monitor the organic carbon content of the water. The TOC content of this water should be less than 2× the MDL value (of TOC).
**8.2** Acids
**8.2.1** Hydrochloric Acid [HCl], ACS grade or equivalent.
**8.2.2** Phosphoric acid [$H_3PO_4$], ACS grade or equivalent.
**8.2.3** Sulfuric acid [$H_2SO_4$], ACS grade or equivalent.
**8.3** Sodium persulfate (Sodium peroxydisulfate) 10% – dissolve 100 g reagent in 1 L or TOC reagent water.
**8.3.1** Alternative to Sodium persulfate: Ammonium peroxydisulfate (Ammonium persulfate) 15% – Dissolve 150 g in 1L of TOC water
**8.3.2** Alternative to sodium persulfate: Potassium peroxydisulfate (Potassium persulfate) 2% – Dissolve 20 g in 1 L of TOC reagent water.
**8.4** Potassium biphthalate – (>99% pure) reagent – Acros, Sigma-Aldrich, Fisher scientific or equivalent.
**8.5** Sodium carbonate (for inorganic carbon measurement if needed) – Fisher scientific or equivalent.
**8.6** Sodium bicarbonate (for inorganic carbon measurement if needed) – Fisher scientific or equivalent.
**8.7** Purge Gas – Nitrogen (>99%)
**8.8** Carrier Gas – Oxygen (>99%)

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

**9.1** Collect a representative grab or composite sample in a clean 125 mL, plastic or 40 mL glass VOA vials.
**9.2** All samples must be preserved to pH <2 using HCl or $H_2SO_4$ or $H_3PO_4$.
**9.3** Preservation should begin preferably at the time of collection in bottles containing one of the above acid preservatives.



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**9.4** Samples may be preserved at the laboratory if field preservation is not possible or considered hazardous due to transport using the acids indicated above.

## 10.0 STORAGE

**10.1** Store samples until the time of analysis in a refrigerator at >0°C and ≤ 6°C to preserve sample integrity.

**10.2** Unpreserved samples must be analyzed as soon as practically possible to produce accurate & representative data.

**10.3** Preserved samples have a maximum holding time of 28 days from the time of collection until analysis unless otherwise stated to meet specific project objectives. Preserved samples should be analyzed preferably within 7 days of collection to minimize changes in TOC concentration.

## 11.0 SAMPLE IDENTIFICATION

**11.1** Samples are received from Sample Receiving with an In-House Chain of Custody form generated from the Laboratory Information Management System (LIMS). This includes client identification, sample number, and test to be performed.

**11.2** Each sample is assigned a unique number and a container number if more than one container is received.

## 12.0 CALIBRATION AND STANDARDIZATION

**12.1** Balance must be QC checked using S Class weights on each day of use.

**12.2** The TOC analyzer must be calibrated prior to sample analysis either on the day of analysis or calibration verified using a mid-point calibration standard on the day of analysis prior to sample analysis.

**12.3** Calibration Standards may be purchased where commercially available or prepared in the laboratory using Potassium biphthalate ($C_8H_5KO_4$) as described below. The same lot of the salt may be used to prepare stock standards as long as they are independently prepared from each other. Use one set to calibrate the instrument and use second to verify the instrument calibration.

**12.3.1** Total Organic Carbon [CS – Calibration stock solution] (TOC) 1000 mg/L: Dissolve 2.1254 g anhydrous potassium biphthalate [$C_8H_5KO_4$, CAS No. 877-24-7] in reagent water and dilute to 1000 mL in a volumetric flask. Acidify the stock solution with HCl or $H_3PO_4$ or $H_2SO_4$ to pH ≤2 and store in a refrigerator.

**12.3.2** Total Organic Carbon [SS – Second source stock solution] (TOC) 1000 mg/L: Dissolve 2.1254 g anhydrous potassium biphthalate [$C_8H_5KO_4$, CAS No. 877-24-7] in reagent water and dilute to 1000 mL in a volumetric flask. Acidify the stock solution with HCl or $H_3PO_4$ or $H_2SO_4$ to pH ≤2 and store in a refrigerator.

*Note: The same reagent [$C_8H_5KO_4$] can be used as second source stock as long as it is prepared independently of the calibration stock solution.*

**12.3.3** Alternatively, commercially available stock solution (1000 mg/L) may be purchased from approved vendors.

*Note: If stock solution is purchased ensure that the lot numbers of the stock are different to satisfy the second source requirement.*



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**12.3.3.1** Working Standard Solution–I [WSS-I] (100 mg/L): Transfer 10mL of the stock solution (12.3.1 or 12.3.2) to a 100 mL volumetric flask and dilute to volume with organic carbon free reagent water. Acidify the solution with HCl or $H_3PO_4$ or $H_2SO_4$ to pH ≤2 and store in a refrigerator.

**12.3.3.2** Working Standard Solution–II [WSS-II] (10 mg/L): Transfer 10 mL of the stock solution (12.3.2.1) to a 100 mL volumetric flask and dilute to volume with organic carbon free reagent water. Acidify the solution with HCl or $H_3PO_4$ or $H_2SO_4$ to pH ≤2 and store in a refrigerator.

**12.3.4** Total Inorganic Carbon (TIC) 1000 mg/L: Dissolve 4.4122 g of anhydrous sodium carbonate in 400 mL of TOC reagent water and add 3.497 g of anhydrous sodium bicarbonate and dilute to 1000 mL of TOC reagent water. Transfer to an air tight bottle and store in a refrigerator to prevent degradation.

**12.3.4.1** Inorganic Carbon Working Standard Solution–I (100 mg/L): Transfer 10 mL of the stock solution (12.3.3) to a 100 mL volumetric flask and dilute to volume with organic carbon free reagent water.

**12.3.4.2** Inorganic Carbon Working Standard Solution–II (10 mg/L): Transfer 10 mL of the stock solution (12.3.3.1) to a 100 mL volumetric flask and dilute to volume with organic carbon free reagent water.

***Note***: *Do not add any acid to the TIC standards prepared above.*

***Note:*** *Store all stock and working standards in a refrigerator at ≥0°C ≤6°C. It is recommended that diluted working standards be prepared monthly.*

**12.4** Refer to Table–A, Appendix B of this SOP for instructions on the preparation of the calibration curve.

**12.5** Prior to sample analysis on the TOC analyzer, a set of calibration standards is analyzed following the guidelines in Table–A, Appendix B.

**12.5.1** Refer to section 13.1 for manufacturer's recommended TOC analyzer operating conditions.

*Note: Please ensure that the TOC analyzer is properly connected to the PC system prior to beginning analysis. Refer to Appendix D, for more information regarding the network connectivity.*

**12.6** Calculate the Relative Standard Error (%RSE) of the calibration curve for analytes with linear or quadratic fits. Determine the %RSE using the equation below.

$$\%\,RSE = 100 \times \sqrt{\sum_{i=1}^{n}\left[\frac{x'_i - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where,

$x_i$ = True value for the calibration standard

$x'_i$ = Measured concentration of the calibration standard



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n = Number of calibration points
p = Number of terms in the fitting equation
(Average = 1, Linear = 2, Quadratic = 3)

**12.7** Coefficient of determination must be >0.920. If this cannot be achieved, the calibration is unacceptable and recalibration is necessary after remedial action to correct the problem.

