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Chew

March 19, 1991

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Mr. C. H. Fancy, P.E.
Chief
Bureau of Air Regulation
FDER-Tallahassee
Twin Towers Office Building
2600 Blair Stone Road
Tallahassee, Florida 32399-2400

Subject: Request to Burn Hazardous Waste
Florida Mining & Materials -
Cement Kiln No. 2 PSD-FL-124A/AC27-138850
(C/TA # F03.178)

Dear Mr. Fancy:

This correspondence is being submitted as an Addendum to the subject permit application. The subject permit application provides information on the burning of Solid Hazardous Waste Fuels (SHWF) when they are fed to the cement kiln at a location approximately 73 feet downstream from the raw materials feed location. This submission provides information on the feeding of SHWF at the feed shelf (the raw materials feed location).

The FM&M Kiln No. 2 provides a thermal environment that is conducive to the destruction of organic compounds. Gas temperatures within the kiln currently range from a low of 1,900°F at the kiln inlet (Station 230, as shown in Figure 1) to approximately 3,500°F in the burning zone of the kiln (Station 57, as shown in Figure 1).

SHWF will be introduced on the feed shelf of the kiln (at the kiln inlet). The initial gas temperature at this point will be approximately 1,900°F. The SHWF will be contained in 6-gallon DOT approved containers. The containers will remain essentially intact for a time period of between one (1) and four (4) minutes - allowing the container to travel between 15 and 30 feet further into the kiln (with the raw materials). The integrity of the container will then be breached and the volatilization of the SHWF will commence. The immediate effect (of the initial SHWF combustion) is to increase the gas temperature at the kiln inlet (the point of SHWF introduction) by 100 to 250°F. Combustion of the SHWF then progresses at a rate dependent upon its specific characteristics.

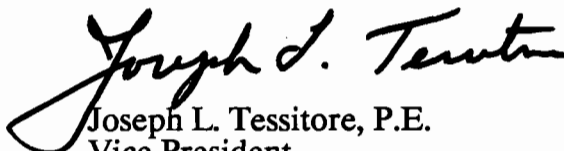
An inspection of the attached Figure 2 (which is an expanded version of Figure 1) shows that at a minimum, the SHWF is always exposed to temperatures in excess of 1,800°F. In the case where the SHWF is introduced at the feed shelf (Station 230 in Figure 2), the solids retention time in the kiln is of the order of 26 minutes. However, the gas retention time of the volatilized organic compounds is a function of where the compounds are introduced to the hot gas stream.

In a worst case scenario, it can be assumed that the integrity of the SHWF containers is breached immediately upon entry into the kiln and the volatilized organic compounds enter the gas stream at Station 230 or Location 1. In this case, Figure 2 shows that these compounds will be exposed to temperatures of 1,800°F or greater for approximately 4 seconds (11.2 seconds at Location 4, minus 7.2 seconds at Location 1). This same figure shows that for the above worst case, the volatilized organic compounds are exposed to a temperature of 2,000°F for more than 2 seconds. In actuality, Southdown experiences have shown that the SHWF containers will survive from 1 to 4 minutes, thereby increasing the exposure time by an additional second.

The above data shows that the feeding of SHWF at the raw material feed shelf provides more than an adequate thermal environment for the destruction of organic compounds. Naturally, this will be verified during the performance testing of Kiln 2, while feeding SHWF at the raw material feed shelf.

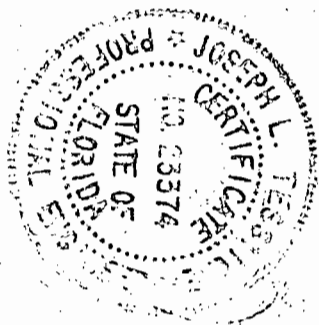
If you have any questions and/or require any additional data, please do not hesitate to call upon me.

Sincerely,


Joseph L. Tessitore, P.E.
Vice President

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GAS RETENTION TIME AND TEMPERATURE BROOKSVILLE KILN SYSTEM

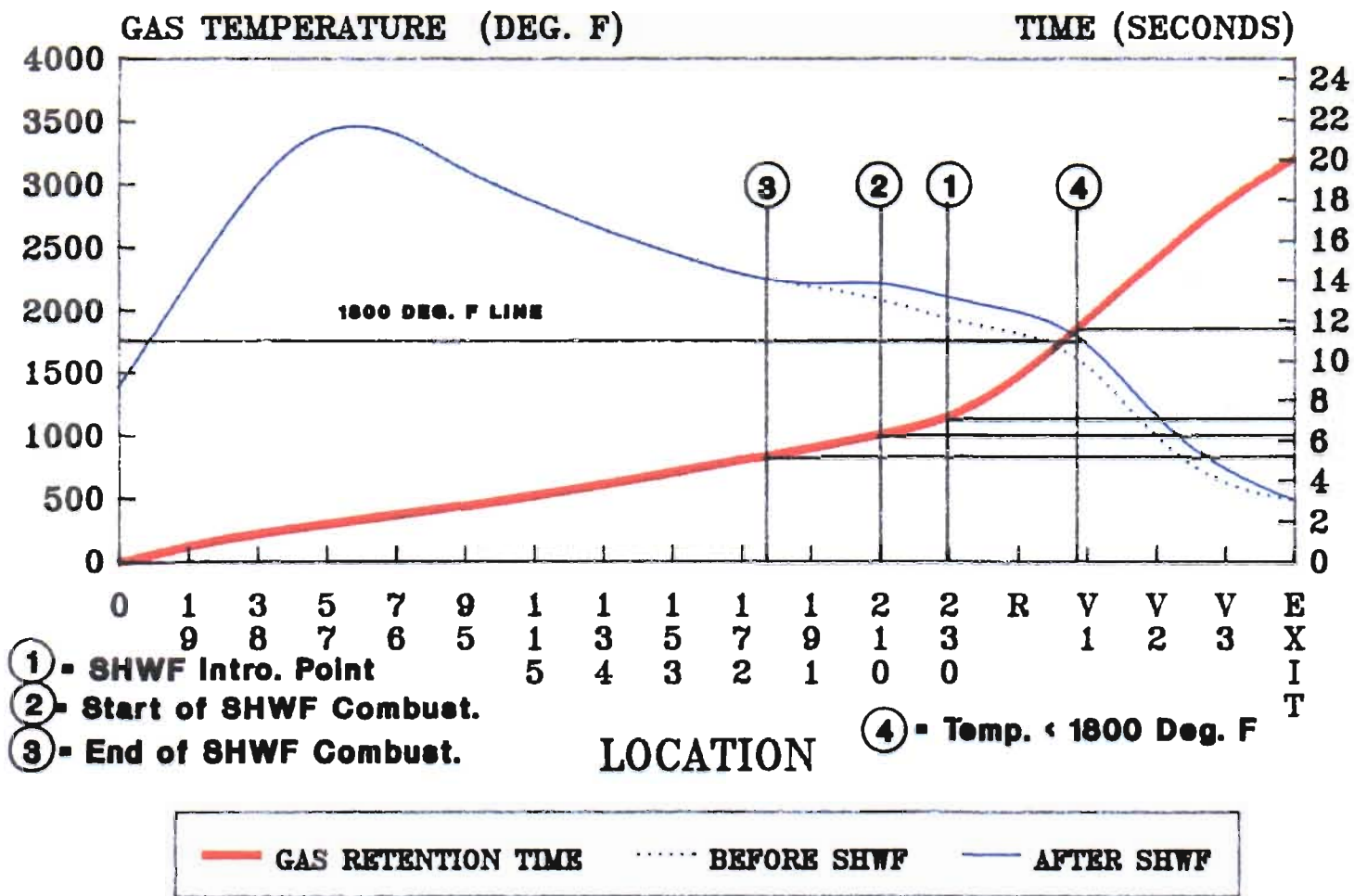


FIGURE 1

GAS RETENTION TIME AND TEMPERATURE BROOKSVILLE KILN SYSTEM (EXPANDED VIEW)

