APPENDIX E-6

Feedstream Analysis Plan

TAPI Puerto Rico, Inc.

Feed Stream Analysis Plan (FSAP)

Rev. 3
5/1/2012

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1.0 Introduction

TAPI Puerto Rico, Inc. (TAPI) has developed this Feed Stream Analysis Plan (FSA) in accordance with the provisions of 40 CFR §63.1209(c)(2). The Plan establishes procedures to obtain information on the physical and chemical characteristics of hazardous waste generated in TAPI's pharmaceutical manufacturing process and treated in any of two on-site hazardous waste incinerators - the Trane 1 and the Trane 2 units. Procedures for sampling and analysis of the kerosene feed to the incinerators as an auxiliary fuel is also included. The objective of the Plan is to gather information sufficient to document compliance with ash, chlorine, and metals feed rate limits established to comply with applicable Hazardous Waste Combustor (HWC) MACT emission standards.

2.0 Hazardous Waste Incinerators

The two incinerators are Sub- X Liquid Waste Oxidizers manufactured by the Trane Thermal Company of Conshohocken, Pennsylvania. They do not have model numbers. Trane 1 was installed in 1977. Trane 2 was installed in 1979. The incinerators are down fired liquid waste incinerators each equipped with a quench tank, high-energy Venturi scrubber and separator (including a mist eliminator). The units are equipped with a common Wet Electrostatic Precipitator (WESP) and stack. A conceptual process flow diagram of the two incinerators and the common WESP and stack is presented in Figure 1.

The incinerators treat hazardous and process wastewater and organic solvents generated at TAPI's facilities during the synthesis of intermediate and final pharmaceutical products. Sources of hazardous waste include, but are not limited to, mother liquors, extraction wash, solvent recovery bottoms, filter wash and equipment wash. Hazardous waste streams are conveyed from various production buildings via dedicated above ground stainless steel pipelines to TAPI's hazardous waste storage tanks.

Process wastes are segregated at the point of generation into two waste streams: low heat value aqueous waste and high heat value organic waste. Both aqueous and organic wastes are treated in TAPI's incinerators. Aqueous and organic waste streams are not blended prior to firing to the incinerators. Each waste stream is injected into the combustion chamber through a dedicated nozzle. The current RCRA Permit limit for aqueous waste feed to each incinerator to 12.5 liters per minute (Lpm) and organic waste feed to each incinerator to 4.5 Lpm. A description of each waste stream is presented below.

Aqueous Waste - Aqueous waste includes waste from the manufacturing processes, Laboratory operations and maintenance activities. The laboratory operations contribute less than 1% of the total process wastewater generated at the facility. The waste is identified as an aqueous process wastewater because it contains greater than 50% water by weight. Aqueous waste is classified as hazardous due to the potential presence of spent solvents including methylene chloride (F002), methyl alcohol (F003),

ethyl acetate (F003), n-butanol (F003), and toluene (F005). In addition, the waste may have an ignitability characteristic (D001) due to the presence of ethyl acetate, ethyl alcohol and isopropanol (among others). The waste may contain trace levels of metals.

Organic Waste - Organic waste includes waste from the manufacturing processes, laboratory operations, and maintenance activities. The laboratory operations contribute less than 1% of the total process wastewater generated at the facility. The waste is identified as organic because it may contain less than 50% water by weight. Organic waste is classified as hazardous due to the potential presence of spent solvents including methylene chloride (F002), methyl alcohol (F003), xylene (F003), ethyl acetate (F003), n-butanol (F003), and toluene (F005). In addition, the waste may have an ignitability characteristic (D001) due to the presence of ethyl alcohol and isopropanol (among others). The waste may contain trace levels of metals.

Each incinerator uses kerosene as an auxiliary fuel at a maximum rate of approximately 4.8 Lpm, although auxiliary fuel is typically not required, or is required at only minimal flow rates. Propane is used as a pilot for the waste burner during the startup sequence but is not used during burning of hazardous waste.

Other than the feed streams described above and process air, there are no feed streams injected into the incinerators, such as vapor from recovery systems.

3.0 Feed Stream Analysis Procedures

The Plan applies to the following feed streams that are fed to the incinerators:

- Aqueous hazardous waste
- Organic hazardous waste
- Kerosene

Characterization of aqueous waste, organic waste and kerosene is accomplished by sampling and analysis of the feed streams in accordance with the procedures established in this Plan. Concentrations of ash, chlorine and metals in feed streams are determined to ensure compliance with applicable Hazardous Waste Combustor MACT emission standards. Feed stream characteristics including heating value, specific gravity, viscosity and pH are determined to ensure safe operation of the incinerators and good combustion practices.

Selection of sampling parameters, sampling and analytical methods, sampling frequency, and quality assurance procedures are discussed in the following sections and are summarized in Table 1.

3.1 Sampling Parameters

Feed streams subject to emissions limits under applicable MACT regulations as well as corresponding regulatory citations are summarized in Table 1. These limits are included in TAPI's Document of Compliance (DOC) which is maintained in the operating record.

Feed stream parameters have been selected for analysis to ensure compliance with the maximum emission limits specified above and to ensure safe incinerator operation and good combustion practices. The feed stream parameters selected for analysis are shown in Table 2.

3.2 Sampling Methods

All feed streams to be sampled in accordance with this Plan are pumpable, free-flowing liquids within pressurized pipe. Accordingly, tap sampling as per ASTM 4057-95 is the most appropriate method for collecting samples (USEPA, 1983).

Samples of aqueous and organic waste are collected from the tap sampling port at the diaphragm pump located at the respective waste feed tank. Organic waste is typically fed to the incinerators from storage tanks V-450 and V-451 and aqueous waste is typically fed to the incinerators from storage tanks V-452 and V-453. All feed tanks are aboveground, vertical stainless steel tanks of 20,000 gallons capacity. A conceptual diagram of the waste feed system including feed stream sample locations is presented in Figure 2. Arrangement of feed tanks may be modified in the future.

Although the tanks for the organic waste tanks (V-450 and V-451) are connected to a common pipe that feeds the incinerators, only one of these tanks is used at anyone time to feed the organic waste to the incinerators. Similarly, only one of the aqueous waste tanks (V-452 and V-453) is used at anyone time to feed aqueous waste to the incinerators. No mixing occurs in the common feed pipes. Overall waste feed to the incinerators may consist of aqueous waste alone, organic waste alone, or combined aqueous and organic waste.

Current waste feed sampling procedures are set forth in TAPI's Standard Operating Procedure entitled "Sampling of Liquid Waste in Tanks." A copy of the SOP is included in Appendix A to this Plan. Procedures identified in the SOP include, but are not limited to:

- Sample collection procedures
- Sample containers, preservation requirements, and holding times
- Chain of custody

- Health and safety requirements
- Quality assurance/quality control

Samples of kerosene are collected from a tap sampling port located at the kerosene storage tank. Sampling procedures for kerosene conform to the procedures for aqueous and organic waste set forth in the above-referenced SOP.

3.3 Analytical Methods

TAPI uses reliable analytical methods to determine feed stream concentrations of ash, chlorine and metals in accordance with 40 CFR §63.1208(b)(8). All analytical work is performed by an offsite laboratory. Parameters and analytical methods used by the laboratory are presented in Table 3.

TAPI may elect to use alternative analytical methods. For all methods to be used to determine concentrations of ash, chlorine and metals in feed streams, the following requirements must be satisfied:

- Analysis procedures are unbiased, precise and that the results are representative of the feed stream
- For each feed stream, the method must demonstrate that each analyte is not present above the reported level at the 80% upper confidence limit around the mean
- For each feed stream, the method must demonstrate that the analysis could have detected the presence of the constituent at or below the reported level at the 80% upper confidence limit around the mean.

3.4 Quality Assurance/Quality Control

In selecting the laboratory for performance of feed stream analyses, TAPI ensures that the laboratory has demonstrated experience and capabilities in the following areas as per USEPA guidance (USEP A, 1994a):

Comprehensive QA/QC Program - The laboratory must document that a comprehensive QA/QC program is in place. The program should conform to the applicable requirements of "Test Methods For Evaluating Solid Waste: Physical, Chemical Methods," (SW-846). The QA/QC program must include qualitative QA/QC elements such as chain-of-custody protocol. The program must also use quantitative QA/QC measures including duplicates, method blanks, calibration blanks, matrix spikes, and/or matrix spike duplicates, as appropriate.