**12.8** Calculate the Relative Error (%RE) for those analytes are calibrated using linear or quadratic curve fits and determine the coefficient of determination using the following equation.

$$\%Relative\ Error = \frac{x'_i - x_i}{x_i} \times 100$$

Where,
$x_i$ = True value for the calibration standard
$x'_i$ = Measured concentration of the calibration standard

**12.9** The relative error percent must be calculated for two of the calibration levels, i.e., the low calibration standard and the mid-point calibration standard. The acceptance criteria for low standard is 50% and the mid-point standard is 10%.

**12.10 Initial Calibration**

**12.10.1** Prior to sample analysis, the TOC system is calibrated using multiple calibration points. The standards may be prepared as described in the appendix of this SOP or are purchased from approved vendors.

**12.10.2** Prepare a set of calibration points as shown in Table A, Appendix B and analyze as per routine instrument conditions.

**12.10.3** Inject the calibration standards (and samples) in triplicate and verify that the precision is within ±10% (%RSD) between the triplicate injections.

**12.10.4** A calibration curve with a correlation coefficient of ≥0.995 is considered valid and sample analysis may begin after calibration is verified using a mid-point standard.

**12.11 Calibration Verification – Initial and Continuing [ICV& CCV]**

**12.11.1** The initial calibration is verified at the beginning of each working day or a batch of 10 samples using a calibration verification standard.

**12.11.2** Initial calibration of the TOC system is verified by analyzing a single point calibration standard (ICV) at 5mg/L once before sample analysis can begin.

**12.11.2.1** Prepare 50 mL of ICV standard fresh on the day of calibration. This standard (ICV) must be prepared from a source stock that is other than the one used for initial calibration.



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**12.11.2.2** Dilute 2.5 mL of the working stock standard 100 mg/L (WSS–I section 12.3.2.1 ) to 50 mL in TOC reagent water to obtain 5 mg/L standard (refer to Table A, Appendix B on how to prepare this standard).

**12.11.2.3** The ICV standard must be within ±15% of the true concentration in order for the calibration to be valid.

**12.11.2.4** If the ICV exceeds ±15% the expected value, then evaluate for possible spiking errors, calculation errors, injection malfunction, change in instrument conditions, etc. If no obvious problems are identified, the stock solution may be suspect.

**12.11.2.5** Prepare fresh stock solution, re-analyze the single ICV standard and verify calibration. If the ICV meets the acceptance criteria, sample analysis may begin.

**12.11.2.6** Failure of the ICV to meet ±15% of the expected value mandates instrument recalibration using freshly prepared calibration stock solution(s).

**12.11.3** A single point continuing calibration verification standard [CCV] at 5 mg/L must be analyzed on each day prior to sample analysis to verify that instrument calibration is still valid.

**12.11.4** Reference method requires that a laboratory control sample (LCS) be prepared from a source other than the calibration stock solution and analyzed after every 10 injections. Therefore, it is recommended that the CCV standard be prepared from a second source stock to meet this requirement. In this procedure ICV/CCV and LCS are used interchangeably and they are prepared at the same level.

***Note:*** *Avoid redundancy and prepare the CCV and LCS from the second source stock solution to meet the method requirement.*

**12.11.4.1** The CCV standard must be within ±15% of the true concentration before sample analysis can begin.

**12.11.4.2** If the CCV exceeds ±15% of the expected value, then evaluate for possible spiking errors, calculation errors, injection malfunction, etc. If no obvious problems are identified, the stock solution may be suspect.

**12.11.4.3** Prepare fresh stock solution, re-analyze the single CCV standard and verify calibration.

**12.11.4.4** Failure of the CCV to meet ±15% of the expected value second time mandates instrument recalibration. Prepare calibration stock solution(s) and calibration verification standard(s) and repeat the procedure as described above.

**12.11.4.4.1** Perform any required instrument maintenance before recalibrating the instrument.

**12.11.4.5** Further corrective actions such as cleaning the TOC system, replacing reagent water, preparing new reagents, etc. may be performed.

**12.11.4.6** After major maintenance is done on the system, two consecutive CCV standards may be analyzed to re-evaluate the calibration and both must meet the acceptance criteria. If both consecutive CCV standards meet the acceptance criteria then samples may begin on the system without recalibrating the instrument as in section 12.7.

**12.11.4.7** If any one of the two CCV standards fail to meet the acceptance criteria then a new calibration curve must be analyzed and evaluated prior to any sample analysis.

**12.12 Recommended system maintenance**



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**12.12.1** In cases where the initial calibration does not meet the acceptable correlation coefficient criteria of ≥0.995 or if the ICV/CCV is not within ±15% of the expected value, system maintenance is required. A short list of the remedial actions is given below:

a. Clean the system by running the "CleanUp" routine built into the instrument.
b. Check the sample and system tubing.
c. Check needle for any clogging clean/replace if necessary.
d. Check all reagent containers for any biological growth and clean as necessary.
e. Refer to the operational manual for other maintenance and suggested troubleshooting techniques.
f. If none of these maintenance tasks resolve the problems, replace the column, or contact the manufacturer for either technical help or service call.

## 13.0 PROCEDURE

### 13.1 Analytical System

**13.1.1** Analytical system is comprised of a Carbon analyzer equipped with an autosampler and a nondispersive infrared detector (NDIR).

**13.1.2** The main external components of the system consist of a digestion/oxidation vessel that is heated by an electrode, sample pump for reagent delivery to the vessel, injection syringe, halide scrubber and drying column.

**13.1.3** Additional internal components consist of various valves, such as electronic flow control (EFC), electronic pressure control (EPC) and gas drying membrane filter and an integrated NDIR.

**13.1.4** In addition to the heated persulfate method components, the TOC system is also capable of analyzing TOC by high temperature combustion, as such it is equipped with a combustion chamber.

**13.1.5** Software for data acquisition and data processing: refer to and follow manufacturer's instructions on the operation of the TOC system and software.

**13.1.6** The following are typical settings recommended by the manufacturer set into TOC software for instrument control. These values may be optimized to achieve better sensitivity toward the instrument. (Refer to Appendix C for screen shots of the instrument software).

| Parameter | Value |
|---|---|
| Acid Volume | 0.50 mL |
| Persulfate Volume | 1.00 mL |
| Reagent water rinse Volume | 15 mL |
| System Pressure | 20 PSI |
| Drain Time | 15 Sec |
| Reaction Time (TIC) | 1:30 min |
| Reaction Time (TOC/TC) | 3:00 min |
| Reaction Temp (TIC) | 70ºC |
| Reaction Time (TOC/TC) | 95ºC ±5ºC |
| Sample Volume | 7mL |
| Sparge Time | 2:00 min |

### 13.2 Sample Preparation and Equipment



1610 S. Laredo Street, San Antonio, Texas 78207-7029 • (210) 229-9920 • Fax (210) 229-9921

**13.2.1** Samples are collected in preserved containers as per sections 9.0 and 10.0.

**13.2.2** Allow samples to equilibrate to room temperature before starting the analysis. Do not allow samples to sit at room temperature in open container.

