ATTACHMENT 1
WASTE ANALYSIS PLAN

APPENDIX 1
QUALITY ASSURANCE PLAN

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3.0 QAP DESCRIPTION

Data of unknown quality is useless. It is this premise which Clean Harbors Aragonite management bases its stance on quality control.

Data of good quality does not just happen. Quality control must be an integral part of Aragonite's day to day operations. It relies on each individual within the program to make data quality his/her primary objective.

It is the goal of Clean Harbors Aragonite to produce high quality data. This Quality Assurance Plan is designed to ensure that all data generated are complete, precise, and accurate. Data quality will be documented.

There are three primary areas where data quality is of concern. These are as follows:

- Waste Approval and Acceptance
- Process Operating Parameters
- Residue Characterization

The objective of the first area is to characterize a particular waste stream and determine if the facility is capable of accepting the material under its permit conditions.

The objective of the second area is to provide analytical support so that Aragonite can operate within its permit conditions. This mainly addresses the blending of waste material so that maximum conditions stipulated in the operating permit such as Btu/hr, total chlorine, etc., are not exceeded. It also addresses other areas which may not be specified in the permit but enable Clean Harbors Aragonite to operate the facility in a more efficient manner. A waste's compatibility with other wastes already being stored at the facility is also assessed.

The last area concerns the by-products which are generated from the thermal treatment of the wastes. The slag from the kiln and the dusts from the spray dryer and baghouse are analyzed to ensure that the incineration process is destroying the organic hazardous constituents in compliance with the permit and Land Disposal Restrictions.

3.1 PURPOSE

The purpose of this Quality Assurance Plan is to ensure that all information, data, and resulting decisions compiled under a specific task are technically sound, statistically valid, and properly documented. Quality Assurance is the program or structure within an organization which plans, designs, and monitors the QA procedures and affirms the data quality in reports.

Quality Control is the mechanism or activities through which Quality Assurance achieves its goals. This is accomplished through a program which defines the frequency and methods of checks, audits, and reviews necessary to identify problems and dictate corrective action.

3.2 SCOPE

The Quality Assurance Plan encompasses the entire measurement system from initial sampling to the final reporting and interpretation of results. This QAP is for the Aragonite laboratory.

3.3 OBJECTIVE

This Quality Assurance Plan is designed to produce accurate and reliable data. In order to accomplish this objective, the following criteria must be achieved:

- All procedures and practices must be accepted by the client and/or regulatory agency.

- A continuing program must be developed to monitor the performance of the program.

- A mechanism must be developed for correcting problems which are determined by the monitoring assessment.

4.0 LABORATORY ORGANIZATION AND RESPONSIBILITY

The organizational structure of the Clean Harbors Aragonite laboratory is shown on the organization chart maintained at the facility.

The initial step in any Quality Assurance Plan begins with the people involved. In addition to the organizational chart, descriptions of those individuals involved in Quality Assurance and their responsibilities are included.

4.1 QUALITY ASSURANCE COMPLIANCE OFFICER

The QA Compliance Officer is responsible for identifying quality problems, to recommend and provide solutions, and to verify the implementation of the solutions. The duties include:

- developing mechanisms to carry out QA/QC objectives;
- administration of quality control procedures;
- implementation of corrective action(s); and
- maintenance of QA/QC records.

4.2 LABORATORY MANAGER

The Laboratory Manager is responsible for the daily operation and management of the Aragonite laboratory. The manager's duties include:

- management of laboratory personnel;
- oversee and coordinate instrument and equipment maintenance;
- review of work procedures and daily laboratory practices;
- work scheduling;
- record keeping;
- training of laboratory personnel; and
- responsibility for the administration of Quality Control at his/her respective laboratory.

4.3 CHEMISTS

The Chemist's duties as they relate to QA/QC are as follows:

- recommendations for technical decisions;
- evaluating and reviewing test procedures;
- reviewing and signing laboratory reports;
- ensuring that results are accurate and reproducible;
- calculations and interpretations of test results;
- equipment and instrument calibration and operation; and
- sample preparation and analysis.

4.4 LABORATORY TECHNICIANS

The laboratory technicians duties as they relate to QA/QC are as follows:

- performing sample preparation and analysis;
- maintaining a clean and safe working environment;
- making recommendations to supervisors regarding analysis or QA/QC performances;
- performing QA/QC analysis; and
- reviewing and signing laboratory reports.

4.5 SAMPLING TECHNICIANS

Sampling technicians are specially trained personnel responsible for sampling containers, vessels, tanks, and process streams. Sampling techs typically are in Production, Receiving, Incineration, or any combination of these areas. These people may be chemists, engineers, laboratory technicians, or operations personnel. They all have specialized training in sampling QA/QC techniques including the use of various sampling apparatus, sample site selection, sampling methodologies, and chain of custody procedures.

The QA/QC Coordinator or the Laboratory Manager interacts with the sampling technicians to assure understanding of selection, collection, storage, transportation, and documentation

practices.

5.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT IN TERMS OF PRECISION, ACCURACY, AND COMPLETENESS.

Data Quality objectives are defined as follows:

- Precision - is the measure of agreement of a set of replicate results among themselves. Precision is assessed by means of duplicate/replicate sample analysis.

- Accuracy - is the nearness of a result or the mean (X) of a set of results to the true value or an established laboratory mean. Accuracy is assessed by means of reference samples and percent recoveries.

- Completeness - is the measure of the amount of valid data derived from a system of measurement as compared to the amount of data which was expected to be obtained.

5.1 ACCURACY

Accuracy information for quantitative measurements is generated by using one or more of the following techniques:

Calibration Checks
Calibration checks determine the acceptability of a calibration. The limits are method specified.

Calibration Check Standards are used as continuing checks for organic analysis. The equation for the Calibration Check Standard is:

% Recovery = 100(result/true value)

Calibration Verification Standards (CVS) are second-source standards (a different brand from those used for generating a calibration curve) to check the accuracy of the calibration curve. The equation for the CVS is:

% Recovery = 100(result/true value)

Method Accuracy Checks

Method Accuracy checks determine the acceptability of a batch of samples that have been subjected to a preparation step (i.e., digestion, extraction, combustion, etc.). The limits are method specified or statistically generated, whichever is the more stringent at the time of analysis. The means and limits are tracked by generating statistical data. If the Method Accuracy check does not fall within the more stringent control limit, the batch is rejected and

rerun for the failed constituent(s).