The laboratory must employ a system to review all analytical results to identify any QA/QC problems experienced. Chain of custody documentation must be checked to ensure the correctness of sample bottle labeling and sample preservation, and to identify any breakage of sample containers, loss of sample integrity, use of improper sample containers, evidence of tampering and other problems. The continuity and completeness of sample tracking with chain of custody papers must be checked. The results must be reviewed to ensure that the samples were analyzed within holding times. On a quantitative basis, quality control sample results must be reviewed to ensure the precision and accuracy of the analytical results. This will include review of such quality control samples as duplicates, method blanks, calibration blanks, matrix spikes, and/or matrix spike duplicates, as appropriate.

Technical Analytical Expertise - The selected laboratory must demonstrate proficiency in using established EPA, ASTM or equivalent analytical methods. The laboratory must demonstrate that analytical equipment is capable of consistently achieving required detection limits. To address the laboratory's proficiency, TAPI may conduct periodic audits of the laboratory addressing the laboratory's knowledge of relevant methods, detection limits achievable for the analytes, versatility of the laboratory to adopt alternative analytical methods, and certification of the laboratory (if applicable). TAPI may review the laboratory's QA/QC plan for consistency with stipulated QA/QC procedures for appropriate methods.

Information Management - The laboratory must maintain effective information management systems to assure that regulatory compliance can be verified and process performance can be evaluated. The laboratory must maintain relevant QA/QC documentation and make it readily available for review upon request.

4.0 Sampling Frequency

Feed stream sampling and analysis is conducted on either a batch basis or a statistical basis, depending on the parameters to be analyzed.

4.1 Batch Sampling and Analysis

TAPI samples and analyzes every batch of aqueous and organic waste to be incinerated for the following Hazardous Waste Combustor MACT required parameters:

- Ash
- Total chlorine
- Metals

Analyses are completed prior to commencement of burning any given batch to ensure that the feed stream limits for these parameters are not exceeded. A batch will generally consist of one tank volume of waste ranging up to 20,000 gallons, although smaller batches that may be burned based on specific needs.

Each batch of aqueous and organic waste will also be sampled and analyzed for heating value, specific gravity, viscosity, and pH prior to burning.

4.2 Statistical Sampling and Analysis

TAPI may also implement a statistical approach in determining sampling frequency of aqueous and organic waste for parameters shown in Table 3.

A statistical approach is appropriate because TAPI's waste feed has demonstrated consistent chemical and physical characteristics over time and because there is a reasonable expectation that the results of these parameters are normally distributed. Also, TAPI has sufficient knowledge of the waste generation process to be aware of any change that is likely to affect the sample distribution and thus trigger the need for a new statistical profile (USEPA, 1994b). It is also expected that the physical and chemical characteristics of kerosene will remain consistent over time. The methodology that may be employed by TAPI in implementing the statistical sampling program is summarized below.

TAPI uses an upper tolerance limit (UTL) approach to statistical analysis. A sampling program has been established for each parameter which demonstrates that, with 95% or greater confidence, 95% or greater (coverage) of all individual samples will not exceed the UTL. For the case of 95% confidence at 95% coverage, the approach is summarized below.

The following equation is used to determine the sampling frequency required to achieve the stated confidence levels:

Number of samples/year = (α_{calc}) x (Y)

Where:

α_{calc} = One minus the level of confidence used to calculate the UTL

Y = Days per year on which waste is generated

For example, at a confidence level of 95%: $\alpha_{calc} = 1 - 0.95 = 0.05$

Number of days the waste is generated: Y = 365

Number of samples per year: Samples = α_{calc} x (365) = 18.25

Therefore, the minimum waste feed and kerosene sampling frequency required to achieve a 95% confidence level is 19 times per year (18.25 rounded upward provide an extra degree of certainty). Sampling events are distributed evenly throughout the year.

The UTL will be statistically derived for waste feed and kerosene after completion of an adequate number of sampling events (minimum of four). The methodology used in determining the UTL is consistent with EPA guidance (Hahn, 1991). The UTL will be used in the feed rate calculations. Until a UTL is determined, the maximum reported value for any parameter is used in the feed rate calculation.

If, after development of the UTL, an analytical result indicates that a UTL for any parameter has been exceeded, the following procedures will be followed:

- Actual values will be used for constituent feed rate calculations whenever an UTL is exceeded.

- Immediately following receipt of an analytical result that exceeds the UTL for any parameter, confirmatory sampling and analysis of the feed stream for that parameter will commence. For waste feed, a sample will be collected from each subsequent batch of waste feed. For kerosene, a sample will be collected after each subsequent delivery and filling of the kerosene tank.

- Confirmatory sampling and analyses will continue until the regulated parameter is below its UTL for three consecutive sampling events.

- If the feed stream exceeds the UTL for the same parameter two or more times while conducting confirmatory sampling, a new feed profile will be developed. The UTL for that parameter will be recalculated by performing statistical analysis using data obtained after the initial UTL exceedance. Sampling shall proceed at the normal frequency. During the period that data is being collected for the new feed profile, the maximum reported value for any parameter from the new data will be used for the feed rate calculation.

- If the feed stream exceeds the UTL for the same parameter less than two times while conducting the confirmatory sampling, then the sampling result indicating an exceedance will be considered an outlier and will be discounted. TAPI will investigate the cause of the outlier (e.g., analytical errors) and will take corrective measures necessary to prevent such incidents in the future.

- Historically, metal concentrations in the hazardous waste incinerator feed have been extremely low, frequently below analytical method detection limits. It is also expected that levels of metals, ash, and chlorine will be low in kerosene. For the purposes of calculating a mean and variance, non-detected results will be replaced by the detection limit, with the assumption that all measurements were observable with the same precision.

- If a normal or log-normal distribution cannot be demonstrated and meaningful statistical analysis cannot be conducted for any parameter because of high rate of non-detects (typically greater than 50% non-detects), then, for the purposes of demonstrating compliance with feed rate limitations, statistical sampling shall continue and the maximum reported value will be input into the feed rate calculations as described in Section 5. Use of the maximum reported value is equivalent to the non-parametric UTL described in USEP A (1992).

- A more conservative confidence level (i.e., greater than 95%) maybe used to decrease the sampling and analysis frequency (USEP A, 1994b). Such frequency will be determined using the equation presented above. For example, at a 99% confidence level, the required sampling frequency would be 4 times per year.

- A coverage level greater than 95% may be used to provide a more conservative UTL (USEPA, 1994b).

- Any data outliers will be identified using ASTM Method E178-02 (Standard Practice for Dealing With Outlying Observations), normal quantile plots, box-plots, or equivalent methods. The rationale for rejection of any data outliers will be documented.

Data entry procedure for analytical data is presented in Appendix B.

5.0 Compliance Documentation

To demonstrate compliance with applicable feed rate limits, TAPI will monitor and record feed rates as follows:

- TAPI will determine and record the value of the each feed stream parameter (ash, chlorine, mercury, semi volatile metals, and low volatile metals) by sampling and analysis, as described in this Plan.

- TAPI will determine and record the volume flow rate for each feed stream (aqueous waste, organic waste, kerosene) by a Continuous Monitoring System.

- TAPI will calculate and record the total mass feed rate of each parameter per unit time (on a 12 hour rolling average) and will continuously compare the value to the corresponding feed stream limit. Exceedance of a feed stream limit will trigger an automatic waste feed cutoff.

6.0 References

Hahn, Gerald J. and Meeker, William Q. 1991. Statistical Intervals, A Guide for Practitioners. John Wiley and Sons, Inc.

U.S. Environmental Protection Agency (EP A). 1983. Sampling and Analysis Methods for Hazardous Waste Combustion. Arthur D. Little, Inc.

U.S. Environmental Protection Agency (EPA). 1992. Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities, Addendum to Interim Final Guidance, Draft.