**13.2.3** Samples must NOT be filtered through a 0.45 µm filter prior to taking a sample aliquot, if not analyzing for DOC.

**13.2.4** If samples contain large amounts of particulate matter, use smaller aliquot and perform diluted analysis to prevent syringe clogging.

**13.2.5** If samples are collected in 40 mL VOA vials, direct analysis can be performed by loading the samples into the autosampler trays.

**13.2.6** If samples are collected in containers other than VOA vials, then draw a well homogenized aliquot by mixing the contents by gently inverting the container several times.

**13.2.7** Program the sequence (refer to Appendix C) on the instrument control panel and initiate the run. Typical sample volume optimized for this method for individual calibration ranges must be used when analyzing samples and standards.

***Note**: Detailed optimal conditions for each calibration range can be found in Appendix C of this procedure.*

**13.2.8** Field samples may be analyzed following calibration standards and after the calibration curve has been established and verified to meet acceptance criteria.

**13.2.9** Prepare a laboratory reagent blank [LRB], laboratory control sample [LCS], matrix spike [MS], sample duplicate [Dup], etc., along with field samples in a batch.

**13.2.10** Using peak areas sample concentration is calculated via calibration curve response.

**13.2.11** Read and report measured TOC values directly from the instrument and calculate according to section 14.0 below.

**13.2.12** When sample concentration exceeds the calibration range, sample must be diluted appropriately so that the concentration will fall within the calibration range.

### 13.3 Data review and Data processing

**13.3.1** All raw data must be reviewed for integration errors by the software to ensure that peaks are correctly integrated.

**13.3.2** Use peak area responses to compute the concentration of the field and QC samples.

**13.3.3** Report values that fall within the lowest and highest calibration points. Sample concentrations that fall beyond the highest calibration point must be diluted and re-analyzed.

**13.3.4** Refer to section 14.0 for the calculation of sample concentrations and LCS, MS, etc., recoveries.

## 14.0 DATA ANALYSIS AND CALCULATIONS

**14.1** Report total organic carbon concentrations in field samples directly from the instrument generated data in mg/L taking into account any dilution factor.

**14.1.1** Calculate the analyte concentration:

Liquid/Water Samples:

$$\text{TOC (mg/L)} = \text{Instrument Reading} \times \text{DF}$$

**14.2** Laboratory Fortified Blank/Laboratory Control Sample [LCS] Recovery



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$$\text{Spike Recovery} = \frac{LCSR}{LCSA} \times 100$$

Where:

LCSR = LCS Spike Result
LCSA = LCS Spike Added

**14.3** Laboratory Fortified Matrix/Matrix Spike [MS] Recovery

$$\text{Spike Recovery} = \frac{MSR - SR}{MSA} \times 100$$

Where:

MSR = MS Result
SR = Sample Result [Un-spiked field sample]

MSA = Matrix Spike Added

**14.4** Relative Percent Difference – Duplicate samples

$$RPD = \left| \frac{SR - SDR}{\left( \frac{SR + SDR}{2} \right)} \right| \times 100$$

Where:

RPD = Relative Percent Difference
SR = Sample Result
SDR = Sample Duplicate Result

## 15.0 QUALITY CONTROL

**15.1** Laboratory's initial demonstration of capability is documented by the analysis of 4 quality control sample spiked with 4 times the LOQ of TOC established.

**15.2** The Limit of Quantitation (LOQ) for this procedure is 0.05 mg/L.

**15.3** Each batch must include a Blank and CCV/LCS (source other than calibration stock) after every 20$^{th}$ sample or less in the sequence.

**15.4** A routine batch of sample analysis must include a reagent water blank, method blank, laboratory control sample (and duplicate) and a sample duplicate. Optionally a matrix spike sample may be analyzed in cases where sample matrix effects need to be evaluated/monitored or project objectives mandate such requirement.

**15.5** Chemicals and standards must be entered upon receipt into the LIMS and assigned a number. The containers must be dated when first opened and discarded by the expiration date. Any chemical or standard that fails to meet Quality Control requirements should be returned to the manufacturer for replacement.

**15.6** Working standards including those prepared/used daily must be entered and assigned a number in LIMS when prepared. All working standards must be discarded by the expiration date.

**15.7** Any working standard that fails to meet Quality Control requirements must be discarded and re-prepared. If the working standard continues to fail, contact the manufacturer of the chemicals, and if necessary, order new supplies.



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**15.8** All Certificates of Analysis must be retained.

**15.9** All analytical records must be backed up monthly as a minimum.

## 16.0 ACCEPTANCE CRITERIA

**16.1** Individual QC/Field sample injections analyzed in triplicate must have ≤10% RSD.

**16.2** Reagent Water Blank's TOC concentration must be less than 2× MDL value.

**16.3** Calculate the LCS recovery as in section14.0. The acceptable range for the LCS is ±15% of true concentration.

**16.4** Calculate the MS recovery if a matrix spike sample is analyzed. The acceptable range for the LFM is 80%-120% until such time there is sufficient data becomes available.

**16.5** Determine the RPD for the sample and sample duplicate. The acceptable range for the RPD between sample and sample duplicate is ≤10%.

**16.6** Refer to Appendix B, Table–B for QC acceptance criteria.

## 17.0 CORRECTIVE ACTIONS FOR NON-CONFORMANCE DATA

**17.1** Should a sample become contaminated or compromised, the preparation and analysis shall be terminated and repeated with a fresh sample aliquot. A Corrective Action must be completed to document the actions taken.

**17.2** When Quality Control measures fail, and the clients' results are affected, the client will be advised that the results may not be reliable. It may be necessary based on clients' needs to recollect the sample and submit at a later time. If the client is unable to recollect a sample, the data will be released with the appropriate documentation. The laboratory staff will complete a Corrective Action form to document this occurrence.

**17.3** When QC samples do not fall within the acceptable range, the analyst shall review the data for obvious errors such as calculations, preparation errors, or inadvertent spiking errors or other such causes that are not resultant of a systemic failure. The data may be released with a qualifying statement after concurring with the quality manager. A Corrective Action must be completed documenting the actions taken when the root cause identified is deemed detrimental to the analysis.

## 18.0 HANDLING NON-CONFORMANCE DATA

**18.1** Non-conformance data are monitored and resolved by identifying categories such as system based, methods based, preparative method based, etc., and are resolved once the problematic areas are identified.