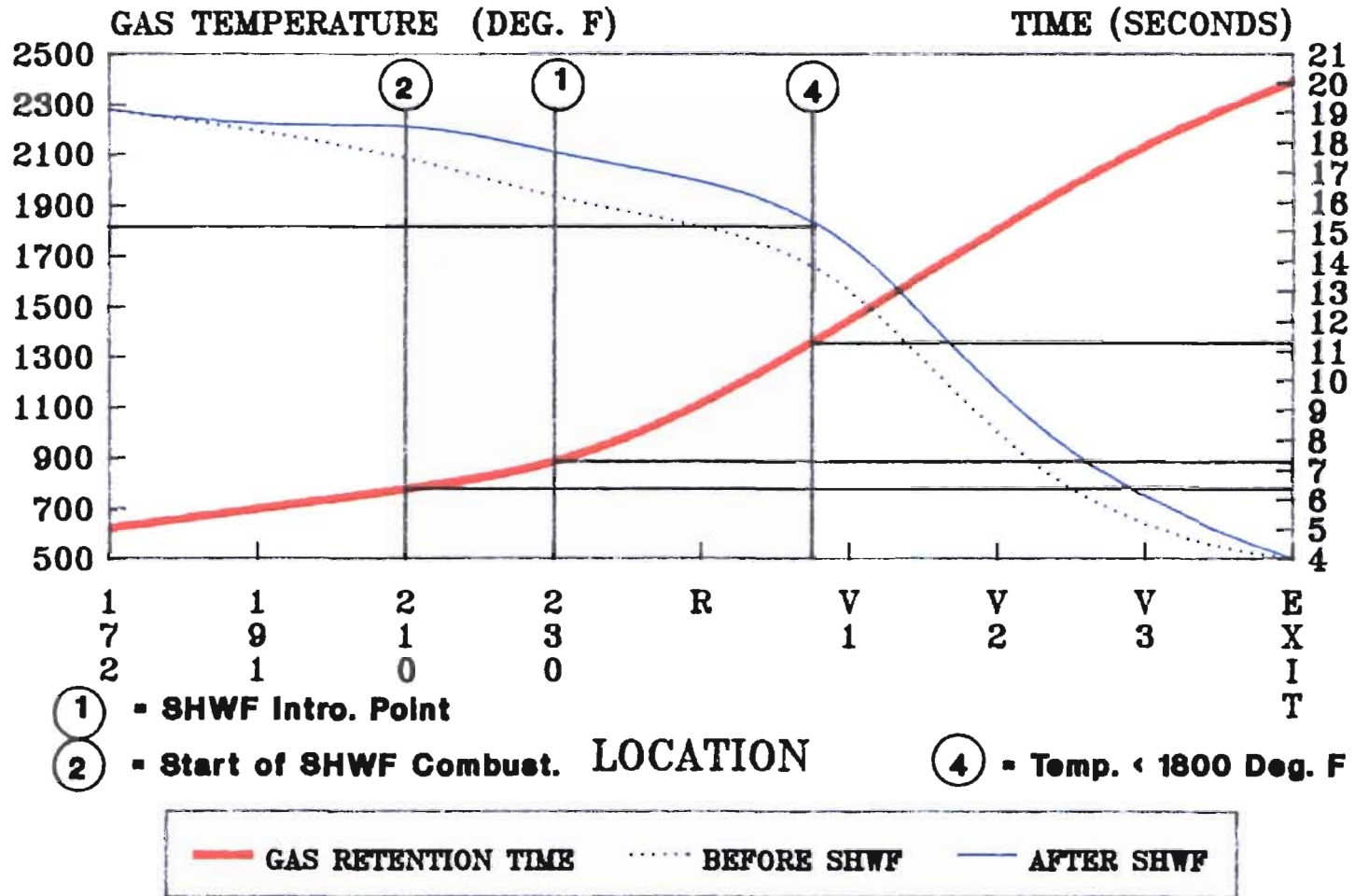


FIGURE 2

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**Performance Test Plan
Florida Mining & Materials**

Brooksville, Florida

February 22, 1991

**Review Draft for the
Department of Environmental Regulation,
State of Florida**

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I. Project Description

1.1 Introduction

Florida Mining & Materials (Brooksville) presently operates a cement clinker manufacturing facility in Hernando County, Florida. Florida Mining and Materials will conduct performance testing to demonstrate compliance with regulatory limitations and to measure emissions of other pollutants. It should be noted that this program will provide detailed level of compliance testing as required by Boiler and Industrial Furnace Regulations (BIF Rules) promulgated by U.S. EPA on February 21, 1991.

Energy and Environmental Research Corporation of Irvine, California has been contracted to provide technical support in terms of Compliance Test Plan development, project management and oversight of sampling and analytical activities.

Entropy Environmental, Inc. of Research Triangle Part, N.C. has been contracted to perform the stack gas sampling.

Triangle Laboratories, Inc. of Research Triangle Park, N.C. and General Engineering Laboratories, Inc. of Charleston, S.C. are providing analytical support.

1.2 Proposed Test Date

The Compliance Test at Brooksville is planned for the week of June 24, 1991. An earlier date is dependent on acceptance of this test plan by DER and review of this test plan by a committee of citizens appointed by the County Commissioner of Hernando County, Florida (Committee). The testing schedule will be coordinated with DER.

1.3 Performance Test Scope

The Performance Test program has been designed to demonstrate the capabilities of the cement kiln to utilize solid hazardous waste fuel (SHWF) as an alternate fuel source, and to destroy difficult-to-incinerate materials while meeting the emission limitations specified in the newly promulgated BIF regulations.

The capability of the unit will be demonstrated by testing stack emissions while feeding SHWF, POHC's and metals. This will require the use of POHC's that are very stable. The compounds have been selected from a listing that ranks compounds by their thermal stability¹. The testing will also demonstrate the facility's capability to handle elevated

¹The University of Dayton Research Institute Thermal Stability Index.

metals at chlorine levels compatible with the cement process without exceeding limitations set forth in the BIF regulations. Florida Mining and Materials will be evaluating the metals emission data to determine appropriate metals mass input limitations.

Under a separate authorization Florida Mining and Materials will demonstrate the facility's capability to handle used oil and tire derived fuel (UO/TDF). Three Performance Test conditions have been selected based on BIF requirements. These conditions are noted below and include as fuels:

- Condition 1: Coal/SHWF
- Condition 2: Coal/TDF-UO
- Condition 3: Coal

More detailed discussion of the test procedures can be found in Section III.

