### 3.3 Sample Collection, Storage and Preservation

Figure 3 details required containers, sample volumes, preservation techniques, and holding times for proper sample collection. A discussion is included pertaining to sample collection, preservation and storage before analysis.

**Sample Collection**

RMBEL supplies clients with various field services and sample collections. These field services include, but are not limited to, Secchi disk readings, lake monitoring sampling, ground water monitoring, surface water monitoring and private drinking water collections. All personnel are trained with sampling protocol and approved standard operating procedures to ensure correct sample handling. If a situation arises where the sampling deviates from said protocols or SOPs, the deviation is recorded in detail on the sampling form and chain-of-custody form. The client is notified of the deviation if applicable.

**Appendix D** lists the Field Services Standard Operating Procedures. **Appendix E** lists the Biological Monitoring Standard Operating Procedures.

**Holding Times**

The integrity of the sample is dependent on strict adherence to standard operating procedures in place in the laboratory. RMBEL maintains and recognizes the importance of sample holding times. Sample holding times are determined by the analytical method. Holding times are noted in Figure 3. Samples received after the holding time has expired are analyzed only under direct request from the client. Appropriate sample and/or data qualifiers are noted on the final report, as applicable (**Appendix J).**

**Preservation Techniques**

Proper preservation practices must be followed and can be found for each analyte in Figure 3. Following collection and during transportation, samples should be kept at 6°C or on ice. Samples requiring preservation should be preserved as soon as possible after collection to maintain the integrity of the sample. Complete collection, storage, and preservation guidelines can be found in 40CFR Part 136.

NOTE: Complete and certain preservation of samples, regardless of source, is a practical impossibility. Regardless of the sample nature, complete stability for every constituent can never be fully attained. At best, sample preservation only slows the biological and chemical changes that inevitably continue after the sample is collected.

Methods of preservation are intended to retard biological action, retard hydrolysis of chemical compounds and complexes, and reduce volatility of constituents. Preservation methods are limited to pH control, chemical addition, amber or opaque bottles, filtration, refrigeration, and freezing.

To minimize the potential for volatilization or biodegradation between sampling and analysis, keep the sample as cool as possible without freezing. Preferably, pack samples in crushed or cubed ice, or a commercial ice substitute before shipment. Avoid using dry ice because it may freeze the samples and cause the containers to break. Dry ice may also affect the pH of the samples. Keep composite samples cool with ice or a refrigeration system set at 6°C during collection. Analyze the samples as quickly as possible upon arrival at the laboratory. If immediate analysis is not possible, storage at 2°-4° C is recommended for most samples.

Use chemical preservation only when it is shown not to interfere with the method of analysis. When chemicals are used, add them to the sample bottle so that all sample portions are evenly preserved as soon as collected. No single preservation method is entirely satisfactory; choose the preservation with regard to the analyses being made. Because a preservation method for one analysis may interfere with the preservation for another, samples for multiple determinations may need to be split and preserved separately. All methods of preservation may be inadequate when applied to suspended matter. DO NOT use formaldehyde as a preservative because it affects numerous analyses.

The previous discussion is by no means exhaustive and/or comprehensive. Clearly, it is impossible to prescribe absolute rules for preventing all possible changes. Additional advice can be found in the analytical methods and/or SOPs under

individual determination, but to a large degree the dependability of an analytical determination rests on the experience and good judgment of the person collecting the sample.

### 3.4 Sample Storage and Disposal

**Sample Storage Prior to Analysis**
All samples are properly stored from the time they arrive at the laboratory to disposal. Samples are refrigerated at 4°C prior to analysis unless method SOPs indicate other storage conditions.

Nature of Sample Changes: Some analyses are more likely than others to be affected by storage before analysis. Certain cations are subject to loss by absorption or ion exchange with the walls of glass containers. These include aluminum, cadmium, chromium, copper, iron, lead, manganese, silver, and zinc, which are best collected in a separate clean bottle and acidified with nitric acid to a pH below 2.0. This minimizes precipitation and absorption to container walls.

Temperature, pH, and dissolved oxygen are best determined in the field. Temperature changes quickly and pH may change significantly in a matter of minutes. Dissolved gases (oxygen, carbon dioxide) may be lost very quickly. With the changes in the pH-alkalinity-carbon dioxide balance, calcium carbonate may precipitate and cause a decrease in the values for calcium and total hardness.