## 19.0 METHOD PERFORMANCE

**19.1** Fourty-seven reagent water samples spiked with 5 mg/L of TOC standard analyzed between January 1, 2020 and December 31, 2020 had an average recovery of 96.8% with a standard deviation of 2.51.

**19.2** A method detection limit study is performed, initially and verified quarterly thereafter for all analytes that are listed for this method.



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**19.2.1** All sample processing steps of the analytical method shall be included in the determination of the MDL.

**19.2.2** The Minimum Reporting Limit (MRL) of quantitation is equivalent to NELAC's Limit of Quantitation (LOQ). The concentration of the LOQ is equal to the low standard used for initial calibration.

**19.3** During the beginning of each quarter, two replicate samples of organic free reagent water are spiked with a known amount of target analytes at the concentration used in the initial determination of the MDL and analyzed on the TOC analyzer.

**19.4** If the analyte is repeatedly not detected in the quarterly spiked sample analyses, or do not meet the qualitative identification criteria of the method, then this is an indication that the spiking level is not high enough and should be adjusted.

**19.5** Prepare and analyze seven spike replicates and seven method blanks on at least three different days carried out through sample preparation steps. Existing routine method blanks can be used for this study.

**19.6** The validity of the MDL shall be confirmed by qualitative identification of the analyte.

**19.7** A minimum of seven MDL replicate samples and seven method blanks are used to calculate the MDL values. For purposes of this method, the MDL is equivalent to TNI's Limit of Detection (LOD).

Calculate the $MDL_S$ (MDL spiked samples) value using the following formula:

$$MDL_s = t_{[n-1,\ 1-\infty = 0.99]}\ S_s$$

Where,

$t_{[n-1,\ 1-\infty = 0.99]}$ = Student's t value for the 99% confidence level with n-1 degrees of freedom,
n = number of replicates.
$S_s$ = the standard deviation of the replicate analyses.

Calculate the $MDL_B$ (MDL blank samples) values using the following formula:

$$MDL = t_{[n-1,\ 1-alpha = 0.99]}\ S_b$$

Where,

$t_{[n-1,\ 1-alpha = 0.99]}$ = Student's t value for the 99% confidence level with n-1 degrees of freedom,
n = number of replicates.
$S_b$ = the standard deviation of the replicate method blank sample analyses.

| Number of Replicates | Degrees (degrees of freedom) | $t_{(n-1,\ 0.99)}$ |
|---|---|---|
| 7 | 6 | 3.143 |
| 8 | 7 | 2.998 |
| 9 | 8 | 2.896 |
| 10 | 9 | 2.821 |
| 11 | 10 | 2.764 |



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**19.8** Current MDL values for method analytes in this SOP can be found in the SATLMDL.xls spreadsheet.

## 20.0 POLLUTION PREVENTION

**20.1** Each method is evaluated prior to use in order to minimize waste volume and toxicity.
**20.2** A non-hazardous or less toxic substitute may be used whenever possible.
**20.3** Purchase only the amount of chemical that is actually needed or that will be used to eliminate the cost of disposal later.

## 21.0 WASTE MANAGEMENT

**21.1** Toxic waste must never be disposed of down the drain.
**21.2** Waste generated from sample analysis must be segregated if the process knowledge indicates the presence of any of the hazardous components listed in Table–1, 40 CFR 261.24 and exceed the limits set in the table.
**21.3** When disposing samples, the analyst must follow current revision of the "Laboratory Waste Handling and Disposal" SOP (SATL#007G) for detailed disposal procedures.
**21.4** All chemicals and containers must be properly identified and labeled at all times to eliminate ambiguity and cost of disposal of unknowns. If an unknown chemical or container is discovered, label it as 'unknown' and attach a note detailing any information about what the chemical may be, what test it may have been used for, and where it was found. If you find an unlabeled chemical that has crystallized or there is any other indication that it may be unstable, notify management immediately.
**21.5** Generally, empty chemical containers are not considered hazardous waste. Check with management if one such container is found and in doubt. To dispose of the container in the regular trash the container must be completely empty and tripled rinsed.
**21.6** The waste drums are picked up upon notification and a copy of the report is submitted to the waste management company.

## 22.0 REFERENCES

**22.1** Total Organic Carbon – Heated–Persulfate Oxidation Method, SM5310C, Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2011.
**22.2** Total Organic Carbon – Heated–Persulfate Oxidation Method, SM5310C, Standard Methods for the Examination of Water and Wastewater, 23rd Edition, 2017.
**22.3** Organic Carbon, Total (Combustion or Oxidation) – EPA 415.1, 1974.

## 23.0 REVISION HISTORY

**23.1** New SOP of the method.



1610 S. Laredo Street, San Antonio, Texas 78207-7029 • (210) 229-9920 • Fax (210) 229-9921

**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF TOTAL ORGANIC CARBON BY HEATED–PERSULFATE OXIDATION**

## APPENDIX A
## SOP History and Version Control

| Version | Date of Review/Revision | Review/Revision Approved by | Brief Description of changes |
|---|---|---|---|
| 1.0 | 02/11/2019 | S. Abburu | New SOP |
| 1.1 | 06/12/2019 | S. Abburu | Updated–<br>Section 12.0; Section 13.0; Section 16.0<br>Appendix B; levels for calibration curve.<br>Appendix D – added. |
| 1.2 | 08/08/2019 | M. Bernard | Corrected Section 15.1, LOQ |
| 2.0 | 02/26/2021 | A.Rosecrance | Biennial review; update title page; change MSDS to SDS. |
| 2.1 | 09/13/2021 | C. Morrow | Revised he following.<br>Update Title Page and headers<br>Section 4 – Update definition.<br>Section 12 – Added details to calibration process to include %RE.<br>Section 15 – Update quality assurance requirements.<br>Section 19 – Update method performance data and update MDL procedure.<br>Section 22 – Update reference method information. |



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**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF TOTAL ORGANIC CARBON BY HEATED–PERSULFATE OXIDATION**

## APPENDIX B

### Table– A

Preparation of Calibration curve(s)

| Cal Pt. | Stock Conc | Std. Conc. (mg/L) | Final Std. Vol | Vol Req. (mL) | Vol Req. (µL) |
|---|---|---|---|---|---|
| 1 | 10 | 0.05 | 50 | 0.250 | 250 |
| 2 | 10 | 0.10 | 50 | 0.500 | 500 |
| 3 | 100 | 0.50 | 50 | 0.250 | 250 |
| 4 | 100 | 1.0 | 50 | 0.500 | 500 |
| 5 | 1000 | 5.0 | 50 | 0.250 | 250 |
| 6 | 1000 | 10.0 | 50 | 0.500 | 500 |
| 7 | 1000 | 20.0 | 50 | 1.000 | 1000 |
| 8 | 1000 | 30.0 | 50 | 1.500 | 1500 |

* Concentration (point) to be used as daily calibration check standard (CCV).