1.4 Performance Test Objectives

The purpose of this test is to demonstrate that SHWF and, separately UO/TDF will be used as fuel for the production of cement clinker and that resulting stack emissions will meet appropriate regulatory limitations.

1.5 Proposed Operating Permit Conditions

This section suggests key operating conditions which Hernando County may elect at some time to incorporate into Florida Mining and Materials' operating permit. The conditions discussed below are based on the BIF rules and the knowledge of facility requirements for producing a quality cement product. It is not necessary under the BIF rules to comply for these limits while under Interim status precompliance.

1.5.1 Continuous Emission Monitor (CEM)

- A continuous emissions monitor (CEM) device measuring CO (carbon monoxide) and THC (total hydrocarbons) shall be installed downstream from the dust collector. The CEM shall be maintained in an operational condition whenever SHWF is being introduced into the kiln system as replacement fuel. The kiln system shall be operated according to best operating practice to ensure efficient combustion and removal of the organic compounds as specified by Tier II 40 CFR266.104, by maintaining:
- Hourly rolling average of THC below 20ppm (measured as propane) and corrected to 7% O₂, dry gas basis

- Hourly rolling average of CO shall not exceed the established CO standard and corrected to 7% O₂). This CO standard is based on data acquired during the Performance Test and is the average over all valid runs of the highest hourly average CO level for each run.

If any of the above stipulated emissions are exceeded, the flow of SHWF to the kiln shall be stopped immediately. This SHWF cutoff shall be automated by respective electronic and control systems interlocks. (This will not be in place for the Performance Test.)

1.5.2 Metals Input

The mass input rate of each metal listed below shall not exceed the specified allowable feed rate in the hazardous waste fuel.

		<u>Allowable Feed Rate</u> (lb./hr.)
Antimony	=	
Arsenic		
Barium		
Beryllium	=	
Cadmium	=	To be determined in accordance with BIF regulations, using data generated by the Performance Test.
Chromium	=	
Lead		
Mercury		
Silver		
Thallium	=	

Florida Mining and Materials shall maintain adequate records of hazardous waste fuel analyses and feed rates to demonstrate that the allowable metals feed rates are not exceeded.

The allowable feed rate for each metal shall be calculated based on the results of emissions testing and dispersion modeling to provide a limitation which will ensure that the reference air concentrations (RAC and RSD) in the BIF rules are not exceeded.

Due to the high temperature and the extremely large amounts of feed stock in the kiln system, the behavior of metals is technically independent of the location of entry and the form of entry. Incorporation of metals into clinker and dust (and metals emissions as particulates) only depend upon the total metals input into the kiln system.²

² S. Sprung: Technological Problems in Pyroprocessing Cement Clinker: Cause and Solutions, Benton-Verlag, Dusseldorf, Germany, 1985; and, Michael von Seebach and J. B. Tompkins: The Behavior Of Metals In Cement Kilns, Paper presented at Rock Product's 26th International Cement Seminar, New Orleans, Louisiana, 1990

Consequently, the mass input rate of each metal is the determining factor for the emissions.

1.5.3 Particulates Limits

The kiln system shall be operated according to best operating practice to ensure efficient removal of particulate matter by maintaining;

Particulate matter emission are not in excess of 180 milligrams per dry standard cubic meter (0.08 grain per dry standard cubis foot) after correction to a stack gas concentration of 7% O₂.

1.5.4 Total Chlorine Input

Total chlorine and chloride feed rate shall not exceed the specified allowable feed rates in the total feed streams. Like the metals, the allowable feed rate limits are to be determined in accordance with the BIF rules, using data generating by the test. These limits are based on an hourly rolling average emission.

II. DESCRIPTION OF THE FACILITY

2.1 Name and Location of Facility

The facility is known as Florida Mining and Materials and is located off U.S. Highway 98, Brooksville, Hernando County Florida. This plan covers Performance Testing activities on kiln #2.

2.2 Major Equipment Installed

2.2.1 Cement Processing Operation

The cement plant consists of two (2) dry process rotary cement kiln systems, two (2) raw mills, three (3) cement finish milling systems and numerous storage and blending silo facilities. The raw materials used to manufacture Portland cement include limestone, sand, flyash. These materials are accurately metered to two (2) pre-mixing bins prior to introduction to the raw mills. The raw mills dry and grind the raw materials to a high fineness - suitable for feed stock for the rotary kiln systems. The raw mill product is subsequently transported to raw meal blending silos where it is further mixed to assure physical and chemical consistency in the kiln feed stocks.

The raw meal is then metered to the rotary kiln systems. In the rotary kilns, the raw meal is heated to temperatures exceeding 2600 degrees F - producing Portland cement clinker. The clinker is subsequently transported to the finish milling department for final processing into Portland cement. The finished product is stored in large silos for shipment by truck or rail in bulk, or bags.

Coal is the primary fuel for the kiln systems. The coal utilized typically has a heating value of 12,500 BTU / LB (as received) and a sulfur content of 0.83 LB / million BTU (in accordance with air permit requirements). Approximately 98,400 tons of coal are consumed annually, for kiln 2.

2.2.2 Major Pyroprocessing Equipment Installed

Finely ground raw material (raw meal) is conveyed via a pneumatic conveying system to the top of the preheater.

The preheater is a 24' diameter by 268' tall modified Gepol design featuring four (4) cyclones, a vortex vessel and two (2) venturi section. Hot kiln gases enters the base of the preheater at approximately 1,900 °F and exits the top cyclone stages at approximately 800 °F. Raw meal enters the top of the preheater at a temperature of approximately 150 °F and exits the preheater onto the kiln feed shelf at a temperature of approximately 1550 °F.

The rotary kiln is a 14'5" diameter unit having a length of 230'. It has a 15'9" expanded diameter section at the feed inlet area. The kiln is driven by a 400 HP adjustable speed drive system. The kiln is fired primarily by coal finely pulverized in an air swept ball mill.

The clinker discharges from the kiln system into a five (5) compartment hydraulically driven grate type clinker cooler. The cooling air, used to reduce the clinker temperature from 2600 °F at the discharge of the kiln to a temperature enabling transport of the clinker via conventional material handling equipment, is supplied by five (5) undergrate fans.

Excess air from the cooler (not required for combustion in the kiln) is vented through an Air to Air heat exchanger before passing through a Nomex bag type dust collector.

FUEL TYPES

Kiln fuel is primarily coal from Eastern Kentucky. It has a typical heat value of approximately 12,500 BTU per pound and has very low sulfur. The kiln system typically requires approximately 3.2 million BTU per ton of clinker. Flolite will mainly be used during start-up of kiln operations and during periods when raw materials feed is stopped and kiln temperature must be maintained, and flolite is normally used only as a substitute for coal.

ELECTRIC POWER

Electric power is supplied by Florida Power Corp. via two supply lines - a 69 kv line delivering through a 7.5 MVA transformer and a 115 KV line delivering power through a 12 MVA transformer.