Control Limit = method specified
or
mean $\pm$3sd, whichever is the more stringent

Laboratory Control Samples (LCS) are purchased Standard Reference Materials that may closely match the matrix that is being analyzed.

Control Blank Spikes (CBS) are blanks that are spiked with the constituents being analyzed.

Matrix Spikes (MS) are samples that are spiked with the constituents being analyzed. They are only used as method accuracy checks when the matrix has demonstrated a lack of interference in the analysis.

% Matrix Spike Recovery = 100(Sample Spike Result-Sample Amount)/Spike Amount

5.2 PRECISION

Precision information for quantitative measurements is generated by duplicating the Method Accuracy Checks. The results of the duplication are compared to the initial method accuracy check. The limits are method specified or statistically generated, whichever is the more stringent at the time of analysis. The means and limits are tracked by generating statistical data. If the precision does not fall within the more stringent control limit, the batch is rejected and rerun for the failed constituent(s).

Control Limit = Method specified
or
Upper Control Limit, which ever is the more stringent

Laboratory Control Sample Duplicate (LCSD), or *Control Blank Spike Duplicates* (CBSD) are analyzed by the same procedure as the initial method accuracy check.

Matrix Spike Duplicates (MSD) are samples that are spiked with the constituents being analyzed. They are only used as precision checks when the matrix has demonstrated a lack of interference in the analysis.

Method Specified Limits for precision are compared to results generated by either:

Relative Percent Difference (RPD) = 100(Range of Results/Average of Results)
or
Coefficient of Variation (CV) = 100(standard deviation/mean)

Upper Control Range Limits are generated by historical statistical techniques.
Upper Control Range Limit = Mean of Ranges x (D_2/d_2)

where: Range = absolute difference between replicates

D_2 = 99% confidence upper limit (equivalent to +3sd) on a population mean of replicate averages (when n=2, D_2=3.686).

d_2 = factor that converts a range into a standard deviation between replicates (when n=2, d_2=1.128).

Source of D_4 and d_2: ASTM Manual, *Quality Control of Materials.*

5.3 METHOD PREPARATION CHECKS

When a method preparation check is outside the prescribed limits, a notation, or *flag*, is documented in the final report. The limits are listed in Table 5.1.

Matrix Spikes (MS) are samples that are spiked with the constituents being analyzed. The results are compared to method specified limits or statistically generated limits for a determination of preparation efficiency.

Matrix Spike Duplicates (MSD) are the same as *Matrix Spikes*. The results are compared to the initial *Matrix Spike* result for a determination of the precision of preparation efficiency.

Surrogates are constituents that are not commonly found in the natural environment or in commercial waste products. They are added to the sample at the beginning of the preparation step. In organic chromatographic analysis, they elute at retention times different than target compounds. They are somewhat less susceptible to inferences and are used as an additional determination of preparation efficiency. The strategy used for evaluating surrogate recovery is as follows:

A. If the surrogate recovery falls outside the ±3sd limits, the analyst must:

(1) Rerun the extract.
If the result is within the limits, the analysis is finished.

If the result is still outside the limits, the sample must be re-extracted and rerun on the instrument. If the result is within the limits, the analysis is finished. If it continues to fall outside the limits, the analysis is finished and the final report must be flagged.

OR

(2) Re-extract the sample and rerun on the instrument.

If the result is within the limits, the analysis is finished. If it continues to fall outside the limits, the analysis is finished and the final report must be flagged.

5.4 COMPLETENESS

A data package is considered complete when the following applicable items are finished:

- All appropriate logbooks contain all essential information;
- Data validation has been performed;
- Data files contain raw data, completed data validation forms, and all worksheets that document acceptable accuracy, precision, and flaggable items; and,
- Final results are in the LIMS.

TABLE 5.1
ACCURACY, PRECISION, METHOD PREPARATION: OBJECTIVES AND LIMITS
ICP Metals, AA Metals, Hg (CVAA), Cyanide

Analysis	Calibration Checks	Limits	Method Accuracy Checks	Limits	Method Precision Checks	Limits	Method Preparation Check (Efficiency)	Limits	Method Preparation Check (Precision)	Limits
ICP Metals (Totals and TCLP)	Calibration Verification Standard Continuing Calibration Blanks High Std Linearity Interelement Interference	90-110% ±3sd of historical mean 95-105% 80-120%	Control Blank Spike	80-120% or ±3sd	Control Blank Spike Duplicate	RPD<20 or <Upper Range Limit	Matrix Spike Post-Digestion Spike TCLP Matrix Spike	80-120% 75-125% >50%[1]	Matrix Spike Duplicates and Unspiked Duplicates	RPD<20
AA Metals	Calibration Verification Standard	90-110%	Control Blank Spike	80-120% or ±3sd	Control Blank Spike Duplicate	RPD<20 or <Upper Range Limit	Matrix Spike	80-120%	Matrix Spike Duplicates and Unspiked Duplicates	RPD<20
Hg	Calibration Verification Standard	80-120%	Control Blank Spike	80-120% or ±3sd	Control Blank Spike Duplicate	RPD<20 or <Upper Range Limit	Matrix Spike TCLP Matrix Spike	80-120% >50%[1]	Matrix Spike Duplicates and Unspiked Duplicates	RPD<20
Cyanide	N/A	N/A	Control Blank Spike	85-115% or ±3sd	Control Blank Spike Duplicate	CV<20 or <Upper Range Limit	Matrix Spike	±3sd	N/A	N/A

[1] Perform Method of Standard Additions when (1) the recovery of the spike TCLP extract is <50% and the unspiked extract does not exceed the regulatory level, or (2) the concentration of the metal in the extract is within 20% of the appropriate regulatory level.

TABLE 5.1 (Cont.)

ACCURACY, PRECISION, METHOD PREPARATION: OBJECTIVES AND LIMITS
GCMS VOLATILES

Analysis	Calibration Checks	Limits	Method Accuracy Checks	Limits	Method Precision Checks	Limits	Method Preparation Check (Efficiency)	Limits	Method Preparation Check (Precision)	Limits
GCMS Volatiles	Initial BFB Tuning	As per Table 4, (8260B)	Control Blank Spike (5 MS Compounds)	±3sd	Control Blank Spike Duplicate	<Upper Range Limit	Matrix Spike (5 MS Compounds)	±2sd	Matrix Spike Duplicates	<Upper Range Limit
	Continuing Calibration Compounds	RF RSD <30	OR		OR		Surrogates (3)	±3sd	N/A	N/A
	System Performance Check Compounds	Min RRF 0.300 (0.250 for Bromoform)	Matrix Spike (5 MS Compounds)	±3sd	Matrix Spike Duplicate	<Upper Range Limit				
	Daily SPCC	Min RRF 0.300 (0.250 for Bfm)								
	CCC	<25% difference from initial								
	Internal Standard EICP	50-200% of prior daily std check								

TABLE 5.1 (Cont.)