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**STANDARD OPERATING PROCEDURE**
**ANALYSIS OF TOTAL ORGANIC CARBON BY HEATED–PERSULFATE OXIDATION**

## APPENDIX B *cont'd*

## Table– B

Quality Control Acceptance Criteria

| QC Element | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| | | | |
| Initial calibration for all analytes | Initial calibration prior to analysis | A correlation coefficient of ≥0.995. | Correct problem then repeat initial calibration |
| Second source calibration verification | Once after initial calibration | Analyte concentration must be within ±15% of the expected value. | If concentration is >15%, correct problem and reanalyze. Second failure repeat initial calibration. |
| Continuing calibration verification (Second source stock solution). | Daily, before sample analysis and every 10th injection. | Analyte concentration within 15%. | Samples after the last verification standard and before the failed verification standard must be rejected/re-analyzed.<br><br>Data may be reported if the check standard fails high and sample concentration is non-detect.<br><br>Data may be reported if the check standard fails low and sample concentration is detected above the reporting limit.<br><br>Any data reported as such must be qualified indicating that the check concentration is outside the limits on the analytical reports. |
| Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of QC check samples at 1-4 times the reporting limit (LOQ). | Once initially, before analysis can begin by the analyst. | Recoveries within acceptable limits, and RPD <10%. | Recalculate results.<br><br>Locate and fix problem with system and the rerun demonstration for those analytes that did not meet criteria. |
| Reagent Blank | Daily, before sample analysis and every 10th injection. | TOC concentration detected <2x MDL | Correct problem then re-prepare and reanalyze, method blank and all samples processed using this reagent water. |
| Method Blank | Once per analytical batch of 10 or fewer. | No analytes detected < RL. | Correct problem then re-prepare and reanalyze method blank and associated samples in the batch. |
| LCS/LCSD | One LCS/LCSD pair per 10 samples per matrix. | Based on control chart limits established over a period of time and updated in ELEMENT LIMS. | Correct problem then re-prepare and reanalyze the LCS and all samples in the affected analytical batch. Qualify data on the analytical report. |
| MS | One MS per 10 samples per matrix. | Based on control chart limits established over a period of time and updated in ELEMENT LIMS. | Qualify data on the analytical report for the sample batch. |
| Sample Duplicates | One per 10 samples per matrix. | Based on control chart limits established over a period of time and updated in ELEMENT LIMS. | Qualify data on the analytical report for the sample batch. |



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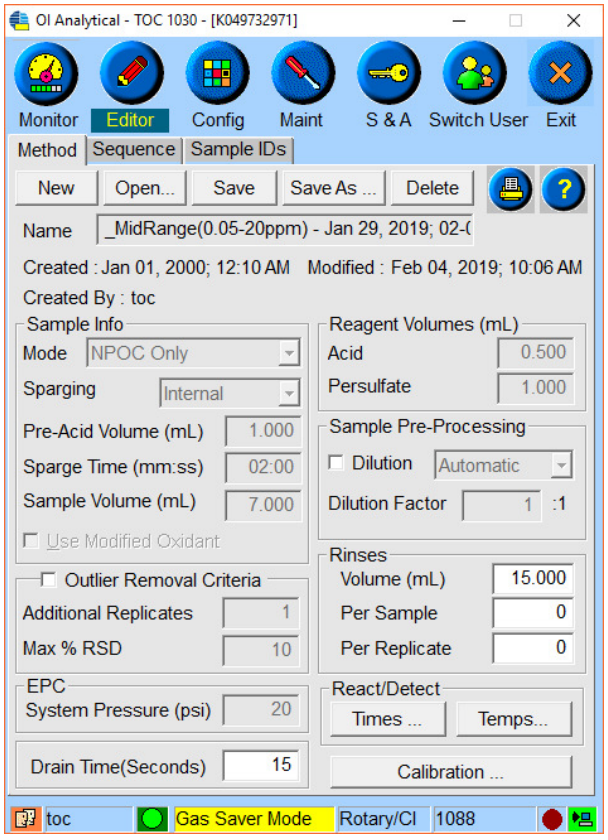
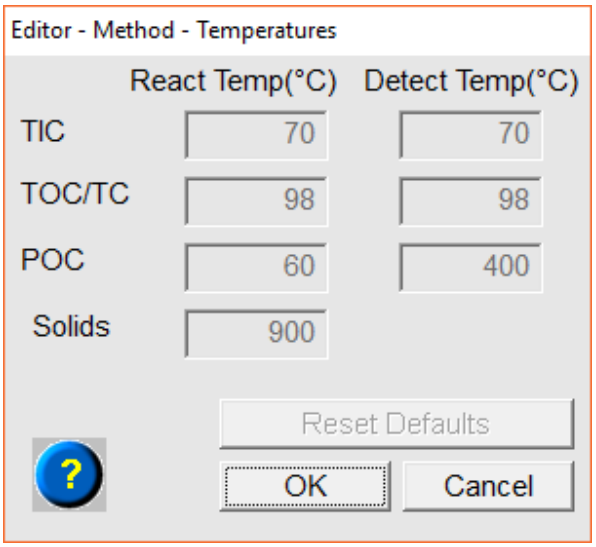
# APPENDIX C

Example method settings – the method can be edited as necessary to adjust the volumes, reaction times, reaction temperatures, sparing time, etc., when a new method need to be optimized.



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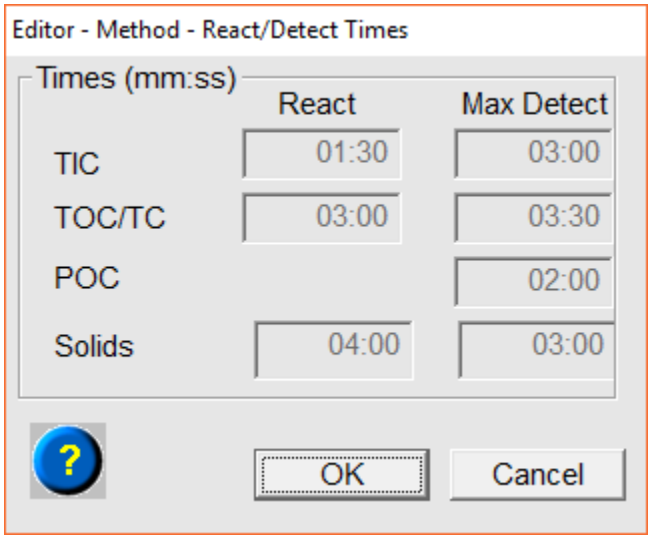