DUST COLLECTION SYSTEMS

Gases exiting the number 2 kiln and raw mill systems is cleaned in an 18 module bag house designed by the Fuller Company. The gasses from the number 1 kiln and raw mill system is cleaned in a similar design Western Precipitation 16 module bag house.

CENTRAL CONTROL SYSTEM

Operation of the entire plant is achieved from a central location. Process controls are supplied by Honeywell and alarm systems by Ronan. Kiln system specific instrumentation features kiln shell radiation scanners and closed circuit TV monitors for observation of the kiln burning zone.

CHEMICAL CONTROL FACILITIES

A complete on-site laboratory exists for the analysis of raw materials, clinker and cement. This facility includes an ARL 5400 X-Ray System (backed up by an EDAX 9500 System),

complete wet chemistry laboratory, atomic absorption flame photometry instruments, and a complete cement physical testing laboratory.

2.3 Operating Conditions Anticipated During Testing

A flow schematic of the kiln system is contained in Appendix B.

During the course of the testing, the pyroprocessing system will be operated in a manner consistent with good operating practice. By virtue of the rigorous requirements in the manufacture of Portland cement clinker temperatures in the burning zone, kiln inlet and preheater tower are required within the following ranges:

- Burning Zone Material Temperature 2500°F to 2700°F (requires flame temperatures in excess of 3500°F)
- Kiln Inlet Material Temperature 1500°F to 1600°F
- Kiln Inlet Gas Temperature 1900°F to 2000°F
- Preheater Exit Gas Temperature 750°F to 850°F

The burning zone temperature indicated above is required to achieve the chemical reaction necessary to form Portland cement clinker. Temperatures lower than these will result in incomplete formation of the clinker and will be evidenced by excessive free lime in the clinker. This clinker would not be suitable for use in the manufacture of Portland cement and would not meet ASTM standards. Temperatures higher than those indicated above will result in formation of a molten load in the kiln - resulting in damage to the kiln refractory and potential damage to the clinker cooler. While there is no reliable method of directly measuring the burning zone temperature, it is impossible to produce clinker at temperatures below the specified ranges.

The kiln inlet material temperature defined above is necessary to properly prepare and partly decarbonize the feed prior to introduction to the rotary kiln. Temperature levels lower than those indicated will necessitate a reduction of kiln feed rate to insure proper clinker formation. Higher temperatures may result in material encrustation in the preheater tower.

The kiln feed rates and resultant clinker production levels anticipated during the course of testing are as follows:

Kiln Feed	130 tons per hour
Clinker Production	79.6 tons per hour

Fuel Rates (dependant upon test condition):

Coal	Approximately 9.5 tons/hr and approx. 10.4 tons/hr during test condition 3 (baseline)
SHWF	Approximately 50 lbs/min *
TDF	Approximately 50 lbs/min
UO	Approximately 35 lbs/min

*See next page.

III. Performance Test Procedures

3.1 Introduction

This program consists of three conditions of three runs each. During each run, the main kiln stack will be monitored using sampling trains and continuous emission monitors (CEM). This data will be basic information such that the effect of certain parameters (i.e. THC) can be isolated from the total stack gas flow.

3.2 Metals

To a great extent, metals are incorporated into the crystalline structure of the clinker. Depending upon the volatility of the specific metal, a portion evaporates in the hot zones of the kiln. From here, the vapors move with the combustion gases to the preheater, where they come in contact with cooler material. Upon contact with cooler raw material, the vapors condense and react with the feed stock. The raw material will then return a portion of the metals to the hotter zones of the kiln, thus, building up internal circulations. Furthermore, portions of the metals are returned with the dust to the baghouse and from there to the raw feed stock on the kiln feed, thus building an external circulation. Both the internal and the external circulations may be influenced by the amount of halogens, primarily chlorine, present in the system. It is, therefore, important to keep the chlorine level in the system during the tests at a level compatible with the kiln system (maximum compatible Chlorine level).

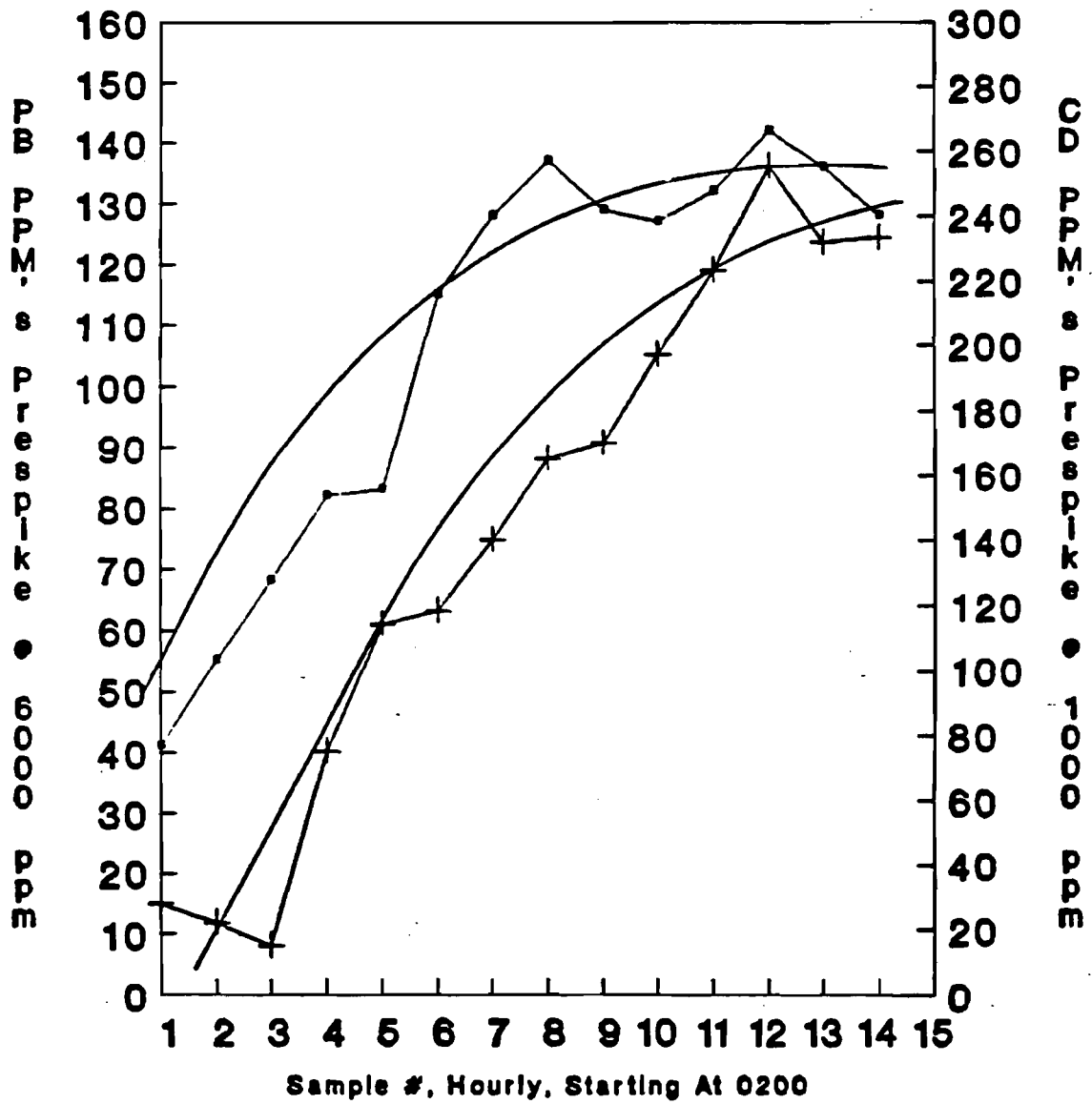
Test Condition 1 is designed to keep metals and chlorine input from SHWF at a given, predetermined level during the test conditions. The metals will simulate maximum specified metals input from the SHWF.