Iron and manganese are readily soluble in their lower oxidation states but are relatively insoluble in their higher oxidation states, therefore, these cations may precipitate or they may dissolve from sediment depending on the redox potential of the sample. Microbial action may be responsible for the changes in the nitrate-nitrite-ammonia content, or decreases in phenol concentration. It may also be responsible for reducing sulfate to sulfide in Biological Oxygen Demand (BOD). Residual chlorine is reduced to chloride. Sulfide, sulfite, ferrous iron, iodide, and cyanide may be lost through oxidation. Color, Odor, and Turbidity may increase, decrease, or change quality. Sodium, silica, and boron may be leached from a glass container. Hexavalent chromium may be reduced to chromic acid.

Biological changes taking place in a sample may change the oxidation state of some constituents. Soluble constituents may be converted to organic bound material in cell structures, or cell lysis may result in release of cellular material into solution. The well-known nitrogen and phosphorus cycles are examples of biological influence on sample composition.

Zero headspace is important in preservation of samples with volatile organics. Avoid loss of volatile materials by filling sample containers completely; achieve this by filling the bottle until there is a positive meniscus before capping and sealing, being careful not to rinse out the acid preservation or come into contact with the acidified sample. Serum vials with septum caps are particularly useful in that a sample portion for analysis can be taken through the cap with a syringe.

In general, the shorter the time that elapses between the collection of a sample and its analysis, the more reliable the analytical results. For certain constituents and physical values, immediate analysis in the field is required. For samples composited in the field, use the time at the end of the composition collection as the sample collection time.

It is impossible to state exactly how much time may be allowed between sample collection and analysis. Changes occurring in the sample depend on the character of the sample, the analysis to be made, and the conditions of storage. Changes caused by the growth of microorganisms are greatly retarded by keeping the sample in the dark and at a low temperature. When the interval between sample collection and analysis is long enough to produce changes in either the concentration or the physical state of the constituent to be measured, follow the preservation practices stated in Figure 3.

**Sample Storage**

Following sample receipt and proper preparations by the laboratory, all samples are stored separate from standards or reagents. A temperature of 4° C is maintained and monitored 24 hours a day by digital controls. AM and PM temperature readings are maintained in a daily temperature log. Samples are kept in the cooler until time of analysis. Samples that require analysis at room temperature (25° C) are brought out of refrigeration and allowed to warm to the desired temperature before beginning analysis. When all analyses are performed on a sample, it is transferred to a cooler for cold storage until the time of disposal.

**Sample Disposal**

All samples are disposed in an environmentally sound manner or returned to the client upon request. Samples may also be returned to the client if they contain hazardous wastes. Samples that contain solvents are evaporated in a fume hood unless the vapors create an environmental concern. Samples that contain hazardous materials, such as mercury and silver, are sent to a hazardous waste collection facility that has a Very Small Quantity Generator (VSQG) program. The remaining samples are stored in a holding tank that is transferred to a Wastewater Treatment Facility for proper disposal. The pH of the waste is verified and adjusted to be above pH 2.0 before being added to the holding tank. Refer to RMBEL SOP 03-005 for Waste Disposal procedures. Samples are typically stored for 30 days following the report date unless otherwise instructed by the client.