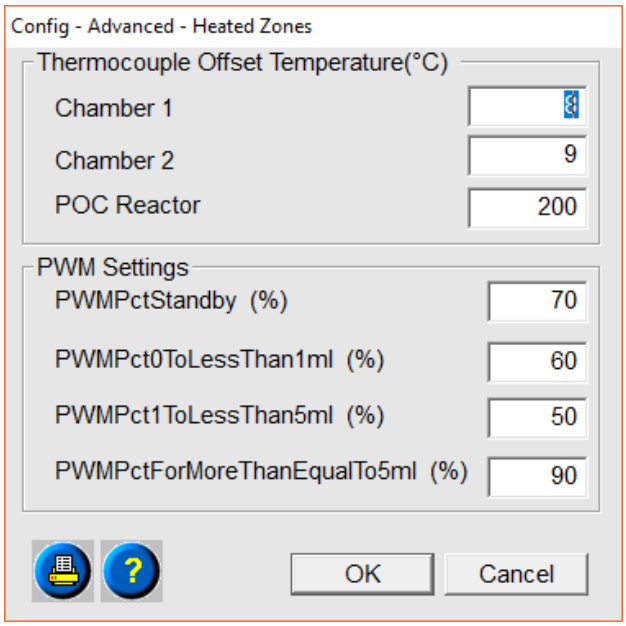


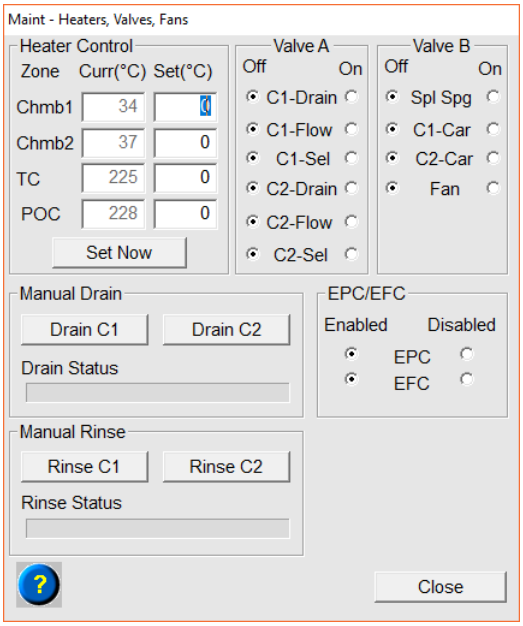


**Example Calibration screen with calibration parameter**



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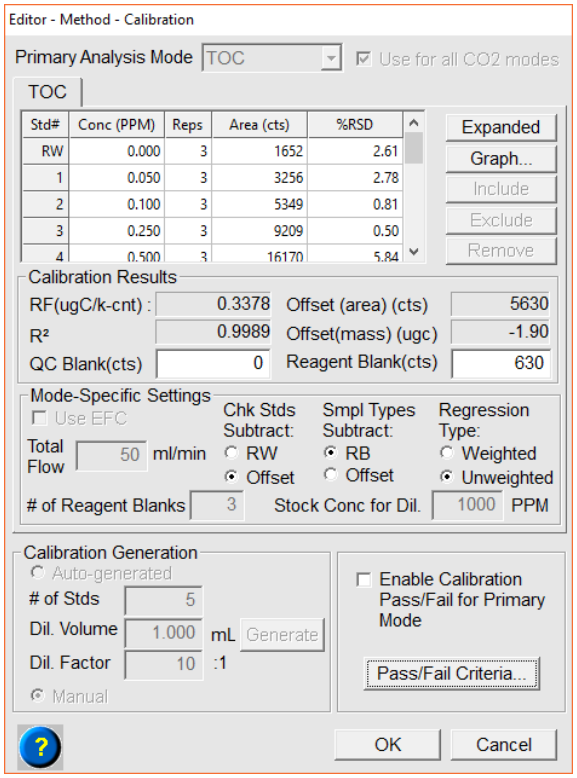
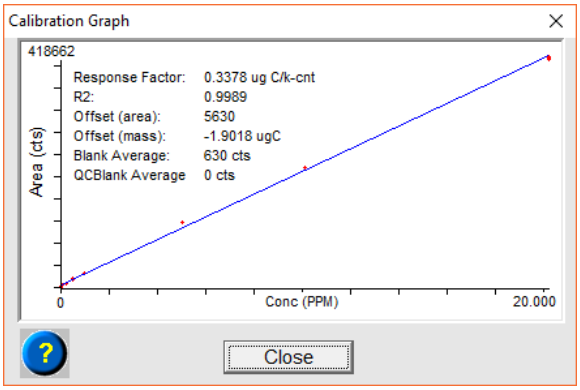
**STANDARD OPERATING PROCEDURE**

**ANALYSIS OF TOTAL ORGANIC CARBON BY HEATED–PERSULFATE OXIDATION**




**Example Calibration sequence**

**Example daily routine sequence**



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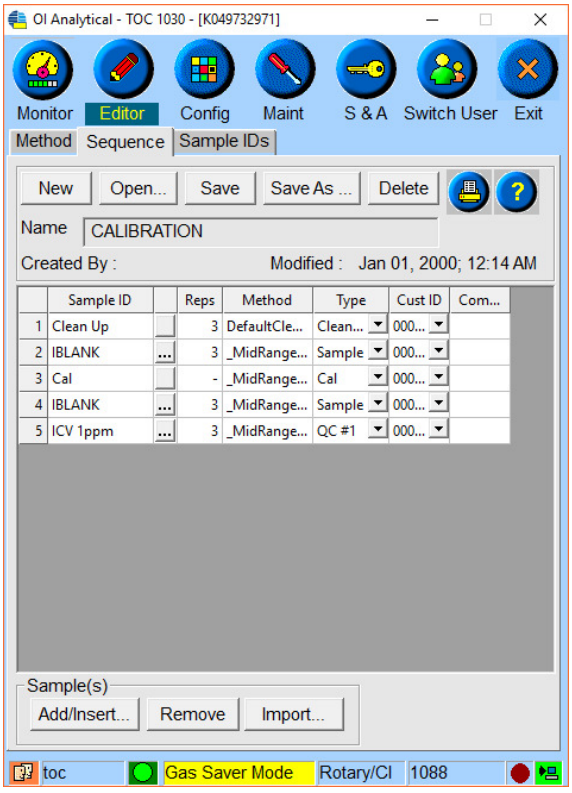
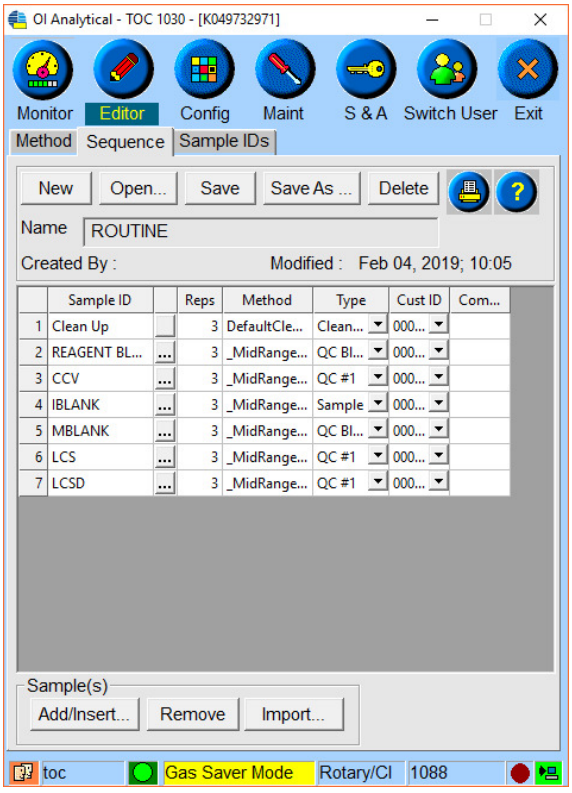
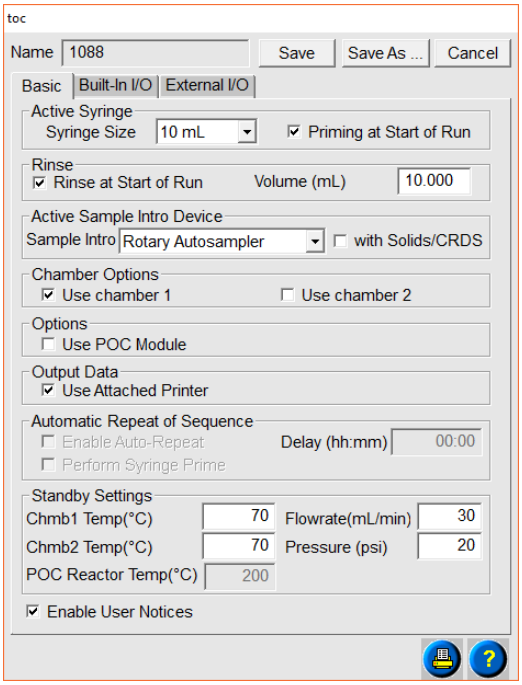
**STANDARD OPERATING PROCEDURE**