Sampling and analysis of emissions from the main stack will determine emission rates of all 10 EPA regulated metals. The sampling will utilize the EPA multi-metals train as specified in the final BIF regulations. This train is operated as a Method 5 particulate train with modification to the impinger configuration to enhance the collection of the above metals as discussed in the QAPP. Sampling for chromium (VI) will be conducted using the EPA Draft Method from the BIF regulations. The results from this train and the multi metals train will provide information on chromium (VI) versus total chromium.

During testing, the concentration of arsenic, lead and cadmium will be maintained at a level representative of fuel quality. For more details on metals spiking, refer to Sections 3.61 and 3.64. Whenever metals feed rates are significantly changed sufficient time will be allowed to stabilize the internal circulation in the system. This is estimated at 12-14 hours as shown on Figure entitled "XRF Analysis of Baghouse Dust" which represents the results of X-ray diffraction analysis of kiln dust during this period of prespike on another kiln.

The ambient impact of metals emissions will be determined using dispersion modeling to relate stack emission concentration to site-specific ambient air concentrations. Dispersion modeling procedures will be as specified in "Guideline on Air Quality Models" EPA-450/2-78-027R. These site-specific ambient air concentrations for each metal will be compared to the acceptable ambient air concentrations; risk specific does (RSD's) for carcinogenic metals and reference air concentrations (RAC's) for noncarcinogenic metals as stipulated in the BIF regulations. RSD and RAC values are based on the premise that the additional risk to the MEI from exposure to pollutant over a 70 year lifetime shall not exceed 10^{-5} . The MEI is a hypothetical person assumed to reside at the point of maximum, off-site, ground level impact.

XRF ANALYSIS OF BAGHOUSE DUST TAKEN 1/09/91



—●— PB —+— CD

3.3 Fuel Preparation

3.3.1 Solid Hazardous Waste Fuel (SHWF)

The SHWF will be manifested to Brooksville in six (6) gallon DOT approved containers. The containers are stacked 36 to a pallet and wrapped in heat shrink plastic. The palletized load arrives at the plant in an enclosed van where the pallets are removed and sampled. Upon laboratory verification, the pallets are placed one at a time into an elevator and taken to the fourth stage kiln floor. The individual containers are placed on a roller conveyor and gravity feed to a timed interjection device where they enter the kiln at the feed shelf through a double air lock. The timed device is set consistent with the level of thermal load permissible process manufacturing constraints. At this stage of planning this is estimated at 15% total fuel substitution.

3.3.2 Used Oil/Tire Derived Fuel

Used oil (UO) will be delivered to the site by tanker truck and stored and fed from the existing on-site fuel oil system. Used oil will be utilized at a substitution rate of 35%.

Tire Derived Fuel (TDF) will be shipped to the site in enclosed trailer vans. Tires will be unloaded from the transport van onto an elevator which will convey the tires to the preheater floor for introduction to the kiln through a timed introduction mechanism with double air locks similar to that used for SHWF. TDF is planned for a 15% substitution rate.

3.4 Detailed Test Protocol

3.4.1 Program Scope

The performance test program consists of two conditions, each with three replicate runs. The fuel rates in terms of heat release and estimated mass rate for the three test conditions are summarized in Tables 3.1 and 3.2. Brief descriptions of each test follow this section.

It is recommended procedure in a conventional hazardous waste incinerator compliance test burn to conduct tests at the "worst case" conditions. These usually include maximum feed rates, maximum gas flow rates and minimum temperatures. Cement production does not allow significant process fluctuations since product quality demands stable operation. Therefore, extreme diversion from normal operation cannot occur. It should be noted that temperatures unacceptable for cement production (less than 2,500°F material temperature) are still well above that of normal operation for a hazardous waste incinerator (1,800°F - 2,100°F). The stringent operating requirements along with the huge thermal mass and inertia inherent in a cement kiln provide ideal conditions for the use of waste fuels as replacement for fossil fuels.

Table 3.1
Summary of Fuel Substitution
Percent of System Fuel Requirement

	SHWF	TDF	UO	Coal
Condition 1	9	0	0	91
Condition 2	0	15	35	50
Condition 3	0	0	0	100

Table 3.2
Summary of Estimated Fuel Feed Rates
Pounds Per Hour

	SHWF	TDF	UO	Coal
Condition 1	2820	0	0	18,860
Condition 2	0	2770	2070	10,400
Condition 3	0	0	0	20,750

Condition 1

This condition will consist of coal and SHWF. Lead, cadmium, and arsenic and will be spiked by attaching pre-weighed packages to the SHWF cans prior to introduction to the kiln.

Condition 2

This condition will consist of coal and TDF and UO. No metals will be spiked during this test condition. All stack data will be gathered as in the previous condition.

Condition 3

Condition 3 is a baseline on coal only. No metals will be spiked during this condition. All stack data will be gathered as in this previous conditions.

3.4.2 Sampling Procedures

This section provides a summary of the sampling and analytical procedures that will be followed during the program. Throughout the program, approved sampling protocol will be utilized. Any deviations from the specified protocol will be fully documented in the final report. Table 3.3 presents the methods for each test condition. A more detailed listing of the sampling and analysis can be found in the QAPP.

Fuel samples (SHWF, TDF/UO and coal) will be collected during the respective runs. These samples will be analyzed to establish total metal inputs into the system. The sample points are summarized in Tables 3.4 and 3.5. The batches of SHWF will be sampled and analyzed by Florida Mining and Materials at the fuel processor. Therefore, sampling during the test runs is not necessary. A more detailed description of sampling may be found in the QAPP.

Process samples will be collected and composited for each condition. These samples will be analyzed for the purposes of confirming the existence or non-existence of certain compounds in process flow increment (i.e. metals, etc.). Tables 3.4, 3.5 and Figure 3.1 provides a summary of the process sampling and analytical tests, but a more detailed description of sampling may be found in the QAPP.

3.4.3 Metals Addition, Pretest Equilibrium and SHWF Test Condition

3.4.3.1 Pretest Equilibrium

While form and location of metals input are of minimum influence, the method by which metals will be input to the kiln system are designed to simulate as closely as possible the

metals introduction via SHWF. The exact method of introduction is however, influenced by the availability of specific compounds as well as the specifics of the kiln system.

During the **pretest equilibrium period** (approximately 12 hours) metals will be introduced to the burning zone of the rotary kiln. The nature of the specific compound will determine its specific method of introduction. Arsenic will be supplied as Arsenic Trioxide. This is a powder and will be metered by a weigh feeder. The weigh feeder will discharge into the kiln coal feed belt.