U.S. Environmental Protection Agency (EPA). 1994a. Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes (Draft). OSWER 9938.4-03.

U.S. Environmental Protection Agency (EPA). 1994b. Waste Analysis Guidance for Facilities that Burn Hazardous Wastes (Draft). EPA/540/R-94-0l9.

Tables

Table 1 Maximum Emission Limits and Regulatory Basis

Regulatory Requirement	Parameter	Emission Standard (Limit)	Feed Rate Limit (Lbs/Hr)
40 CFR § 63.1219(a)(1)(ii)	Dioxins and Furans	0.40 ng TEQ/dscm corrected to 7% oxygen	12.8 lpm aqueous waste and 4.7 lpm organic waste
40 CFR § 63.1219(a)(2)	Mercury	130 µg/dscm corrected to 7% oxygen	356 E-06
40 CFR § 63.1219(a)(3)	Lead	230 µg/dscm, combined emissions, corrected to 7% oxygen	3,970 E-06
	Cadmium		
40 CFR § 63.1219(a)(4)	Beryllium	92 µg/dscm, combined emissions, corrected to 7% oxygen	5,210 E-06
	Arsenic		
	Chromium		
40 CFR § 63.1219(a)(5)(i)	Carbon Monoxide	100 ppmv, hourly rolling average, dry basis, corrected to 7% oxygen	NA
40 CFR § 63.1219(a)(6)	Hydrogen Chloride and Chlorine Gas	32 ppmv, hourly rolling average, dry basis, corrected to 7% oxygen	241 Lbs/hr
40 CFR § 63.1219(a)(7)	Particulate Matter	0.013 gr/dscf corrected to 7% oxygen	226 Lbs/hr
40 CFR § 63.1219(c)(1)	Destruction and Removal Efficiency	99.99 for each principal organic hazardous constituent	12.8 lpm aqueous waste and 4.7 lpm organic waste

Notes

Ng – nanogram

TEQ – toxic equivalent quotient

dscm – dry standard cubic meter

µg - microgram

ppmv – parts per million by volume

mg – milligram

Gr - grains

dscf – dry standard cubic feet

Table 2 Chemical Analyses

Stream	Aqueous and Organic Waste	Kerosene
Chemical Analyses Parameters	Mercury	Mercury
	Lead	Lead
	Cadmium	Cadmium
	Beryllium	Beryllium
	Arsenic	Arsenic
	Chromium	Chromium
	Ash	Ash
	Total Chlorine	Total Chlorine
	Heating Value	
	Viscosity	
	Specific Gravity	
	pH	

Table 3 Parameters and Analytical Methods

Parameter	Analytical Method
Ash	ASTM D482
Total Chlorine	ASTM D808
Mercury	SW-846 Method 7470A
Cadmium	SW-846 Method 6010C
Lead	SW-846 Method 6010C
Beryllium	SW-846 Method 6010C
Arsenic	SW-846 Method 6010C
Chromium	SW-846 Method 6010C
Heating Value	ASTM D240-02
Specific Gravity	ASTM D891B-95
Viscosity	ASTM D445-03
pH	SW-846 Method 9040C

Notes:

ASTM: American Society for Testing and Materials

SW-846: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA Publication SW-846

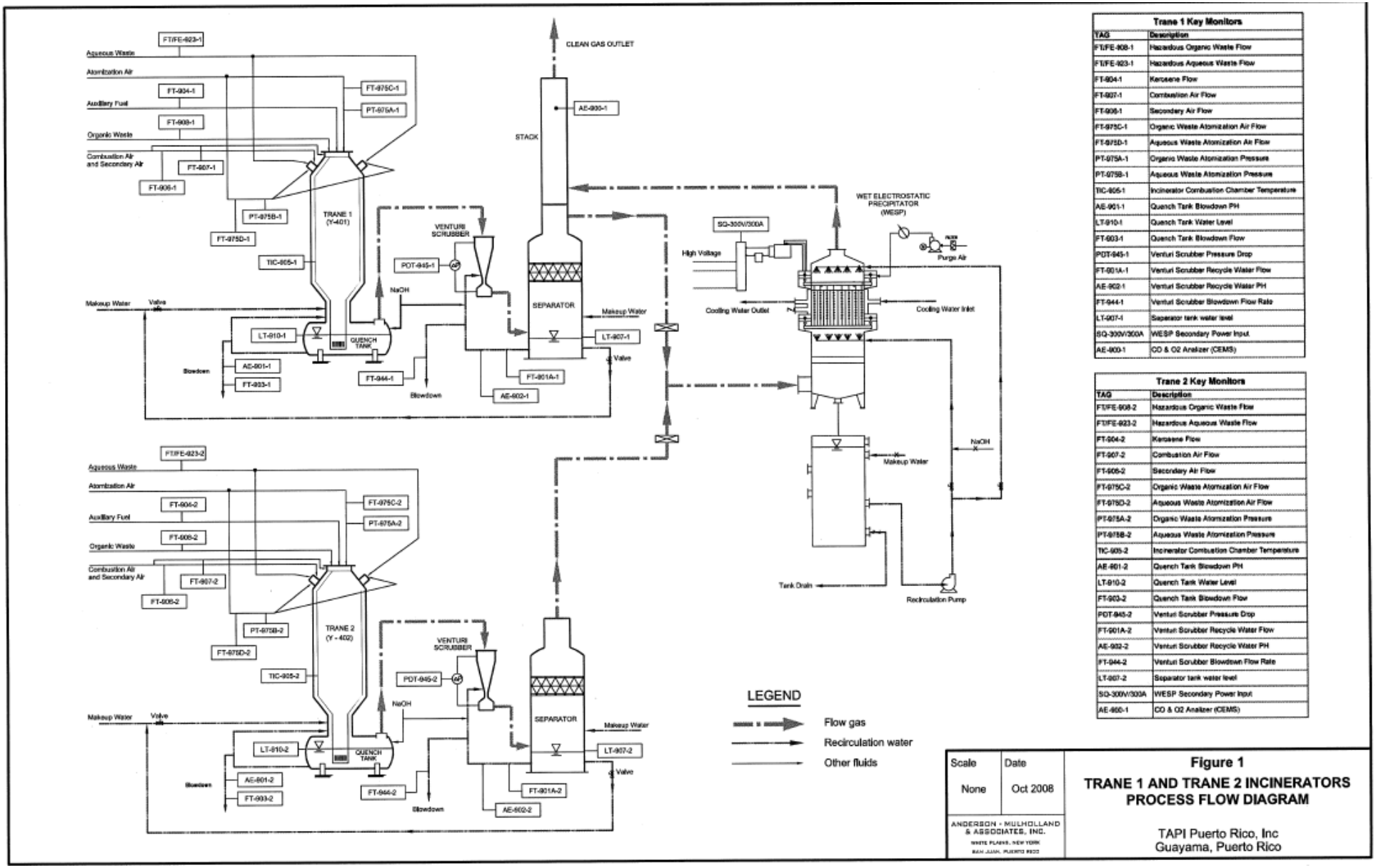
Table 4 Summary of Feed Stream Sampling and Analysis Program

Feed Stream	Parameter	ASTM Sampling Method	Sample Preparation Method[1]	Analytical Method	Frequency[2]
Hazardous Waste (aqueous and organic)	Ash	4057-95	NA	ASTM D482	Batch
	Total Chlorine	4057-95	NA	ASTM D808	Batch
	Mercury	4057-95	3020A	SW-846 Method 7470A	Batch
	Cadmium	4057-95	3010A/3020A	SW-846 Method 6010C	Batch
	Lead	4057-95	3010A/3020A	SW-846 Method 6010C	Batch
	Beryllium	4057-95	3010A/3020A	SW-846 Method 6010C	Batch
	Arsenic	4057-95	3010A/3020A	SW-846 Method 6010C	Batch
	Chromium	4057-95	3010A/3020A	SW-846 Method 6010C	Batch
	Specific Gravity	4057-95	NA	ASTM D891B-95	Batch
	Heating Value	4057-95	NA	ASTM D240-2	Batch
	pH	4057-95	NA	SW-846 Method 9040C	Batch
	Viscosity	4057-95	NA	ASTM D445-03	Batch
Kerosene	Ash	4057-95	NA	ASTM D482	Statistical
	Total Chlorine	4057-95	NA	ASTM D808	Statistical
	Mercury	4057-95	3020A	7470A	Statistical
	Cadmium	4057-95	3010A/3020A	6010C	Statistical
	Lead	4057-95	3010A/3020A	6010C	Statistical
	Beryllium	4057-95	3010A/3020A	6010C	Statistical
	Arsenic	4057-95	3010A/3020A	6010C	Statistical
	Chromium	4057-95	3010A/3020A	6010C	Statistical

[1] Unless otherwise noted, methods are from SW-846
[2] "batch" –every batch is tested prior to firing; "Statistical" – frequency based on a statistical method as per Section 4 of this plan.