**Figure 3: Sampling Requirements**

| Parameter | Matrix | Container | Minimum Sample Size | Preservative | Holding Time |
|---|---|---|---|---|---|
| **Microbiology** | | | | | |
| Coliform, Total or Fecal | Water & Wastewater | Sterile Container | 100 mL | Refrigerate 6 C | 8 Hrs EPA compliance 24 Hours |
| Coliform, Total or Fecal, Chlorinated Water | Water & Wastewater | Sterile Container w/ thiosulfate | 100 mL | Refrigerate 6 C | 8 Hrs EPA compliance 24 Hours |
| E. coli | Water & Wastewater | Sterile Container | 100 mL | Refrigerate 6 C | 8 Hrs EPA compliance 24 Hours |
| Total Coliform or E. coli | Drinking Water | Sterile Container | 100 mL | Refrigerate 6 C | 8 Hrs EPA compliance 30 hours |
| **Inorganics and Nutrients** | | | | | |
| Acidity, Free | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 14 Days |
| Alkalinity, Total | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 14 Days |
| BOD | Water & Wastewater | Plastic or Glass | 650 mL | Cool 6 C | 48 Hours |
| BOD, Carbonaceous | Water & Wastewater | Plastic or Glass | 650 mL | Cool 6 C | 48 Hours |
| Chloride | Water & Wastewater | Plastic or Glass | 100 mL | None | 28 Days |
| | Solid | Plastic | 50 mL | None | 28 Days |
| Chlorine, Residual | Water & Wastewater | Plastic or Glass | 500 mL | None | Analyze Immediately |
| Chlorophyll-a | Water | Amber Glass | 1000 mL | Cool 6 C, Dark | 48 Hours |
| Chromium, Hexavalent | Water & Wastewater | Plastic or Glass, rinsed w/ 1:1 HNO3 | 200 mL | Cool 4 C | 24 Hours |
| COD | Water & Wastewater | Plastic or Glass | 100 ml | H2SO4 to pH < 2, Cool 6C | 28 Days |
| | Solid | Plastic | 50 mL | None | 28 Days |
| Color | Water & Wastewater | Plastic or Glass | 250 mL | Cool 6 C | 48 Hours |
| Conductance, Specific | Water & Wastewater | Plastic or Glass | 500 ml | Cool 6 C | 28 Days |
| | Solid | Plastic | 50 mL | None | 28 Days |
| Cyanide, Total | Water & Wastewater | Plastic or Glass | 500 mL | NaOH to pH > 12, Cool 6C | 14 Days |
| | Solid | Plastic | 50 mL | None | 14 Days |
| Fluoride | Water & Wastewater | Plastic | 100 mL | None, Cool 6C | 28 Days |
| | Solid | Plastic | 50 mL | None | 28 Days |
| Hardness, Total | Water & Wastewater | Plastic or Glass | 500 mL | HNO3 to pH < 2 | 6 Months |
| Iodide | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 24 Hours |
| Nitrogen, Kjeldahl Total | Water & Wastewater | Plastic or Glass | 500 mL | H2SO4 to pH < 2, Cool 6C | 28 Days |
| | Solid | Plastic | 50 mL | Cool 6 C | 28 Days |
| Nitrogen, Nitrate + Nitrite | Water & Wastewater | Plastic or Glass | 250 mL | H2SO4 to pH < 2, Cool 6C | 28 Days |
| | Solid | Plastic | 50 mL | Cool 6 C | 28 Days |
| Nitrogen, Nitrate | Water & Wastewater | Plastic or Glass | 250 mL | Cool 6 C | 48 Hours |
| | Solid | Plastic | 50 mL | Cool 6 C | 48 Hours |

**Figure 3: continued**

| Parameter | Matrix | Container | Minimum Sample Size | Preservative | Holding Time |
|---|---|---|---|---|---|
| Nitrogen, Nitrite | Water & Wastewater | Plastic or Glass | 250 mL | Cool 6 C | 48 Hours |
| | Solid | Plastic | 50 mL | Cool 6 C | 48 Hours |
| Nitrogen, Total | Water and Wastewater | Plastic or Glass | 250 mL | H2SO4 to pH < 2, Cool 6C | |
| Nitrogen, Ammonia | Water & Wastewater | Plastic or Glass | 500 mL | H2SO4 to pH < 2, Cool 6C | 28 Days |
| | Solid | Plastic | 50 mL | Cool 6 C | 28 Days |
| Oil & Grease | Water & Wastewater | Amber Glass | 1000 mL | H2SO4 to pH < 2<br>Cool 6 C | 28 Days |
| Organic Carbon, Total | Water & Wastewater | Plastic or Glass | 500 mL | H2SO4 to pH < 2, Cool 6C | 28 Days |
| | Solid | Plastic | 50 mL | Cool 6 C | 28 Days |
| Oxygen, Dissolved | Water & Wastewater | Glass bottle w/ top, no headspace | 500 mL | None | Analyze Immediately |
| pH | Water & Wastewater | Plastic or Glass | 100 mL | None | Analyze in 15min |
| | Solid | Plastic | 50 mL | None | Analyze in 15min |
| Phaeophytin | Water | Amber Glass | 1000 mL | Cool 6 C | 48 Hours |
| Phenolics, Total | Water & Wastewater | Amber, Plastic, or Glass | 1000 mL | H2SO4 to pH <2, Cool 6C | 28 Days |
| Phosphorus, Total | Water and Wastewater | Plastic or Glass | 500 mL | H2SO4 to pH <2, Cool 6C | 28 Days |
| | Solid | Plastic | 50 mL | Cool 6 C | 28 Days |
| Phosphorus, Ortho | Water & Wastewater | Plastic or Glass | 500 mL | Filter, Cool 6 C | 48 Hours, Filter within 15 minutes |
| | Solid | Plastic | 50 mL | None | 48 Hours |
| Salinity | Water & Wastewater | Glass | 500 mL | None | Analyze Immediately |
| Settleable Matter | Water & Wastewater | Plastic or Glass | 1000 mL | Cool 6 C | 48 Hours |
| Silica | Water & Wastewater | Plastic | 500 mL | Cool 6 C | 28 Days |
| Solids, Total | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 7 Days |
| | Solid | Plastic | 50 mL | None | 7 Days |
| Solids, Total Volatile | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 7 Days |
| | Solid | Plastic | 50 mL | None | 7 Days |
| Solids, Total Suspended | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 7 Days |
| Solids, Total Settleable | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 7 Days |
| Solids, Total Dissolved | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 7 Days |
| Solids, Suspended Volatile | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 7 Days |
| Sulfate | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 28 Days |
| | Solid | Plastic | 50 mL | None | 28 Days |
| Sulfide | Water & Wastewater | Amber Glass | 500 mL | Cool 6 C<br>2 mL Zinc Acetate | 7 Days |
| | Solid | Plastic | 50 mL | None | 7 Days |
| Sulfite | Water & Wastewater | Plastic or Glass | 500 mL | None | Analyze Immediately |
| Surfactants | Water & Wastewater | Plastic or Glass | 500 mL | Cool 6 C | 48 Hours |
| Temperature | Water & Wastewater | Plastic or Glass | 100 mL | None | Analyze Immediately |
| Turbidity | Water & Wastewater | Plastic or Glass | 250 mL | Cool 6 C | 48 Hours |
| **Metals** | | | | | |
| Metals, General | Water & Wastewater | Plastic or Glass, Rinsed w/ 1:1 HNO3 | 500 mL | HNO3 to pH < 2 | 6 Months |
| | Solid | Plastic | 50 mL | Cool 6 C | 6 Months |
| Mercury | Water & Wastewater | Plastic or Glass, Rinsed w/ 1:1 HNO3 | 500 mL | 1:1 HNO3 to pH < 2 | 28 Days |
| | Solid | Plastic | 50 mL | Cool 6 C | 28 Days |
| Lead | Water & Wastewater | Plastic or Glass | 1000 mL | HNO3 to pH <2 | 6 Months |
| | Drinking Water | Plastic or Glass | 1000 mL | HNO3 to pH <2 | 6 Months |