ACCURACY, PRECISION, METHOD PREPARATION: OBJECTIVES AND LIMITS
GCMS SEMIVOLATILES

Analysis	Calibration Checks	Limits	Method Accuracy Checks	Limits	Method Precision Checks	Limits	Method Preparation Check (Efficiency)	Limits	Method Preparation Check (Precision)	Limits
GCMS Semi-volatiles	Initial DFTPP Tuning Continuing Calibration Compounds System Performance Check Compounds Daily SPCC CCC Internal Standard EICP	As per Table 3, (8270D) RF RSD <30 Min RRF 0.050 Min RRF 0.050 <30% difference from initial 50-200% of prior daily std check	Control Blank Spike (11 MS Compounds) OR Matrix Spike (11 MS Compounds)	±3sd ±3sd	Control Blank Spike Duplicate OR Matrix Spike Duplicate	<Upper Range Limit <Upper Range Limit	Matrix Spike (11 MS Compounds) Surrogates (6)	±2sd ±3sd	Matrix Spike Duplicates N/A	<Upper Range Limit N/A

TABLE 5.1 (Cont.)

ACCURACY, PRECISION, METHOD PREPARATION: OBJECTIVES AND LIMITS
PESTICIDES, PCBs, HOMOLOGS

Analysis	Calibration Checks	Limits	Method Accuracy Checks	Limits	Method Precision Checks	Limits	Method Preparation Check (Efficiency)	Limits	Method Preparation Check (Precision)	Limits
Pesticides/PCBs/ Homologs	Initial Calibration Factor (External Std Method)	RSD<20	Control Blank Spike (6MS Compounds)	±3sd	Control Blank Spike Duplicate	<Upper Range Limit	Matrix Spike (6 MS Compounds)	±2sd	Matrix Spike Duplicates	<Upper Range Limit
			OR		OR		Surrogates	±3sd	N/A	N/A
	Response Factor (Internal Std Method)	RSD<20	Matrix Spike (6 MS Compounds)	±3sd	Matrix Spike Duplicate	<Upper Range Limit				
	4,4'-DDT and Endrin Breakdown	<20%								
	Daily Continuing Calibration Compounds	85-115%								
PCBs only	Initial Calibration Factor (External Std Method)	RSD<20	Laboratory Control Sample	±3sd	Laboratory Control Sample Duplicate	<Upper Range Limit	Matrix Spike	±2sd	Matrix Spike Duplicates	<Upper Range Limit
			OR		OR		Surrogates	±3sd	N/A	N/A
	Daily Continuing Calibration Compounds	85-115%	Matrix Spike	±3sd	Matrix Spike Duplicate	<Upper Range Limit				

TABLE 5.1 (Cont.)

ACCURACY, PRECISION, METHOD PREPARATION: OBJECTIVES AND LIMITS
HERBICIDES, METHANOL

Analysis	Calibration Checks	Limits	Method Accuracy Checks	Limits	Method Precision Checks	Limits	Method Preparation Check (Efficiency)	Limits	Method Preparation Check (Precision)	Limits
Herbicides	Initial Calibration Factor (External Std Method) Daily Continuing Calibration Compounds	RSD<20 85-115%	Control Blank Spike (3 MS Compounds) OR Matrix Spike (3 MS Compounds)	±3sd ±3sd	Control Blank Spike Duplicate OR Matrix Spike Duplicate	<Upper Range Limit <Upper Range Limit	Matrix Spike (3 MS Compounds) Surrogates	±2sd ±3sd	Matrix Spike Duplicates N/A	<Upper Range Limit N/A
Methanol & Other GC Volatiles	Initial Calibration Factor (External Std Method) Daily Continuing Calibration Compounds	RSD<20 85-115%	Control Blank Spike OR Matrix Spike	±3sd ±3sd	Control Blank Spike Duplicate OR Matrix Spike Duplicate	<Upper Range Limit <Upper Range Limit	Matrix Spike Surrogates	±2sd ±3sd	Matrix Spike Duplicates N/A	<Upper Range Limit N/A

TABLE 5.1 (Cont.)

ACCURACY, PRECISION, METHOD PREPARATION: OBJECTIVES AND LIMITS
DIOXINS/FURANS (LOW RESOLUTION)

Analysis	Calibration Checks	Limits	Method Accuracy Checks	Limits	Method Precision Checks	Limits	Method Preparation Check (Efficiency)	Limits	Method Preparation Check (Precision)	Limits
Dioxins/Furans (Low Resolution)	Initial Relative Response Factor	RSD<15 Triplicate injections of each level.	N/A	N/A	N/A	N/A	Internal to Recovery Standard	40-120%	N/A	N/A
	Initial Tuning Isotopic Ratio Measurements w/ Column Performance Check Mixture	As per 8280B Table 9								
	Valley Percent Resolution for 2,3,7,8-TCDD and 1,2,3,4-TCDD	<25								
	Daily/Continuing Mid-level Check Standard	±30% of the Initial Calibration RRFs								
	Daily Tuning Same as Initial Tuning	Same as Initial Tuning								

TABLE 5.1 (Cont.)

ACCURACY, PRECISION, METHOD PREPARATION: OBJECTIVES AND LIMITS
DIOXINS/FURANS (HIGH RESOLUTION)

Analysis	Calibration Checks	Limits	Method Accuracy Checks	Limits	Method Precision Checks	Limits	Method Preparation Check (Efficiency)	Limits	Method Preparation Check (Precision)	Limits
Dioxins/Furans (High Resolution)	Initial Relative Response Factor 17 unlabeled 9 labeled	 RSD<20 RSD<30	N/A	N/A	N/A	N/A	Internal to Recovery Standard	40-135%	Matrix Spikes and Matrix Spike Duplicates	RPD<20
	Initial Tuning Isotopic Ratio Measurements for 17 unlabeled 11 labeled	As per 8290A Table 8							Unspiked Duplicates	RPD<25
	Valley Percent Resolution for Column Performance Check Standard	<25								
	Valley Percent PFK m/z 304.09824 & TCDF m/z 303.9016	<10								
	Daily/Continuing High Resolution Calibration Compound-3 17 unlabeled 9 labeled	 ±20% ±30% of the Initial Calibration RRFs								
	Daily Tuning Same as Initial Tuning	Same as Initial Tuning								
	End Cal Check HRCC-3 17 unlabeled 9 labeled	 RPD<25 RPD<35 of the previous 12hr HRCC-3 Check								

TABLE 5.1 (Cont.)