Figures

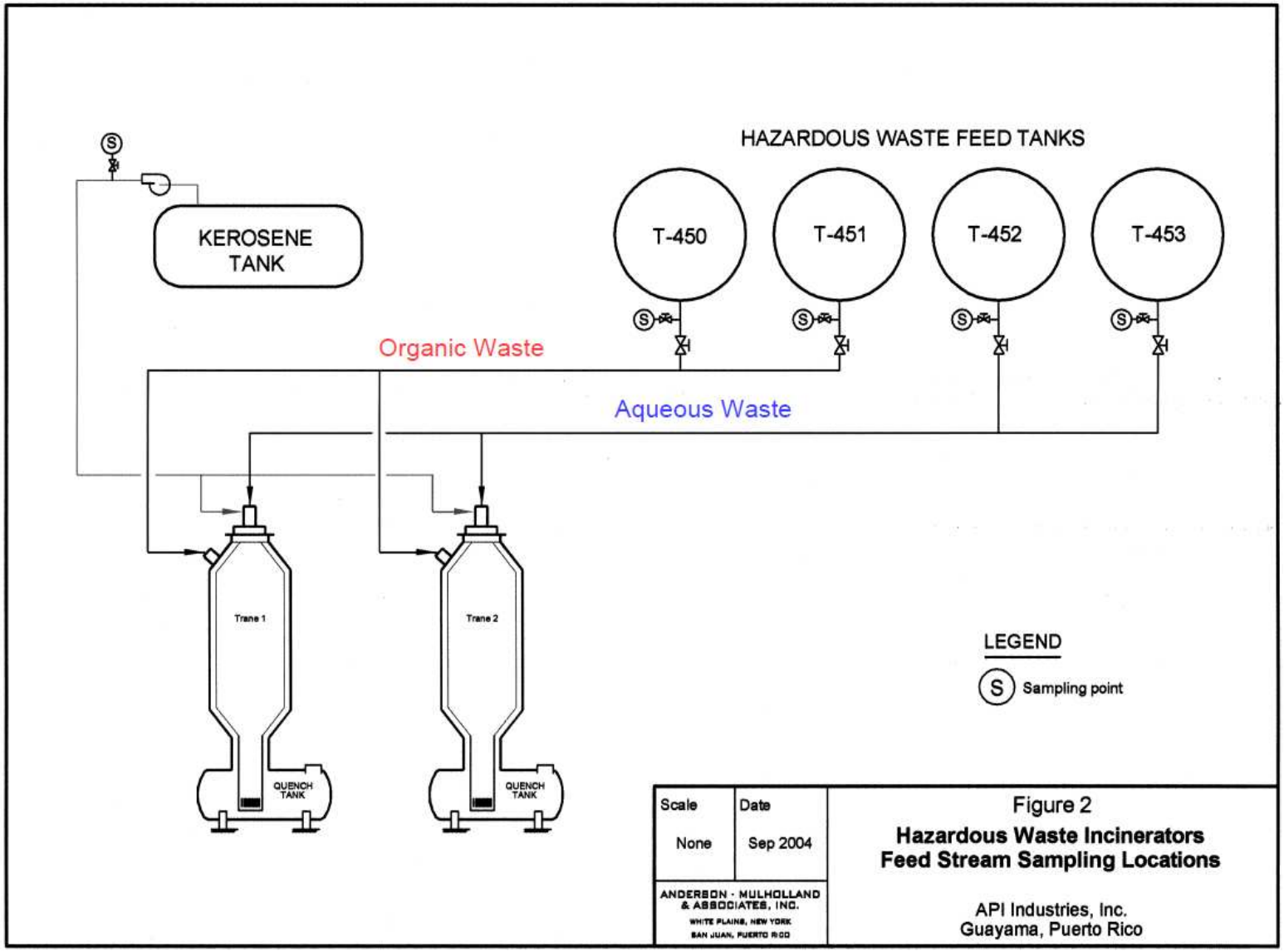
Figure 1 Trane 1 and Trane 2 Incinerators Process Flow Diagram



Trane 1 Key Monitors	
TAG	Description
FT/FE-908-1	Hazardous Organic Waste Flow
FT/FE-923-1	Hazardous Aqueous Waste Flow
FT-904-1	Kerosene Flow
FT-907-1	Combustion Air Flow
FT-906-1	Secondary Air Flow
FT-975C-1	Organic Waste Atomization Air Flow
FT-975D-1	Aqueous Waste Atomization Air Flow
PT-975A-1	Organic Waste Atomization Pressure
PT-975B-1	Aqueous Waste Atomization Pressure
TIC-905-1	Incinerator Combustion Chamber Temperature
AE-901-1	Quench Tank Blowdown PH
LT-910-1	Quench Tank Water Level
FT-903-1	Quench Tank Blowdown Flow
PDT-945-1	Venturi Scrubber Pressure Drop
FT-901A-1	Venturi Scrubber Recycle Water Flow
AE-902-1	Venturi Scrubber Recycle Water PH
FT-944-1	Venturi Scrubber Blowdown Flow Rate
LT-907-1	Separator tank water level
SQ-300V/300A	WESP Secondary Power Input
AE-900-1	CO & O2 Analizer (CEMS)

Trane 2 Key Monitors	
TAG	Description
FT/FE-908-2	Hazardous Organic Waste Flow
FT/FE-923-2	Hazardous Aqueous Waste Flow
FT-904-2	Kerosene Flow
FT-907-2	Combustion Air Flow
FT-906-2	Secondary Air Flow
FT-975C-2	Organic Waste Atomization Air Flow
FT-975D-2	Aqueous Waste Atomization Air Flow
PT-975A-2	Organic Waste Atomization Pressure
PT-975B-2	Aqueous Waste Atomization Pressure
TIC-905-2	Incinerator Combustion Chamber Temperature
AE-901-2	Quench Tank Blowdown PH
LT-910-2	Quench Tank Water Level
FT-903-2	Quench Tank Blowdown Flow
PDT-945-2	Venturi Scrubber Pressure Drop
FT-901A-2	Venturi Scrubber Recycle Water Flow
AE-902-2	Venturi Scrubber Recycle Water PH
FT-944-2	Venturi Scrubber Blowdown Flow Rate
LT-907-2	Separator tank water level
SQ-300V/300A	WESP Secondary Power Input
AE-900-1	CO & O2 Analizer (CEMS)

Figure 2 Hazardous Waste Incinerators Feed Stream Sampling Locations



Appendixes

Appendix A Standard Operating Procedure for the Sampling of Liquid Waste in Tanks

Muestreo y Análisis de los Desperdicios Líquidos a Incinerar

APPROVALS

#	Role	Approver Name	Approval Date
	SOP BU Maintenance (MT)	Hector Rivera	27/Mar/2012 11:10:41 PM
	SOP BU QA	Zaida Caban	30/Mar/2012 8:55:41 PM

This SOP was approved and signed electronically
and this is the manifestation of the electronic signatures.

1. PROPOSITO

1.1 Establecer las guías para el muestreo y análisis de los desperdicios líquidos a incinerar.

2. ALCANCE Y APLICACION

2.1 El contenido de este procedimiento es para ser utilizados por el personal de operaciones ambientales durante el recogido y manipulación de muestras de desperdicios peligrosos líquidos.