Lead and cadmium will be input during this "equilibrium phase" to the kiln burning zone via a liquid solution. Metering pumps will be employed to discharge 55 gallon drums of the metal compound. The flow rate from the metering pumps will be controlled to achieve the specific mass flow rate as required to simulate the specified metal content. The flow rate will be measured by a loss in weight method (Figure 3.2). The specific compounds used will be a solution of Lead 2-Hexolate in mineral spirits with cadmium nitrate as an aqueous solution.

3.4.3.2 SHWF Test Condition

Metals will be introduced with the SHWF to simulate specified metals content in those fuels. The specific compounds to be utilized are;

Lead Oxide
Cadmium Nitrate

Arsenic Trioxide

The metals salts will be delivered to the Brooksville plant in pre-weighed packages. The powders will be contained in plastic bags. The packages will be affixed to the cans containing SHWF prior to introduction to the kiln system. The bags will be wrapped in alluminum foil and duct taped to the can lids.

**TABLE 3.3
STACK GAS SAMPLING
SUMMARY OF PERFORMANCE TEST
METHODS**

Analysis	Sampling Method	Cond. 1	Cond. 2	Cond. 3
Main Stack				
Particulate	M5	+	+	+
Volatile Organics - Benzene	EPA SW-846-0030(VOST)		+	+
PCDD/PCDF and PAH	EPA Draft Method 23		+	+
TCB ⁺	EPA Draft Method 23	+		
SF-6 ⁺	Modified Method 18	+		
Metals	EPA Draft Multi Metals Train	+	+	+
Hydrogen Chloride	EPA SW846-0050 [*]	+	+	+
Sulfur Dioxide	M6C	+	+	+
Oxygen	M3A	+	+	+
Carbon Dioxide	M3A	+	+	+
Carbon Monoxide	M10	+	+	+
Oxides of Nitrogen	M7E	+	+	+
Visible Emissions	M9	+	+	+
Total Hydrocarbons	M25A	+	+	+
Chromium (VI)	EPA Draft Method	+		+

* Entropy will use a 0.3 micron filter which is allowed in the method and will quantify all cationic interferences as defined in this method. Appendix D contains correspondence from USEPA on these issues.

+ TCB is 1,2,4 Trichlorobenzene which is the DE-POHC. Sulfur hexafluoride as a POHC is directly measured by GC on Stack Gas.

**TABLE 3.4
SUMMARY OF
PROCESS SAMPLING**

Sample Location	Description	Sampling Frequency	Testing Frequency
1	Raw Mill Feed	Hourly	Comp/Cond
2	Raw Mill Product	Hourly	Comp/Cond
3	Kiln Feed	Hourly	Comp/Cond
4	Coal Feed To Mill	30 Minutes	Comp/Cond
5	Coal To Kiln	30 Minutes	Comp/Cond
6	Clinker	Hourly	Comp/Cond
7	Kiln Bag House Catch	Hourly	Comp/Cond
8	SHWF (TDF/UO)	Presample	Comp/Cond
9	Limestone/Wet Flyash	Hourly	Comp/Cond
10	Sand	Hourly	Comp/Cond
11	Flyash	Hourly	Comp/Cond
12	Staurolite (if used)	Hourly	Comp/Cond
13	Mill Scale (if used)	Hourly	Comp/Cond

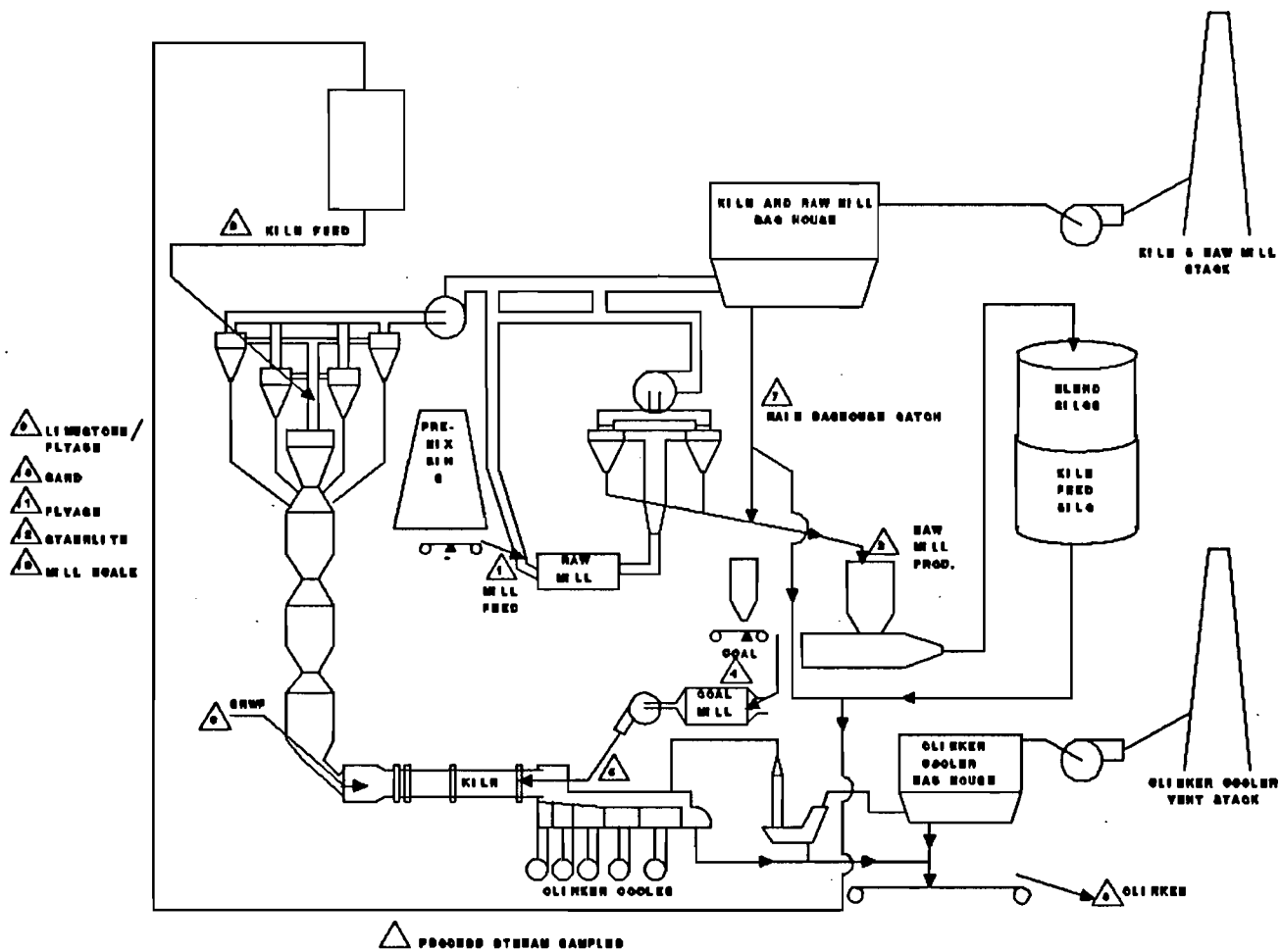
**TABLE 3.5
SUMMARY OF
PROCESS ANALYTICAL³**

Sample Location	Description	Chloride & Sulfur	Metals	** Phys/Chem Characteristics
1	Raw Mill Feed	+	+	
2	Raw Mill Feed	+	+	
3	Kiln Feed	+	+	
4	Coal Feed To Mill	+	+	+
5	Coal Feed To Kiln	+	+	+
6	Clinker	+	+	
7	Kiln Baghouse Catch Waste Fuel	+	+	
8	SHWF (TDF/UO)	+	+	+
9	Limestone/Wet Flyash	+	+	
10	Sand	+	+	
11	Fly Ash	+	+	
12	Staurolite (if used)	+	+	
13	Millscale (if used)	+	+	

** Physical and Chemical Characteristics (Caloric Value, Viscosity, Specific Gravity, pH and Ash)

³ Not all of these analysis are necessary for emission purposes, some are for process operational issues.