ACCURACY, PRECISION, METHOD PREPARATION: OBJECTIVES AND LIMITS
WET CHEMISTRY

Analysis	Calibration Checks	Limits	Method Accuracy Checks	Limits	Method Precision Checks	Limits	Method Preparation Check (Efficiency)	Limits	Method Preparation Check (Precision)	Limits
Heat of Combustion (BTU)	Initial Generate an EE value with 6 runs of benzoic acid on two non-consecutive days Daily Benzoic Acid	Results must be within 56 BTU/lb of each other 11373 BTU/lb ±56	Laboratory Control Sample	±200 BTU/lb or ±3sd of historical mean (use the more stringent)	Laboratory Control Sample Duplicate	Within 56 BTU/lb of initial LCS run or <Upper Range Limit (use the more stringent)	N/A	N/A	N/A	N/A
Chloride (for Total Halogens)	Calibration Verification Standard	90-100%	Laboratory Control Sample	±3 sd of historical mean	Laboratory Control Sample Duplicate	<Upper Range Limit	Matrix Spike	±3sd	Matrix Spike Duplicates	<Upper Range Limit
Setaflash Ignitability	n-Butanol	98°F±2, in duplicate	Select a compound with a flashpoint near 140°F	±3°F	N/A	N/A	N/A	N/A	N/A	N/A
Pensky-Marten Ignitability	p-Xylene	81°F ±2, in duplicate	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Percent Moisture:										
Evaporation	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	Duplicate	RPD<10
Karl Fischer	Hydranal	90-100%	N/A	N/A	N/A	N/A	N/A	N/A	Duplicate	RPD<10

TABLE 5.1 (Cont.)

ACCURACY, PRECISION, METHOD PREPARATION: OBJECTIVES AND LIMITS
Wet Chemistry

Analysis	Calibration Checks	Limits	Method Accuracy Checks	Limits	Method Precision Checks	Limits	Method Preparation Check (Efficiency)	Limits	Method Preparation Check (Precision)	Limits
Percent Ash	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	Duplicate	RPD<10
Fluoride (from Combustate)	Calibration Verification Standard	90-100%	Laboratory Control Sample	±3sd of historical mean	Laboratory Control Sample Duplicate	<Upper Range Limit	Matrix Spike	±3sd	Matrix Spike Duplicates	<Upper Range Limit
Viscosity	Calibration Verification Standard	90-100%	N/A	N/A	N/A	N/A	N/A	N/A	Duplicate	RPD<10
Specific Gravity	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	Duplicate	RPD<10
pH:										
Water	pH 4,7,10 Buffers	N/A	Calibration Verification Standard	±0.05 pH Units	N/A	N/A	N/A	N/A	Duplicates on all water samples	±0.1 pH Units
pH Paper	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	Duplicate on all samples	±1 color increment on narrow range paper
Waste	pH 4,7,10 Buffers	N/A	Calibration Verification Standard	±0.05 pH Units	N/A	N/A	N/A	N/A	N/A	N/A
Solids	pH 4,7,10 Buffers	N/A	Calibration Verification Standard	±0.05 pH Unites	N/A	N/A	N/A	N/A	N/A	N/A

6.0 SAMPLING PROCEDURES

A comprehensive program is essential in order to ensure that all samples taken are representative of the waste, that the analysis is complete and accurate, and that the final reports contain sufficient information to achieve their intended purpose. That purpose primarily being the safe and efficient treatment and disposal of hazardous waste.

A sample may or may not require special handling and storage procedures. This is dependent upon the parameter to be analyzed, the sample matrix, and the amount of time prior to analysis. Since the type of sample preservation required varies depending on the sample type and the parameter to be measured, more than one container per sample may be necessary.

All samples are preserved in accordance with the parameter to be measured as specified by the analytical method for that parameter. The analytical methods included in this Quality Assurance Plan refer to the optimum means of preservation. Since the chemical make-up of certain samples can alter the effectiveness of the sample preservation measures, all samples are analyzed as soon as possible after sampling and before the maximum recommended holding time has expired.

Table 6-1 indicates the parameter of interest, appropriate container, preservation, and maximum holding times for samples of various matrix types.

6.1 SAMPLE COLLECTION

The first step in any analysis is the collection of the sample. A wide range of techniques and sampling devices are utilized to sample waste materials in containers, tanks, and process streams.

The sampling methodology is determined by the sampling strategy employed. The methods and equipment used for sampling waste material vary with the form and consistency of the waste materials. The following sampling procedures are utilized for the following types of materials:

Extremely viscous liquids. . . . . ASTM D140-70; SW-846
Crushed or powdered material . . . . ASTM D346-75; SW-846
Soil or rock-like material . . . . . ASTM D420-69; SW-846
Soil-like material . . . . . ASTM 1452-65; SW-846
Fly-ash-like material. . . . . ASTM D2234-76; SW-846
Stratified liquids EPA-600/2-80-018; SW-846

Table 6.1
SAMPLING CONTAINERS, PRESERVATION, AND HOLDING TIMES

MATRIX	ANALYSIS	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME
Solids, Organic Liquids, Sludges	Semi-Volatile Organics	Glass	4°C	Extraction: 14 Days Extract: 40 Days
	Volatile Organics	VOA Vial	4°C	14 Days
	ICP Metals	Glass, Plastic	4°C	6 Months
	Mercury	Glass, Plastic	4°C	38 Days w/Glass 13 Days with/Plastic
	Cyanide	Glass, Plastic	4°C	14 Days
	Wet Chemistry and Fingerprint	Glass, Plastic	4°C	24 Hours
Aqueous Liquids	Semi-Volatile Organics	Glass	4°C	Extraction: 7 Days Extract: 40 Days
	Volatile Organics	VOA Vial	4°C	14 Days
	ICP Metals	Glass, Plastic	4°C, HNO_3 to pH<2	6 Months
	Mercury	Glass, Plastic	4°C, HNO_3 to pH<2	38 Days w/Glass 13 Days w/Plastic
	Cyanide	Glass, Plastic	4°C, NaOH to pH>12	14 Days
	Wet Chemistry and Fingerprint	Glass, Plastic	4°C	24 Hours

6.2 SAMPLING CONTAINERS

The term "container" refers to receptacles designed for transporting materials, e.g., drums and other small receptacles as opposed to stationary tanks. This section addresses sampling of containers that are of a size that could be stored in the container storage building. Sampling of bulk materials in large containers such as rolloffs, tank trucks, etc. is addressed in section 6.4. COLIWASAs, tubes, shovels, drum thieves, and triers are the devices used to sample containers.