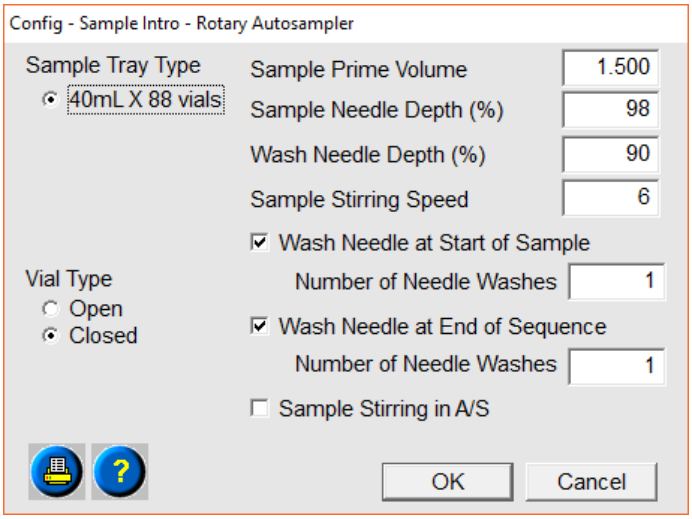
**ANALYSIS OF TOTAL ORGANIC CARBON BY HEATED–PERSULFATE OXIDATION**




## Auto Sampler settings



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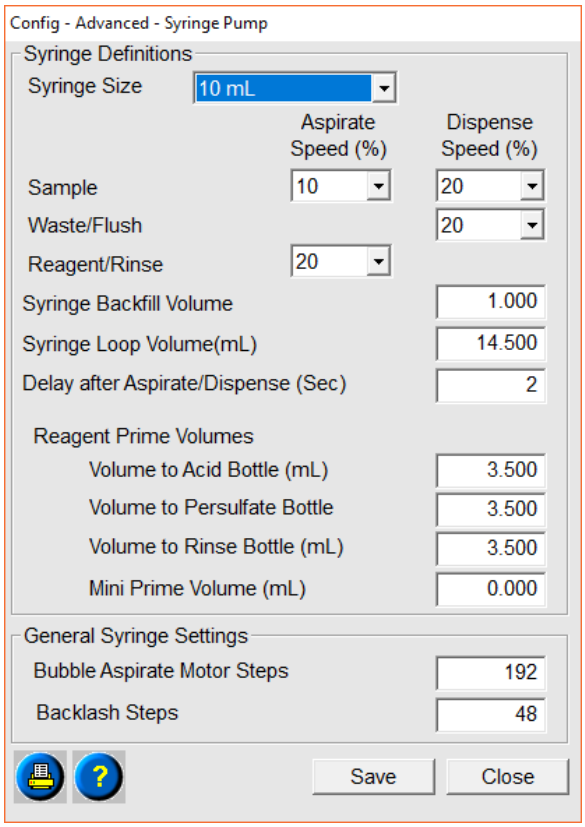
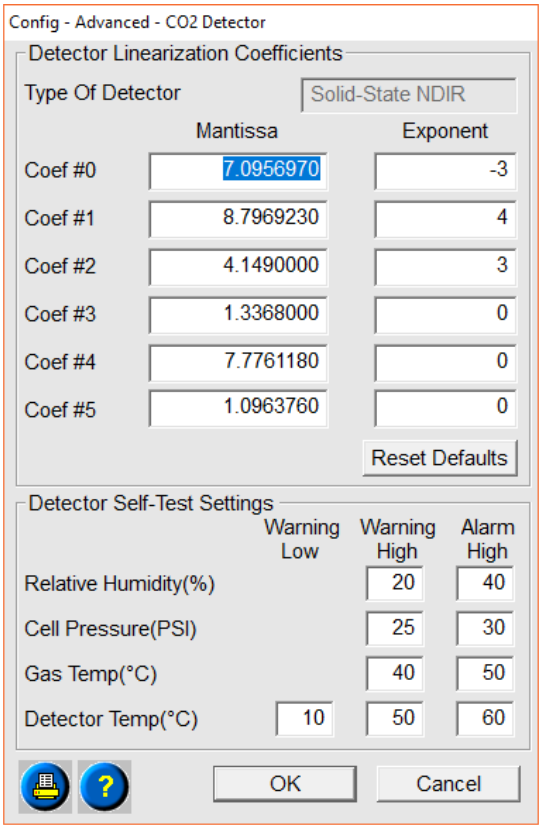
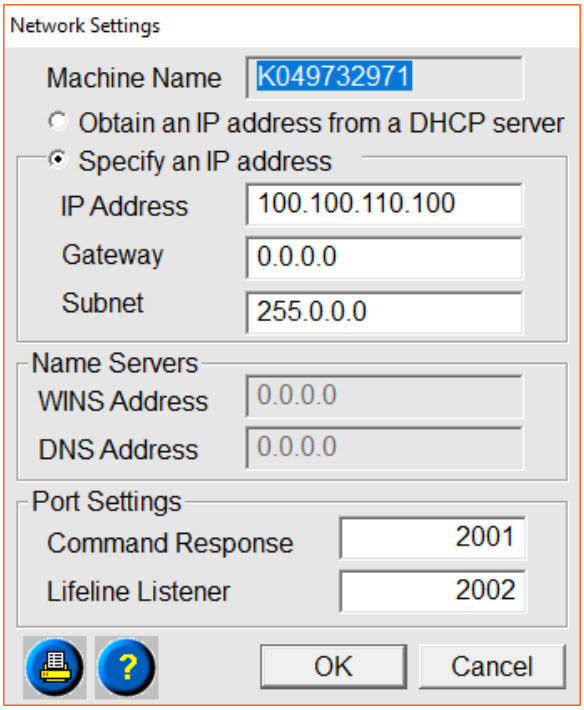
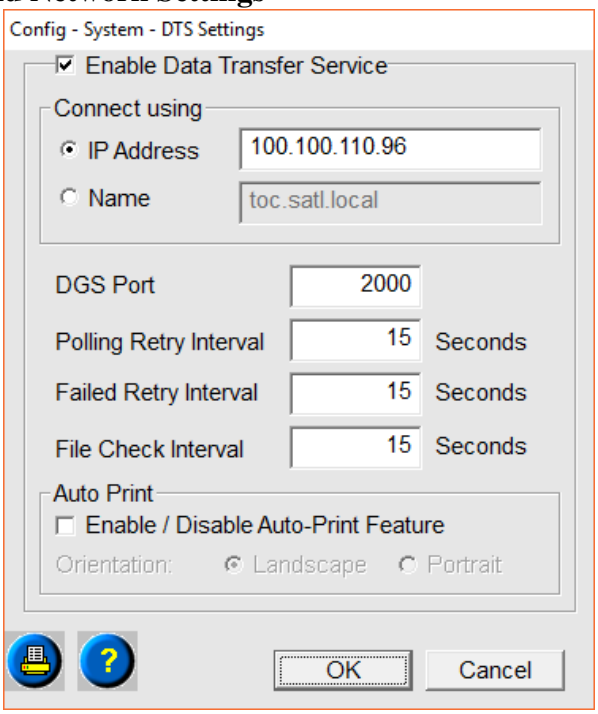