FIGURE 3.1 PROCESS SAMPLING LOCATIONS



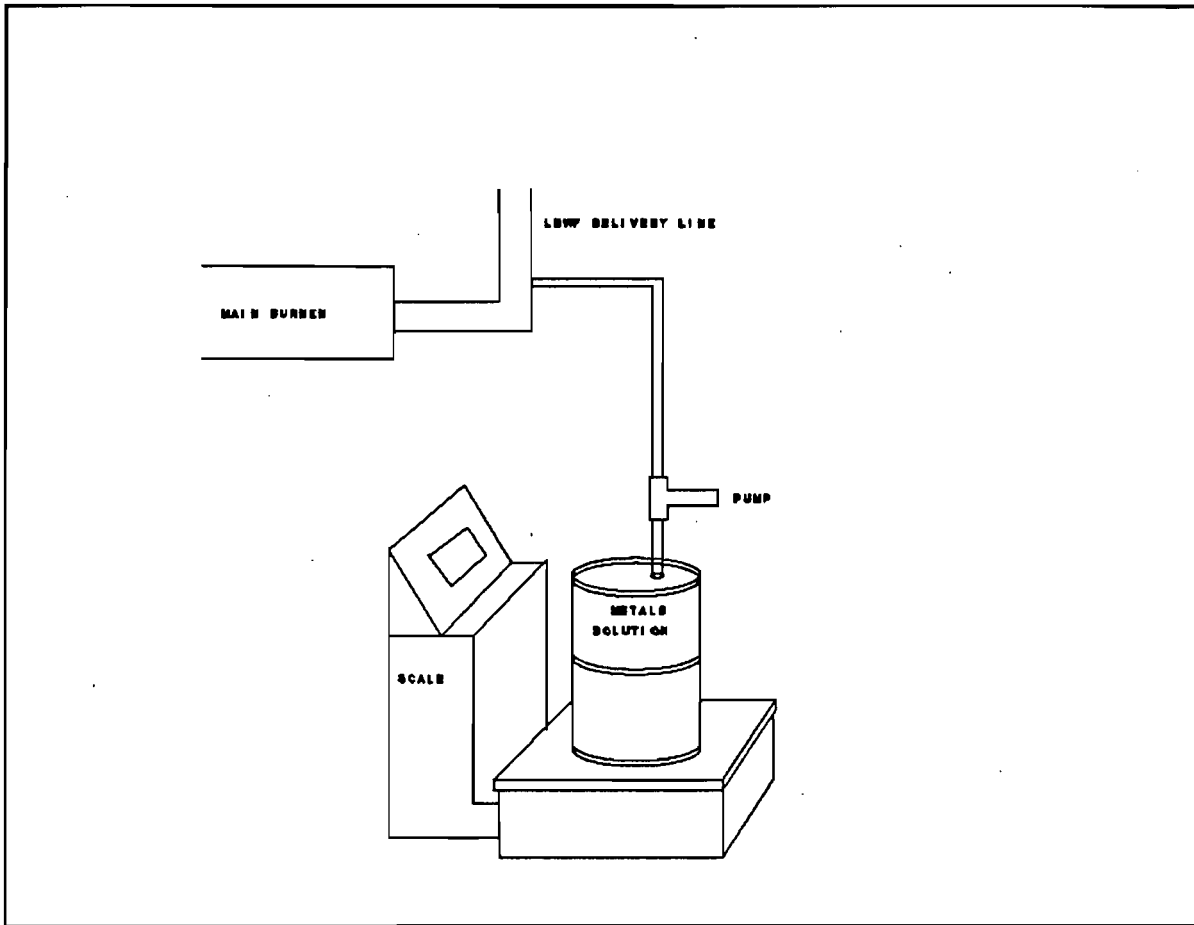


Figure 3.2 Metals solutions Injection (Equilibrium Phase)