A random sampling strategy is employed to sample incoming shipments of containerized waste. Samples from containers holding the same type of waste may be composited. The following procedure will be used to determine how many containers will be sampled and which samples will be composited. Each container will be opened and visually inspected with the exception of those wastes allowed otherwise by the Waste Analysis Plan. Wastes on a single load that have the same profile number and DOT description (excluding waste codes) and appear to be of the same waste type will be grouped together. Ten percent (rounded up) of the containers in each of

these groups will be sampled as described below. The samples within each separate group may be composited for analysis.

A unique tracking number is assigned to each container.

Samples are taken from locations displaced both vertically and horizontally throughout the waste. For liquids (or liquids with precipitated solids), the sampling person uses a COLIWASA or equivalent. The sampling device is inserted into the container from the top and is pushed down slowly until the bottom of the container is reached. The device is sealed to retain the contents. The contents of the sampling device are then transferred to a polyethylene or glass bottle, which is labeled with waste identification information. The sampling device may also be stoppered at both ends, wiped dry with a disposable cloth, and then transferred to the lab for analysis.

A trier or thief is used to sample containers that are solid in nature. These containers are generally filled with dirt and sludges. Several areas from the container are sampled and composited into a jar in order to ensure a representative sample. The sampling person removes a sample that uniformly represents the waste composition of the container, i.e., all layers and phases are represented in the sample.

6.3 SAMPLING TANKS

Liquid and sludge storage and blend tanks at Aragonite are agitated. The tanks are agitated by either a propeller-type mixer or recirculation. The agitation capabilities of the tanks make it possible to obtain a representative sample via a sampling valve. The tanks are agitated prior to drawing a sample. The waste is sampled from a valve on the side or bottom of each tank.

Bulk solids which have been mixed in the bulk solids storage tanks are sampled at a minimum of six locations in the tank. A scoop is taken with the backhoe, or equivalent, from as deep a cross section as possible at each location. A trier, thief or shovel is used in order to collect a sample from each backhoe scoop. The samples are composited together so that there is one sample which represents that particular mix of bulk solids.

6.4 SAMPLING BULK CONTAINERS OF WASTE

Where sampling of bulk loads is required, each bulk container of each load will be sampled as described below.

Bulk solids in rolloffs or end dumps are sampled at two locations in the waste container. A trier, thief or shovel is used in order to draw a sample from as deep a cross section as possible at each location. The samples are composited together so that there is one sample which represents that particular bulk solids shipment.

Bulk liquids are sampled by using a COLIWASA or similar device which can sample vertical anomalies. Bulk sludges are sampled with a device appropriate for the consistency of the

material. That may be a COLIWASA, trier, dip tube, or thief, etc. Each compartment of tanker trucks is sampled. Compartment samples from the same generator and waste stream may be composited prior to analysis.

Tank trucks without man-ways are sampled through the valve. The valve is flushed prior to the sample actually being drawn.

An exception to the requirement for sampling each load of bulk load shipments is where a rail car of liquids or visibly similar solids is divided into multiple bulk tanker or truck loads for final shipment to Aragonite. This will only occur at the Bulk Solids Rail/Truck Transfer facility, Unit 255, and the Bulk Liquids Rail/Truck Transfer Bay, Unit 535, at the Clive facility. In such cases, a representative sample will be taken from each rail car and that sample may be used as the incoming load sample for each of the individual truck or tanker loads from that rail car. For bulk solids, the sample from the rail car will consist of at least six sub-samples taken from equal areas in the rail car at depths of at least one foot. Alternatively, the sample could be collected by compositing at least three grab samples from the backhoe bucket while the waste is being transferred from the rail car to the end dumps or rolloff boxes. For liquids, a representative sample will be taken with a COLIWASA from the hatch of the rail car. Samples will follow chain-of-custody procedures for transport to Aragonite.

Additionally, analyses of samples taken at the Clive facility by Aragonite personnel and analyzed according to the methods specified in the Waste Analysis Plan (Attachment 1) may be used for acceptance and management at Aragonite. These are the only cases in which the incoming load sample may be collected off site.

6.5 SAMPLING SURFACES

40 CFR 761.123 contains standardized EPA procedures for taking PCB surface wipe samples. The definition constitutes the minimum requirements for an appropriate wipe testing protocol. A standard size template (10 cm x 10 cm) is used to identify the sampling area; the wiping media is an all collection gauze pad which has been saturated with hexane. The wipe is performed quickly once the gauze is exposed to air.

7.0 TRACEABILITY

Clean Harbors Aragonite routinely follows sample traceability for all internal sampling and analysis. This involves the documentation of procedures so that a set of data can be traced back through the analyst, to the person performing the sampling, and then to the waste itself. All samples receive a unique sample identification number to facilitate this process.

Should Chain-of-Custody be warranted, i.e., shipping samples off-site, then procedures in Section 7.4, Chain-of-Custody are followed:

In order to trace sample possession from the time of collection, a traceability record is filled out

and accompanies the sample. The record contains the following information:

- sample number;
- signature of the collector;
- date and time collected;
- waste type;
- signature of persons involved;
- inclusive date of possession; and
- cross reference to manifest (if applicable).

7.1 SAMPLE LABELS

Sample labels are necessary to prevent misidentification of samples. The labels are gummed and affixed to the containers prior to or at the time of sampling. The labels are filled out at the time of collection.