**Figure 3: continued**

| Parameter | Matrix | Container | Minimum Sample Size | Preservative | Holding Time |
|---|---|---|---|---|---|
| **Organic Compounds** | | | | | |
| GRO (Gasoline Range Organics), PVOC | Water | (4) 40 mL amber, glass bottle w/ 0.5mL 1:1 HCL, no headspace | 40 mL | 4 drops conc. HCL, Cool 6 C | 14 Days |
| | Soil | (2) 60mL amber, glass bottle w/ 25mL MeOH | 20- 35 grams | 25 mL Methanol, Cool 6 C | 21 Days |
| DRO (Diesel Range Organics) | Water | (2) 1 L Amber, glass bottle w/5.0 mL 1:1 HCL | 1 L | 5 mL 1:1 HCL, Cool 6 C | 7 Days |
| | Soil | (2) 60 mL amber, glass bottle w/tare weight, no preservative | 20- 35 grams | Cool 6 C | 10 Days |
| BTEX | Water | (3) 40 mL glass vial w/ septa cap, zero headspace | 40 mL | 4 drops conc. HCL, Cool 6 C | 14 Days |
| | Soil | (2) 4 oz Amber Glass w/ septa cap, zero headspace | 25 grams | Cool 6 C | 14 Days |
| VOCs: | Water & Wastewater | (4) 40 mL glass vial w/ septa cap, zero headspace | 40 mL | 0.5 mL 1:1 HCL | 14 Days |
| | Soil | (2) 60 mL amber | 20-35 g | 25 mL MeOH | 14 days |
| Pesticides, General | Water | (2) Amber Glass w/ TFE septa cap | 1 Liter | Cool 6 C | 7 Days |
| | Soil | (1) 4 oz Amber Glass | 50 mL | Cool 6 C | 14 Days |
| TCLP, Toxicity Characteristic Leaching | Water | 1 L Amber Glass | 1 Liter | Cool 6 C | 14 Days |
| | Soil | (2) 4 oz Amber Glass | 200 grams | Cool 6 C | 14days |

| Solids & Semi-Solid - Microbiology | Matrix | Container | Minimum Sample Size | Preservative | Holding Time |
|---|---|---|---|---|---|
| Coliform, Fecal | Semi-Solid/Solid | Sterile Container | 100 mL | Cool 6 C | 8 Hrs EPA compliance 24 Hours |

| **Solids & Semi-Solid - Inorganics and Nutrients** | | | | | |
|---|---|---|---|---|---|
| % Solids | Semi-Solid/Solid | Plastic or Glass | 100 grams | Cool 6 C | 7 days |