3. RESPONSABILIDADES

3.1 Es la responsabilidad del Operador Ambiental el llevar a cabo éste procedimiento tomando las debidas precauciones de seguridad en conformidad con las normas de seguridad establecidas para minimizar la exposición a los riesgos potenciales a la salud mediante el uso de ropa protectora, gafas de seguridad y guantes compatibles con la sustancia que se pretende muestrear.

3.2 Es la responsabilidad del Ingeniero de Operaciones Ambientales el proveer las directrices y seleccionar los equipos necesarios para garantizar que la implementación de este procedimiento no represente un riesgo a la salud y al medio ambiente.

3.3 Es la responsabilidad del Gerente de Cumplimiento Ambiental seleccionar un laboratorio ambiental externo para el análisis de la muestra.

4. DEFINICIONES

4.1 N/A

5. REFERENCIAS

5.1 29 CFR 1910 Subpart I
5.2 40 CFR Parts 260-265

6. PROCEDIMIENTO

6.1 Operador Ambiental-Muestreo

6.1.1 Como medidas de Seguridad se requiere como mínimo utilizar gafas de seguridad, guantes de nitrilo y respirador de media cara, antes de tomar la muestra.

6.1.2 Utilizar botella de cristal limpia, color ámbar con capacidad de 1 litro.

6.1.3 Identificará la botella con una etiqueta que contenga la siguiente información:

a. Fecha / Hora
b. Nombre (ej.: Desperdicios acuosos, Desperdicio Orgánico, etc.)
c. Origen (ej.: V-450, etc.)
d. Nombre personal colectando la muestra
e. Número de muestra

6.1.4 Coordinar el recogido de la muestra con su supervisor.

6.1.5 Encender la bomba del tanque a ser muestreado y dejarlo rccircular por 2 horas.

6.1.6 Abrir la válvula del punto de muestreo hasta remover la acumulación de sólidos utilizando un envase para recogerlo.

6.1.7 Tomar una muestra para verificar que no exista más de una fase.

6.1.8 Si existen fases, detener el mezclado y transferir la fase acuosa a otro tanque, de lo contrario tomar la muestra.

6.1.9 De existir más de una fase y haber transferido la fase acuosa, deberá repetir los pasos 6.1.5, 6.1.6 y 6.1.7 para luego tomar la muestra.

6.1.10 Entregar la muestra al técnico del laboratorio y completar la forma de "Chain of Custody Record" utilizando un bolígrafo con tinta azul.

6.2 Laboratorio Ambiental Externo

6.2.1 Recibir la muestra y completar la cadena de custodia (Ver anejo).

6.2.2 Preservar la muestra como se indica a continuación:

Parameters	Preservative	Holding Time
Gross Heat of Combustion	Cool 4 °C	99 days
Viscocity	Cool 4 °C	7 days
pH	Cool 4 °C	48 hours
Mercury - Total	HNO3 pH<2, Cool 4°C	28 days
Beryllium - Total	HNO3 pH<2, Cool 4°C	180 days
Cadmium - Total	HNO3 pH<2, Cool 4°C	180 days
Chromium - Total	HNO3 pH<2, Cool 4°C	180 days
Lead - Total	HNO3 pH<2, Cool 4°C	180 days
Arsenic - Total	HNO3 pH<2, Cool 4°C	180 days
Ash	Cool 4 °C	7 days
Chlorine - Total	Cool 4 °C	28 days
Specific Gravity @ 25 °C	Cool 4 °C	14 days
Nonvolatile Matter	Cool 4 °C	28 days

6.2.3 Analizar la muestra como se indica a continuación:

Parameters	Method
Gross Heat of Combustion	ASTM D 240
Viscocity	ASTM 445
pH	EPA 9040C
Mercury - Total	EPA 7470 A
Beryllium - Total	EPA 6010C
Cadmium - Total	EPA 6010C
Chromium - Total	EPA 6010C
Lead - Total	EPA 6010C
Arsenic - Total	EPA 6010C
Ash	ASTM D 482
Chlorine - Total	ASTM D 808
Specific Gravity @ 25 °C	ASTM D 4052
Nonvolatile Matter	ASTM D 1353

6.3 Control de Calidad "QA/QC"

TAPI ha seleccionado dos (2) laboratorios externo para el análisis de las muestras. Estos son Altol Chemical Environmental Laboratory "ALCHEM" y Environmental Quality Laboratories "EQLab" para el análisis de los desperdicios a ser incinerados. Ambos han demostrado que poseen la capacidad y experiencia en el manejo y análisis de desperdicios peligrosos según las guías establecidas por la agencia de protección ambiental (USEPA, 1994a).

Ambos laboratorios poseen un programa comprensivo de control de calidad "QA/QC". Estos programas estan en conformidad con los requisitos aplicables (Test Methods For Evaluating Solid Waste: Physical, Chemical Methods, (SW-846). Los programas de control de calidad poseen elementos cualitativos tales como protocolos para cadena de custodia, métodos para duplicados, blancos, "Spikes" y/ o duplicados de "Spikes".

El laboratorio emplea un sistema para la revisión de los resultados analíticos para identificar cualquier desviación o problema relacionado con su programa de QA/QC. Las cadenas de custodia son verificadas para asegurar que están correctas y en acuerdo con la información contenida en la etiqueta de la muestra, así como el método de preservación, la integridad de la muestra, la utilización de botellas apropiadas entre otros. La continuidad y el seguimiento de la muestra con la cadena de custodia es verificado para asegurar que las muestras son analizadas dentro del tiempo de retención "Holding Time" requerido.

Basado en un análisis cuantitativo, los análisis de las muestras para control de calidad son revisados para asegurar la precisión y exactitud de los resultados analíticos. Los laboratorios seleccionados han demostrado ser competentes en el uso de los métodos analíticos establecidos por EPA, ASTM u otros métodos analíticos equivalentes.

6.3.1 El análisis de las muestras debe ser realizado por uno o ambos de los laboratorios seleccionados.

ANEJO I

CHAIN OF CUSTODY RECORD

"Cadena de Custodia"

(EE-039f2)

ANEJO II

Ejemplo

Cadena de Custodia

7. HISTORIAL DE CAMBIOS

Fecha	*Revisión*	*Descripción*
10/14/98	00	Nuevo
01/26/01	01	Se eliminaron los pasos 6.2.3 y 6.2.8. Se modificaron los pasos 6.2.7, 6.3.1 y 6.3.2. Se le añadió Número de libreta de referencia a los Anejos III y IV.
01/31/03	02	Se revisó debido a su fecha de expiración.
01/24/08	03	Se modificó el formato y se cambió logo y nombre de la compañía. No hubo cambios en el contenido.
06/06/11	04	No hubo cambios en el contenido.
03/26/12	05	Se añadieron reglas de seguridad, pasos para describir el muestreo, tablas para identificar los métodos analíticos para cada parámetro a analizarse y una descripción para demostrar cumplimiento con el control de calidad QA/QC.

Attachment 1

Chain of Custody used by

EQ Labs

ENVIRONMENTAL QUALITY LABORATORIES, INC.

SAMPLE DELIVERY SLIP & CHAIN OF CUSTODY

PO BOX 11458, SAN JUAN, PR 00910-1458 • TEL. (787) 288-6420, FAX (787) 288-6465, e-mail: info@eqlab.com

CLIENT NAME: CLIENT ID: W.O. #: SITE: CLIENT REP:

P.O. #: PWSID #: FOLDER #: PROJECT: EQLAB REP:

SAMPLE INFORMATION		CONTAINER INFORMATION	FIELD TESTING		ANALYSIS REQUESTED
SAMPLE #: MATRIX: SOURCE:	DATE: TIME: TYPE:	TYPE COLOR VOLUME PRESERVATIVE			
SAMPLE #: MATRIX: SOURCE:	DATE: TIME: TYPE:	TYPE COLOR VOLUME PRESERVATIVE			
SAMPLE #: MATRIX: SOURCE:	DATE: TIME: TYPE:	TYPE COLOR VOLUME PRESERVATIVE			
SAMPLE #: MATRIX: SOURCE:	DATE: TIME: TYPE:	TYPE COLOR VOLUME PRESERVATIVE			

CUSTODY RECORD	SIGNATURE	DATE	TIME
Collected in field by:			
Fixed in field by:			
Authorized by:			
Received by EQLF:			
Released to EQLL by:			
Received by EQLL:			

SPECIAL INSTRUCTIONS / COMMENTS:

*EQLF = Eqlab's Field Personnel.
*EQLL = Eqlab's Log-in Personnel.