Examples of types of sample labels used are shown below (for illustration purposes):

Clean Harbors Aragonite Site Label

I.D.#_____DATE_____
MANIFEST #_____TIME_____
GEN. NAME_____
SAMP. LOCATION_____
PERSON SAMP._____DEPT._____

Clean Harbors Aragonite Laboratory LIMS Label

#9202056-01A ALLIANCE_____
ID APT-0-AT-1-NAOH_____
LOC D3B_____ 02/08/92
CL_IC

7.2 SAMPLE SEALS

Sample seals are used to detect any tampering during shipment for samples sent off site. The seals are initialed, dated, and then affixed to the sample containers or shipping containers before the samples leave the custody of the Aragonite lab. Sample seals are not necessary for samples taken onsite at the Aragonite facility and sent to the onsite laboratory. They are required for

Chain of Custody events.

7.3 SAMPLING LOGBOOK

All information pertinent to field surveys or sampling is recorded in a logbook. Since sampling situations vary widely, no set of rules can be given as to the extent of information that must be entered in the logbook. However, sufficient information is recorded to allow someone to reconstruct the sampling without reliance on the collector's memory. This information is recorded in a bound log book or electronically and includes at a minimum the following information:

- location of sampling point;
- volume of samples taken;
- date and time of collection;
- sample identification number;
- person sampling;
- comments or observations;
- sampling methodology;
- number of samples and disposition

7.4 CHAIN-OF-CUSTODY RECORD

Sample chain-of-custody is maintained as required by the client or regulatory agency. A chain-of-custody is used to ensure legal defensibility of the data from sample collection to data reporting. This includes the ability to trace the possession and handling of samples from the time of collection through analysis and final disposition.

The components of the chain-of-custody include the following: sample seals, a logbook, chain-of-custody record, and sample analysis request sheets. The procedures for their use are described in further detail.

A sample is considered to be under a person's custody if:
- it is in a person's physical possession;
- in view of the person after possession has taken place;
- secured by that person so that no one can tamper with the sample; or
- secured by that person in an area which is restricted to authorized personnel.

Upon receipt of the sample(s) in the laboratory they are entered into the sample receipt logbook. All chain-of-custody samples are directed to the sample custodian. The shipping containers and sample bottles are inspected for proper seals and labels. The contents of the containers are then checked against the chain-of-custody record.

If the chain-of-custody information is complete and the integrity of the samples has not been broken, each sample is assigned a unique identification number.

The samples are then put into storage to await analysis. Maximum holding times for the samples are described in Section 6 of this Quality Assurance Plan.

8.0 CALIBRATION PROCEDURES AND FREQUENCIES

All instruments are calibrated in accordance with the appropriate analytical method. The methods commonly utilized by Clean Harbors Aragonite are referenced in Section 5.0 of the Waste Analysis Plan. These methods cite the appropriate calibration procedures and frequencies. In addition, all instruments are calibrated in accordance with the manufacturer's procedures.

Prior to the analysis of samples, instruments are either calibrated or their calibrations verified. Calibration curves of signal response versus concentration are generated on each applicable analytical instrument. Calibration curves are established for each analyte of interest.

Most methods use multi-point calibrations, usually employing standards at either three or five different concentrations. Calibrations are evaluated using calibration check standards. Should this sample fall outside of acceptable limits as specified by the method, the instrument is recalibrated. Table 8.1 summarizes instrument calibration procedures and frequencies.

Sources of reference materials include the National Bureau of Standards, and reputable commercial vendors.

TABLE 8.1
CALIBRATION PROCEDURES AND FREQUENCIES

Instrument	Standards	Frequency
GC	Mid-level Standard	Daily and every 10th sample.
	5-7 Standards	Recalibration if CVS is greater than 15% of expected value.
GC/MS	Mid-level Standard	Daily
	5-7 Standards	Recalibration if CCC* is greater than 30% for semi-volatiles and 25% for volatiles.
	Mass Calibration (GC/MS tuning)	Every 12 hours.
ICP	Calibration Verification Standard (CVS)	Beginning and end of analytical run and every 10th sample.

	3-5 Standards	Recalibration if CVS not within ± 10% of expected value.
AAS	3-5 Standards	Analysis of standards at the beginning of an analytical run.

* CCC = Continuing Calibration Check

9.0 ANALYTICAL METHODS

The analytical methods which Clean Harbors Aragonite uses are listed in Section 5.0 of the Waste Analysis Plan.

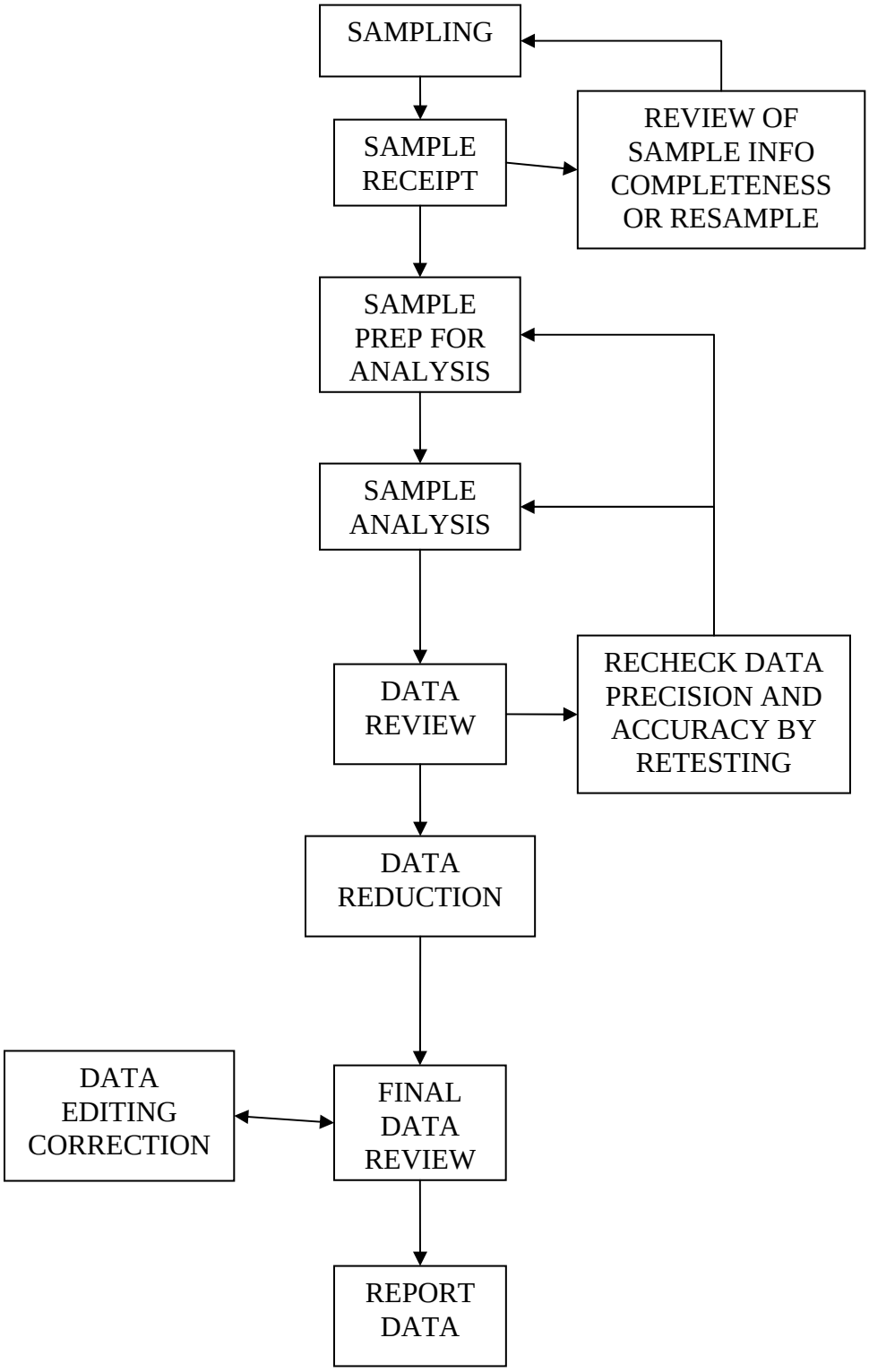
10.0 DATA REDUCTION, VALIDATION, AND REPORTING