## Data Transfer Service and Network Settings



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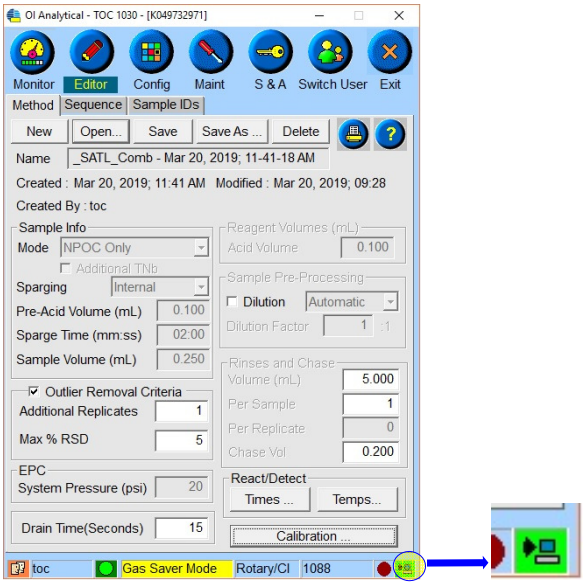
**STANDARD OPERATING PROCEDURE**
**ANALYSIS OF TOTAL ORGANIC CARBON BY HEATED–PERSULFATE OXIDATION**

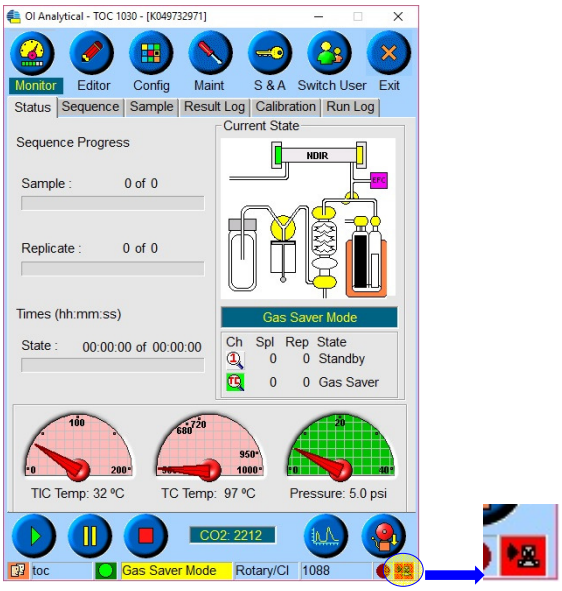
# APPENDIX D

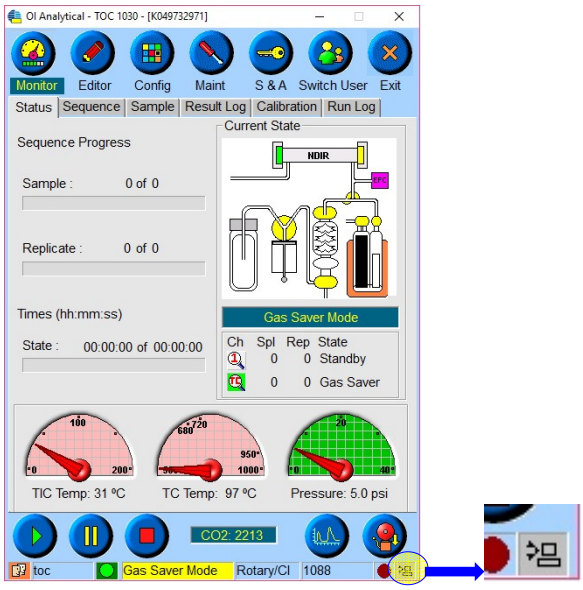
**PC – TOC Instrument Connectivity troubleshooting –** The TOC Data Gathering Service (DGS) must be enabled and running on the instrument and the PC for the data to be available on the PC. The picture below indicates a green icon with an arrow pointing toward the PC. If this is not seen then the system is not connected to the PC and therefore no data will be available to review or print on the PC. The data will only be available on the instrument hard drive and cannot be downloaded. This icon below indicates that the instrument is connected and ready to send data to the PC system.




***Proper Connection***

The following two scenarios indicate that there is nonconnectivity




*DGS enabled but not connected.* *DGS disabled, no connection exists, no data gathered*



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If this is the case, to connect to the instrument do the following to connect the instrument to the PC prior to beginning the sequence.

- Click on Windows Start button
- Type “Services” in the windows search bar
- This will open the – Services Window
- Scroll down the items and locate – “TOCDatagatheringService” and ensure this service is running. If it is stopped, click the “start” button to initiate the service.
- Exit the “Services” by closing the window.
- Return to the TOC instrument control screen
- The PC icon should turn green and ready to acquire and download data.
- Sometimes it may be necessary to restart the computer and/or the instrument or both to establish the connection.



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