Arrival Temperature: _____ Signature: _____

Eqlab's general terms and conditions on reverse side of this document.

Attachment 2

Chain of Custody used by

Alchem Laboratory

ALCHEM LABORATORY
ALTOL CHEMICAL ENVIRONMENTAL LABORATORY, INC.

SABANETAS INDUSTRIAL PARK - PONCE, PR
P.O. BOX 359 • MERCEDITA, PR 00715
TELS. (787) 848-6050 • (787) 848-6085 • (787) 848-6265
FAX (787) 848-6299

IAR

CUSTODY #P 37795

Customer Name:	Customer Signature:
Project:	Cont. liters _____ gal. _____

Lab. Sample No.	Date	Time	Sample Description

COPIA NO CONTROLADA
para Referencia Solamente

- ___ API Gravity
- ___ Specific Gravity
- ___ Water by Distillation
- ___ Carbon residue, Conradson
- ___ Pour Point
- ___ Purity by GC
- ___ Permanganate Time
- ___ Sulfur Content
- ___ Viscosity
- ___ Flash Point
- ___ Non Volatile Matter
- ___ Heating Value
- ___ Carbon, Ramsbottom
- ___ Copper Corrosion
- ___ Asphaltenes
- ___ Oxidation Stability
- ___ Water Content, KF
- ___ Doctor Test
- ___ Freezing Point
- ___ Water Miscibility
- ___ Distillation
- ___ Refractive Index
- ___ Existent Gum
- ___ Acid Wash Color
- ___ Octane No.
- ___ Color $K_2CR_2O_7$
- ___ RVP
- ___ Aromatics
- ___ MSEP Reading
- ___ Water and Sediment
- ___ Cloud Point
- ___ Ash
- ___ Nitrogen
- ___ Color Saybolt
- ___ Color Pt-Co
- ___ Olefins
- ___ Cetane Index
- ___ Cetane Number
- ___ Suspended Sediment
- ___ Appearance
- ___ Spot test
- ___ TOH-TOX
- ___ Iron
- ___ Sodium
- ___ Magnesium
- ___ Lead
- ___ Calcium
- ___ Vanadium
- ___ Potassium
- ___ Acidity
- ___ Alkalinity
- ___ Others _____

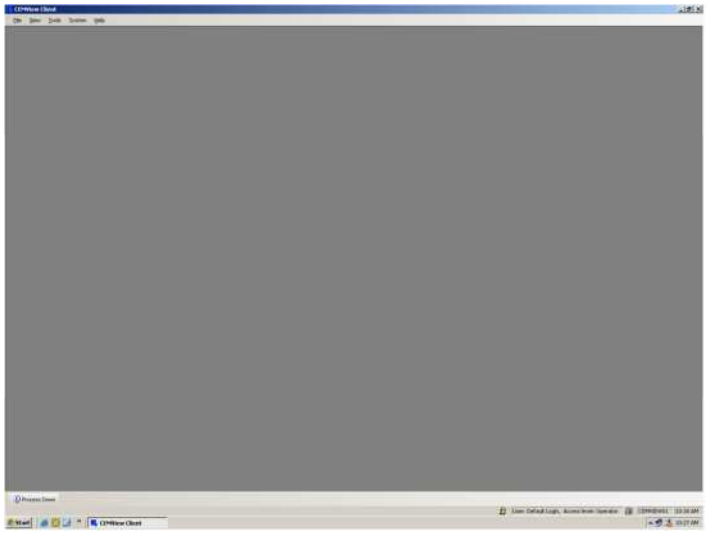
Comments: _____

Sample collected by: (Signature)	Relinquished by:	Date: Time:	Received by:	Date: Time:	Relinquished by: Date: Time:
Received by:	Date: Time:	Delivery To Lab. by:	Date: Time:	Received at Lab. by:	Date: Time:

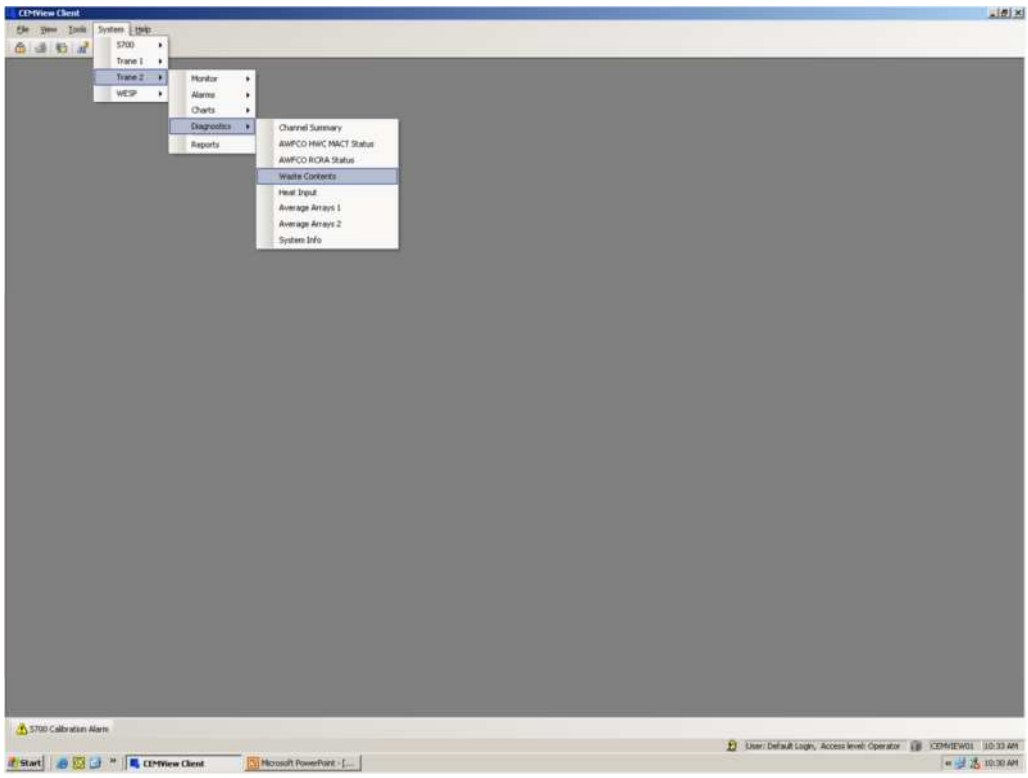
Appendix B Data Entry Procedure

Procedimiento para entrar datos de análisis de desperdicios y kerosene al sistema de CEMView