Clean Harbors Aragonite data reduction procedures are designed to include several levels of data review. Data validation begins with the person generating data. The chemist or analyst makes the initial calculations and records the results in his/her notebook or on the appropriate worksheet. Each section supervisor or designee is then responsible for reviewing data and calculations generated by their respective group. Final review and case narratives are performed by the Laboratory Manager or designee.

Discrepancies and errors are referred back to the chemist or analyst performing the analysis. If necessary, the samples are reprepared and reanalyzed.

Figure 10.1 depicts the data reporting scheme.

FIGURE 10.1 DATA REPORTING SCHEME


SAMPLING
SAMPLE RECEIPT
REVIEW OF SAMPLE INFO COMPLETENESS OR RESAMPLE
SAMPLE PREP FOR ANALYSIS
SAMPLE ANALYSIS
DATA REVIEW
RECHECK DATA PRECISION AND ACCURACY BY RETESTING
DATA REDUCTION
DATA EDITING CORRECTION
FINAL DATA REVIEW
REPORT DATA

10.1 DATA REDUCTION

Raw data from chromatographs, spectrometers, recorders, and physical measurements are reduced to yield concentrations of the analytes of interest. All data reduction is performed in accordance with the applicable method as referenced in Section 9.0.

Data reduction which is not computerized is recorded in ink on worksheets or in lab notebooks.

10.2 DATA VALIDATION

All data are validated prior to being disseminated from the laboratory. The data are reviewed for both editorial and technical validity.

The editorial review consists of a check for typographical, transpositional, and omissional errors. This review also includes a review of any text which may accompany the data.

The technical review consists of a check to see that all precision, accuracy, and detection limit requirements have been met. In addition, the data are also reviewed for completeness and representativeness.

10.3 DATA REPORTING

Once data have been reviewed and all requirements for completeness, representativeness, precision, accuracy, and limits of detection have been met, results are reported to the client.

Typically, only the final reduced data and case narrative are reported. Clean Harbors Aragonite retains in its records all QC data, calculations, chromatograms, etc., which support the reported data.

11.0 INTERNAL QUALITY CONTROL CHECKS

Clean Harbors Aragonite maintains a minimum level of quality control as described in Chapter 1 "Quality Control," SW-846.

Table 5.1 describes the quality control strategies for each analysis. A glossary of terms is listed in Section 11.2.

11.1 FIELD QUALITY CONTROL

The procedures used in the field to ensure data quality include:

- The use of accepted sampling techniques.

- The justification and documentation of any field action contrary to accepted or specified techniques.
- The documentation of activities, such as container preparation, instrument calibration, etc.
- The documentation of field measurement Quality Control Data.
- The documentation of field activities.
- The documentation of post-field activities including sample shipment and receipt, equipment check in, and de-briefing.
- The generation of Quality Control Samples, including duplicates.

11.2 ANALYTICAL QUALITY CONTROL

The procedures used in the laboratory to ensure analytical data quality include:

Duplicate Spike - is analyzed (when applicable) with every analytical batch or once in ten samples, which ever is more frequent. Analytes stipulated by the method applicable regulations, or agreement with the client, are spiked into the sample. Selection of the sample to be spiked, split, or both depends on the information required and the variety of conditions within a typical matrix. In some situations, requirements of the site being sampled may dictate that the person sampling select a sample to be spiked and split based on a pre-visit evaluation or on-site inspection. Thus does not preclude the laboratory's spiking a sample of its own selection. In most cases, the laboratory will select the sample to be spiked. The laboratory's selection is based on the attempt to determine the extent of matrix bias or interference on the analyte recovery and sample to sample precision.

Blanks - accompany each batch of samples and are carried through the entire analytical procedure.

Surrogate Standards - are spiked into samples according to the appropriate analytical methods. Surrogate spike recoveries will fall within the control limits set by Clean Harbors Aragonite in accordance with the procedures specified in the method.

Check Samples - containing a representative subset of the analytes of interest are used to evaluate equipment performance. The concentration of the analytes approaches the estimate quantification limit in the matrix of the check samples.

Clean-Ups - are used to eliminate interferences in organic extracts. Samples which undergo clean up are checked for percent recovery.

Column-Check Sample - is used to verify column performance. The elution pattern is reconfirmed after activating or de-activating a batch of absorbent.

Instrument Adjustment - requirements and procedures are instrument and method specific. Analytical instrumentation is tuned and aligned in accordance with requirements which are

specific to the instrumentation procedures employed.

Calibration - is performed in accordance with the manufacturers' requirements and the procedures specified in the applicable method.