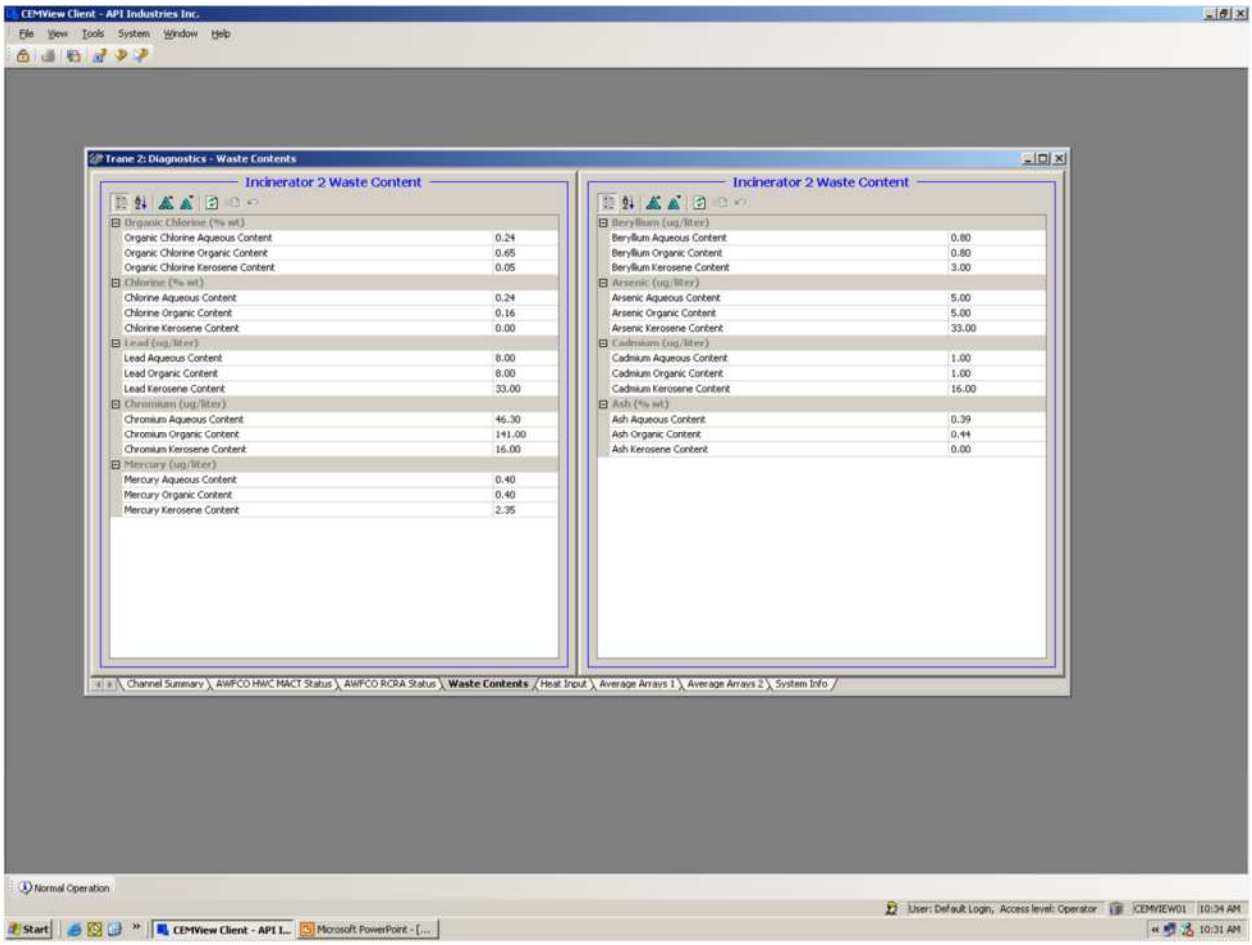
1) Se actualizará el banco de datos de CEMView antes de comenzar la incineración de lotes de desperdicios. Se entraran los resultados de análisis del desperdicio orgánico o acuoso según aplique, y si se tienen resultados nuevos del combustible, se actualizarán los resultados correspondientes.



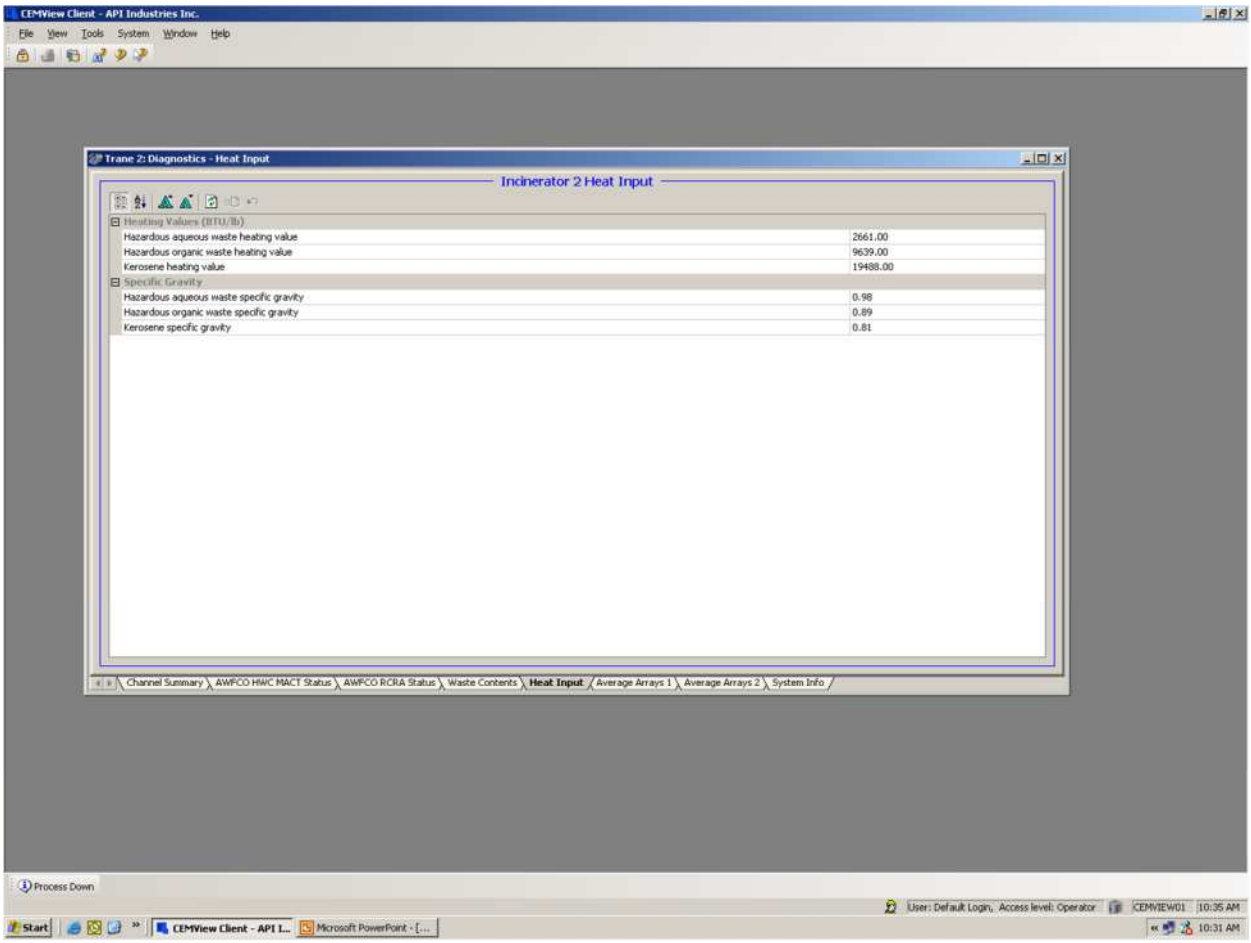
2) Seleccione el incinerador que se usará, luego escoja “Diagnostics”, y luego “Waste Contents”.



3) Entre los datos para el desperdicio orgánico y acuoso (según aplique) de Cloruros Orgánicos (“Organic Chlorine”) en % por peso, Cloruro (“Chlorine”) en % por peso, Plomo (“Lead”) en µg/L, Cromo (“Chromium”) en µg/L, Mercurio (“Mercury”) en µg/L, Berilio (”Beryllium”) en µg/L, Arsénico (“Arsenic”), Cadmio (“Cadmium”) en µg/L, y contenido de Ceniza (“Ash Contents”)) en % por peso. Escoja luego la hoja “Heat Input”.



4) Entre los resultados de análisis de laboratorio para Valor Calórico (“Heating Value”), Gravedad Especifica (“SpGr”) para los desperdicios orgánico y el acuoso (según aplique) , y, si hay un resultado reciente, los valores del keroseno


CEMView Client - API Industries Inc.
File View Tools System Window Help
Trane 2: Diagnostics - Heat Input
Incinerator 2 Heat Input
Heating Values (BTU/lb)
Hazardous aqueous waste heating value 2661.00
Hazardous organic waste heating value 9639.00
Kerosene heating value 19488.00
Specific Gravity
Hazardous aqueous waste specific gravity 0.98
Hazardous organic waste specific gravity 0.89
Kerosene specific gravity 0.81
Channel Summary
AWFCO HWC MACT Status
AWFCO RCRA Status
Waste Contents
Heat Input
Average Arrays 1
Average Arrays 2
System Info
Process Down
User: Default Login, Access level: Operator
CEMVIEW01
10:35 AM
Start
CEMView Client - API I...
Microsoft PowerPoint - [...
10:31 AM

Appendix C Calculation of Kerosene, Organic and Aqueous Waste Feed Rates

1.0 Operational Parameters Limitations

TAPI has set operational limits for metals, ash, chlorine /chlorides as presented in Table A below. In order to make sure these limits are not exceeded, on a per incineration batch basis, the contribution of the particular parameter by the kerosene, aqueous and organic waste must be added up before allowing the start of an incineration batch. In the following sections, the calculation procedure for metals, hydrogen chloride/chlorides, and particulate matter (ash) will be presented.

Table A Feed Stream Rate Limits for Individual and Group Parameters

Individual or Group Parameters	Individual Parameters	Feed Rate Limit (Lbs/Hr)
Mercury	Mercury	356 E-06
Low Volatile Metals	Lead	3,970 E-06
	Cadmium	
Semi Volatile Metals	Beryllium	5,210 E-06
	Arsenic	
	Chromium	
Hydrogen Chloride and Chlorine	Hydrogen Chloride and Chlorine	241 Lbs/hr
Particulate Matter	Particulate Matter	226 Lbs/hr

2.0 Metals Feed Stream Content Calculation

In order to calculate the Feed Stream contents of regulated metals, the metal concentration in kerosene, aqueous waste, and organic waste must be determined by chemical analyses using approved methods such as those contained in Table 4 (page 13) of this Feed Stream analysis plan. The actual amount of a particular metal being added is calculated by multiplying the flow rate of a particular stream (Q_{Stream}) by its own metal concentration ($[Metal]_{Stream}$). This is:

$Q_{Stream} \times [Metal]_{Stream}$

If the flowrate units are in liters per minute (L/min) and the metal concentrations are in µg/L, then the actual metal feed rate in pounds (Lbs) per minute can be calculated as follows (let's say we are calculating the Mercury feed rate:

Mercury (Hg) Feed Rate (Lbs/Hr)

$= (Q_{Kerosene} \times [Hg]_{Kerosene} + Q_{Aqueous\ Waste} \times [Hg]_{Aqueous\ waste} + Q_{Organic\ waste} \times [Hg]_{Organic\ Waste}) \times 1/10^9 \times 2.2046$ Lbs/Kg x 60 min/hr

$= (Q_{Kerosene} \times [Hg]_{Kerosene} + Q_{Aqueous\ Waste} \times [Hg]_{Aqueous\ waste} + Q_{Organic\ waste} \times [Hg]_{Organic\ Waste}) \times 1/10^9 \times 2.2046$ Lbs/Kg x 60 min/hr

$= (Q_{Kerosene} \times [Hg]_{Kerosene} + Q_{Aqueous\ Waste} \times [Hg]_{Aqueous\ waste} + Q_{Organic\ waste} \times [Hg]_{Organic\ Waste}) \times 1/10^9 \times 132.276$ Lbs/Kg x min/hr

Let's say that the Mercury concentration is 5 µg/L in the aqueous waste, 2 µg/L in the organic waste, 1 µg/L in kerosene and that the kerosene flow rate is 2 L/min, the aqueous waste flow rate waste is 12.5 Lpm, and the organic flow rate value is 4.5 Lpm, then the Mercury feed rate is:

$= (Q_{Kerosene} \times [Hg]_{Kerosene} + Q_{Aqueous\ Waste} \times [Hg]_{Aqueous\ waste} + Q_{Organic\ waste} \times [Hg]_{Organic\ Waste}) \times 1/10^9 \times 132.276$ Lbs/Kg x min/hr

$= (2\ L/min \times [1]_{Kerosene} + 12.5 \times [5]_{Aqueous\ waste} + 4.5 \times [2]_{Organic\ Waste}) \times 1/10^9 \times 132.276$ Lbs/Kg x min/hr

$= (2 + 62.5 + 9) \times 1/10^9 \times 132.276$ Lbs /hr $= 9.722286 \times 10^{-5}$ Lbs/Hr

Mercury Feed Rate = .00009722 Lbs/Hr

We need now to check the calculated feed rate value against the established limit of 356×10^{-6}

The calculated waste feed value is 97.22×10^{-6} which is a lower than the established limit.

The above calculation must be performed for all the other metals and we must make sure we do not exceed the maximum federate limit for the individual metals nor we exceed the group federate limits.

In the following tables we present how the different calculations can be presented showing that we do not exceed feed rate limits.

Let's start with the chemical analysis results for an organic waste, an aqueous waste, and kerosene:

Calculation of Mass Feed Rates (Lbs/Hr)

Parameters	Aqueous Waste	Organic Waste	Kerosene	Mass Feed Rate (Lbs/Hr)
Flow Rate (Lpm)	12.5	2.7	2	NA
Mercury (µg/L)	1.3	1.16	2.5	3.22513E-06
Lead (µg/L)	4	278	570.9	0.000256931
Cadmium (µg/L)	4.6	40	10	2.4537E-05
Arsenic (µg/L)	2	20	10	1.30952E-05
Beryllium (µg/L)	4	40	2.0	2.14286E-05
Chromium (µg/L)	116	11,020	26.2	0.004134444
Ash (mg/L)	107,624	15,900	0.010	0.18362831
Total Chlorides/Chlorine (mg/L	54800	2300	0.01	0.091429897
Viscosity (Centipoise)	2.27	1	1.77	NA

We now must compare the individual feed rates for metals, ash, and chlorides against those allowed.

Determination of Allowed Feed Rate Values for Organic Waste, Aqueous Waste, and Kerosene

Feed Stream	Flow Rate (Lpm)	Maximum Allowed Values (Lpm)	Alarms
Aqueous Waste	12.5	12.5	**OK !**
Organic Waste	2.7	4.5	**OK !**
Kerosene	2	3	**OK !**

Compliance Assurance

Batch Parameters comply with all Operational Limits

Parameter or Group	Individual Parameters	Target Individual Mass Rate Value (Lbs/Hr)	Actual Individual Mass Rate Value (Lbs/Hr)	Alarms (Individual Rates)	Target Group Mass Rate Values* (Lbs/Hr)	Actual Group Mass Rate Values (Lbs/Hr)	Alarms
Mercury	Mercury	0.000356	3.22513E-06	**OK !**	0.000356	3.22513E-06	**OK !**
Low Volatility Metals (LVM)	Lead	0.003481	0.000256931	**OK !**	0.0035297	0.000281468	**OK !**
	Cadmium	0.0000487	2.4537E-05	**OK !**			
Semi Volatile Metals (SVM)	Arsenic	0.000891	1.30952E-05	**OK !**	0.005208	0.004168968	**OK !**
	Beryllium	0.000139	2.14286E-05	**OK !**			
	Chromium	0.004178	0.004134444	**OK !**			
Ash (Particulate Matter)	Ash	226	0.18362831	**OK !**	226	0.18362831	**OK !**
Chlorides/Chlorine	Chlorides/Chlorine	241	0.091429897	**OK !**	241	241	**OK !**

* Regulatory Limitations

Other Parameters

Parameter or Group	Target Value	Actual Value	Alarms
Viscosity (Centipoise)	3	2.01	**OK !**
Dioxins and Furans	NA	Maintain Aqueous, Organic, and Kerosene Flow Rate values within limits	
Destruction and Removal Efficiency %	99.99	Maintain Aqueous, Organic, and Kerosene Flow Rate values within limits	

When the flow rates of Organic Waste, Aqueous Waste, and Kerosene yield compliance conditions, an incineration batch is ready for burning.

The current installed version of CEMView allows the burning of waste based on Ash Feed Rate, Total Chlorine/Chlorides Feed Rate, and Heating Values. As the incineration process continues, if there is the possibility for a twelve hours metal Feed Rate to be exceeded, and alarm will go off to alert the operator that the feed rate for a particular parameter must be decreased. The operator may decide to lower the flow rate of the Organic Waste, Aqueous Waste, and/or Kerosene based on the concentration of the particular parameter in these streams. Should the condition persist, the Automatic Waste Feed Cut Off (AWFCO) will be activated thus avoiding an exceedance of a Feed Rate Limitation.