11.3 SPECIFIC REQUIREMENTS FOR INORGANIC ANALYSIS

Standard curves used in the determination of inorganic analytes are prepared as follows.

Standard curves derived from data consisting of one reagent blank and three to five concentrations are prepared for each analyte. The response for each prepared standard is based upon the average of three replicate readings of each standard. Sample results must fall within the concentration range of the standard curve. If the results of the verification are not within ±10% for ICP and 20% for Atomic Absorption of the original standard curve, a reference standard is employed to determine if the discrepancy is with the standard or with the instrument.

New standards are prepared on a quarterly basis. All data used in drawing or describing the curve are indicated on the curve or its description and a record is made of this verification.

Standard deviations and relative standard deviations are calculated for the percent recovery of analytes from the spike sample duplicates from the check samples.

11.4 SPECIFIC REQUIREMENTS FOR ORGANIC ANALYSIS

The following requirements are applied to the analysis of samples by gas chromatography, liquid chromatography and gas chromatography/mass spectrometry.

The calibration of each instrument is verified at frequencies specified in the methods. Standard curves are prepared as specified in the methods.

The tune of each GC/MS system used for the determination of organic analytes is checked with 4-bromofluorobenzene (BFB) for determinations of volatiles and with decafluorotriphenylphosphine (DFTPP) for determination of semi-volatiles. The required ion abundance criteria are met before determination of any analytes.

If the system does not meet the required specification for one or more of the required ions, the instrument is retuned and rechecked before proceeding with sample analysis. The tune performance check criteria are achieved daily or for each 12 hour operation period, whichever is more frequent.

The background subtraction is straightforward and designed only to eliminate column bleed or instrument background. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting special requirements are contrary to the objectives of Quality Assurance

and are unacceptable.

For determinations by HPLC or GC, the instrument calibration is verified as specified in the methods.

12.0 PERFORMANCE AND SYSTEM AUDITS

The laboratory is subject to both internal and external audits, in order to monitor the capability and performance of the total measurement systems.

The systems audit consists of evaluation of all components of the measurement system to determine their proper selection and use. This audit includes a careful evaluation of both field and laboratory quality control procedures. System audits are normally performed prior to or shortly after a new system has been implemented. Performance audits are then performed on a routine basis, at least quarterly, during the lifetime or continuing operation of the system.

12.1 EXTERNAL AUDITS

Clean Harbors laboratories participate in EPA WP Study semiannual blind round robin tests with other laboratories who perform environmental analysis.

A set of blind samples are split among the laboratories. This helps Clean Harbors Aragonite evaluate the precision and accuracy of its own laboratories, as well as provide information about the amount of interlaboratory deviation which can be associated with a particular method.

Corrective action is taken as described in Section 14 of this QAP.

12.2 INTERNAL AUDITS

Internal audits are performed on a quarterly basis. The audit is conducted by the Quality Assurance Officer under the direction of the Laboratory Manager. The audit report is due 30 days following the conclusion of the quarter.

The audit evaluates the system from the receipt of samples to the reporting of results. Specific areas which are addressed include: sample flow through the lab, sample storage, sample preparation, analysis, data reduction, data reporting, QC samples, logbooks, and raw data storage.

13.0 PREVENTATIVE MAINTENANCE

Clean Harbors laboratories are equipped and maintained to provide the best conditions possible for performing laboratory analysis. Equipment which has become obsolete by the advancement of technology is replaced or upgraded. All equipment is inspected regularly to ensure that it is in proper working order.

Equipment is maintained in accordance with the manufacturer's recommendations. All major pieces of equipment are covered by service contracts from the manufacturer. Whenever possible, Clean Harbors Aragonite maintains an inventory of spare parts which typically need replacement, this includes such compounds as septa, GC columns, ion volumes, torches, regulators, and so forth.

Table 13.1 lists pieces of equipment or components which are routinely maintained, the frequency at which they are serviced and the type of maintenance performed.

TABLE 13.1
MAINTENANCE SCHEDULE

EQUIPMENT COMPONENT	MAINTENANCE PERFORMED	FREQUENCY
Gas Chromatographs		
septa	replace	as required
column	replace/condition	as required
syringes	replace	as required
inlet liner (tube)	clean/replace	as required
ELCD (HALL)	leak check	as required
Ni catalyst	replace/condition	as required
solvent resin	replace	as required
ECD	wipe test	semi-annually
	leak check	as required
	factory clean/recondition	as required
PID	leak check	as required
lamp	replace	as required
FID	leak check	as required
jets	clean	as required
ICP		
nebulizer	clean/replace	as required
pump tubing	replace	weekly
air filters	clean	as required
torch	clean/replace	as required
MERCURY ANALYZER		
drying tube desiccant	replace	daily
sample tubing	replace	twice/week
stannous chloride tubing	replace	once/2 weeks
drain tubing	replace	once/2 weeks
lamp	replace	as required
optics	clean	as required
CALORIMETER		
bombs	calibration/certification	after 500 firings
tubing	check/replace	daily
COMPRESSED GASES		
fittings	leak checks	as required
traps	replace	as required

14.0 CORRECTIVE ACTION

Quality Control procedures are designed to identify the need for corrective action. Most corrective actions are performed by the chemists doing the analysis, and are usually as simple as recalibrating an instrument should the instrument check sample be out of its acceptable range. Most corrective actions are found in methods, standard operating manuals, and instrument manuals.

Corrective actions may also be initiated as a result of various Quality Assurance activities, including:

1) performance audits,
2) system audits,
3) laboratory or interfield comparison studies,
4) program audits, and
5) final review of data reports

Corrective action reports will be sent to the Laboratory Manager for review and implementation.

However, standard operating procedures are to:

1) define the problem,
2) determine the cause(s) of the problem,
3) determine possible solutions to the problem,
4) implement the corrective action, and
5) verify that the corrective action is effective.

All employees are encouraged to bring to their supervisor's attention any problem or practice which they feel may affect data quality.

15.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Clean Harbors Aragonite Quality Control Officer is responsible for reporting to the Laboratory Manager every four months on the performance of measurement systems and data quality. The Laboratory and Plant Manager reviews and returns the report. These reports include:

1) Assessment of measurement data accuracy, precision, and completeness.
2) Results of performance audits.
3) Results of system audits.
4) Significant Quality Assurance problems and recommended solutions.