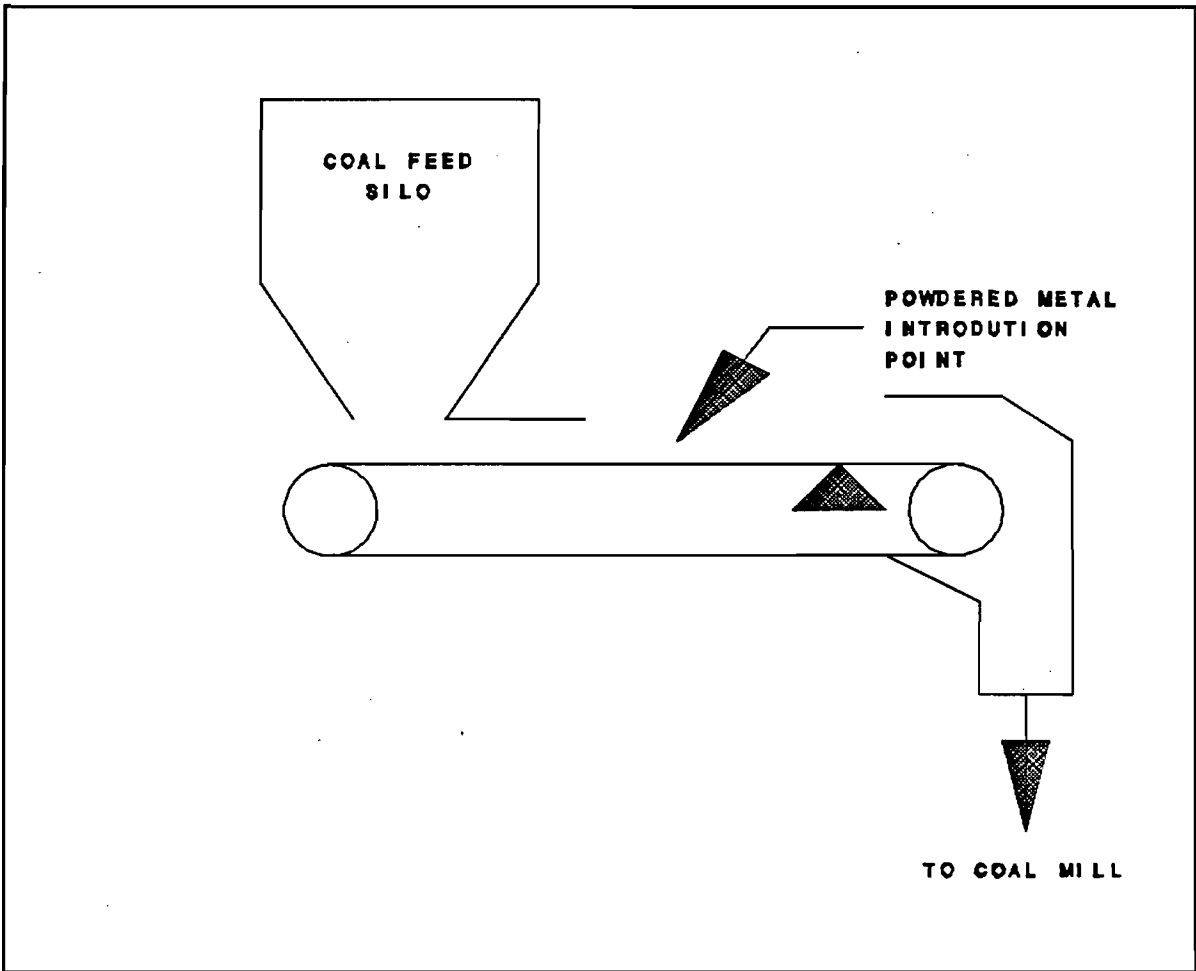


FIGURE 3.3 Powdered Metals Injection (Equilibrium Phase)

Appendix A

Quality Assurance Project Plan

**Performance Test Plan
Florida Mining & Materials
Brooksville, Florida**

February 22, 1991

**Review Draft for the
Department of Environmental Regulation
State of Florida**

QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM

Testing Initiation Date: _____

Task Title: Performance Test at Florida Mining and Materials

Approval Signatures:

_____ Contractor Project Director	_____ Signature	_____ Date
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_____ Contractor Project Manager	_____ Signature	_____ Date
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_____ Contractor QA Coordinator	_____ Signature	_____ Date
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_____ Florida Mining & Materials, Project Manager	_____ Signature	_____ Date
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_____ Florida Mining & Materials, QA Officer	_____ Signature	_____ Date
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Sampling Procedures	6	0.0	February 22, 1991
Sample Custody	7	0.0	February 22, 1991
Calibration Procedures and Frequency	8	0.0	February 22, 1991
Analytical Procedures	9	0.0	February 22, 1991
Data Reduction, Validation and Reporting	10	0.0	February 22, 1991
Internal Quality Control Checks	11	0.0	February 22, 1991
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Preventative Maintenance	13	0.0	February 22, 1991
Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness	14	0.0	February 22, 1991
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PROJECT DESCRIPTION

3.1 Introduction

Florida Mining and Materials (FM&M) presently operates a cement clinker manufacturing facility in Brooksville, Florida. FM&M will conduct performance testing to demonstrate emissions for Solid Hazardous Waste Fuel, Tire Derived Fuel and Used Oil.

Triangle Laboratories, Inc. of Research Triangle Park, N.C. and General Engineering Laboratories, Inc. of Charleston, S.C. are providing analytical support.

3.2 Performance Test Scope

The Performance Test program has been designed to demonstrate the capabilities of the cement clinker process equipment to utilize various regulated waste fuels as an alternate fuel source, by destroying difficult-to-incinerate materials and meeting the emission limitations.

The designated POHCs for the performance test on SHWF are sulfur hexafluoride and, 1,2,4-trichlorobenzene. Each test condition will consist of three VOST runs for POHCs, and benzene, three Method 23 runs for dioxins/furans and polynuclear aromatic hydrocarbons, three runs for Cr6 using the BIF method, three EPA Method 5 and EPA Draft Method 26 runs for particulate and hydrogen chloride, three EPA Draft Multi-Metals Method runs for metals, three EPA Method 9 runs for opacity, and continuous emission monitoring for oxygen, carbon dioxide, carbon monoxide, nitrogen oxide, sulfur dioxide, and total hydrocarbons performed at the main stack. THC will be monitored in the bypass gas stream simultaneously. Dioxin/furan measurements will also be made during all conditions. Process sampling performed during the trial burn will include rawmill feed, rawmill product, kiln feed, preheater discharge, kiln baghouse catch, clinker dust, clinker, solid waste fuel coal to mill, coal to kiln. Solid hazardous waste fuel will be sampled as the containers are packaged, prior to the test. Stack gas sample collection will be performed by Entropy Environmental, Inc. Process sample collection will be performed by Florida Mining & Materials personnel. General Engineering Laboratories, Inc. and Triangle Laboratories, Inc. have been subcontracted to perform process and stack gas sample analyses. Tables 6-1 to 6-3 list the sampling and analytical methods for each test condition.

3.3 Performance Test Objectives

The purpose of this test is to demonstrate that hazardous organic substances are adequately destroyed during the production of cement and that the resulting stack emissions will meet the appropriate regulatory limitations. The tests described in this plan are intended to provide sufficient data to support the operator of the facility the ability to feed hazardous waste fuels, as identified in the facility's RCRA Part B permit.

3.4 Test Schedule

The performance test is tentatively scheduled for the week of June 24, 1991. As outlined below eight days are necessary to complete this test program.

- Day 1 - Setup test equipment
- Day 2 - Conduct testing for Condition I
- Day 3 - Standby
- Day 4 - Conduct testing for Condition II
- Day 5 - Standby
- Day 6 - Standby
- Day 7 - Standby
- Day 8 - Conduct Testing for Condition III

QUALITY ASSURANCE PROGRAM ORGANIZATION AND RESPONSIBILITY

Entropy assumes responsibility for all measurements made in connection with this project, and therefore responsibility for the implementation of an adequate quality assurance (QA) program. This section discusses Entropy's approach to management of the QA aspects of the subject test program. Provisions and procedures which are incorporated in the overall management structure to promote implementation of QA procedures and adherence to QA guidelines are described.

The management structure and organization employed by Entropy facilitates the development and performance of quality assurance/quality control (QA/QC) functions by accurately defining the QA/QC direct lines of communication and authority between different levels of project management and the QA management structure. Entropy's QA program is designed in a way that facilitates interaction between QA program personnel and the project team. QA program personnel interface independently with project team members at all levels, monitoring data representativeness, accuracy, precision, and completeness. QA program personnel are free to interact directly with project team members at any time QA considerations in one of these areas need to be addressed.

The organization of the Entropy project team, including QA functions, is shown in the project organization chart (see Figure 4-1). Note that the QA structure is independent of the organizational groups who will generate environmental measurement data during this project.

The focus of the Entropy QA program is the quality assurance manager. He is responsible for the day-to-day oversight of QA activities. The quality assurance manager reports to the quality assurance coordinator. The quality assurance coordinator occupies a level higher than the project manager, and reports directly to the project director.

4.1 Project Director

The Project Director (PD) oversees the Project Managers' test program planning, organization, and performance. He also assists in technical supervision and oversees protocol development and review, data interpretation, and report preparation. The PD for this sampling project will be Tony Wong. Mr. Wong serves as the PD for all testing projects

4.2 Project Manager

[To be specified.]

4.3 Quality Assurance/Quality Control Officer

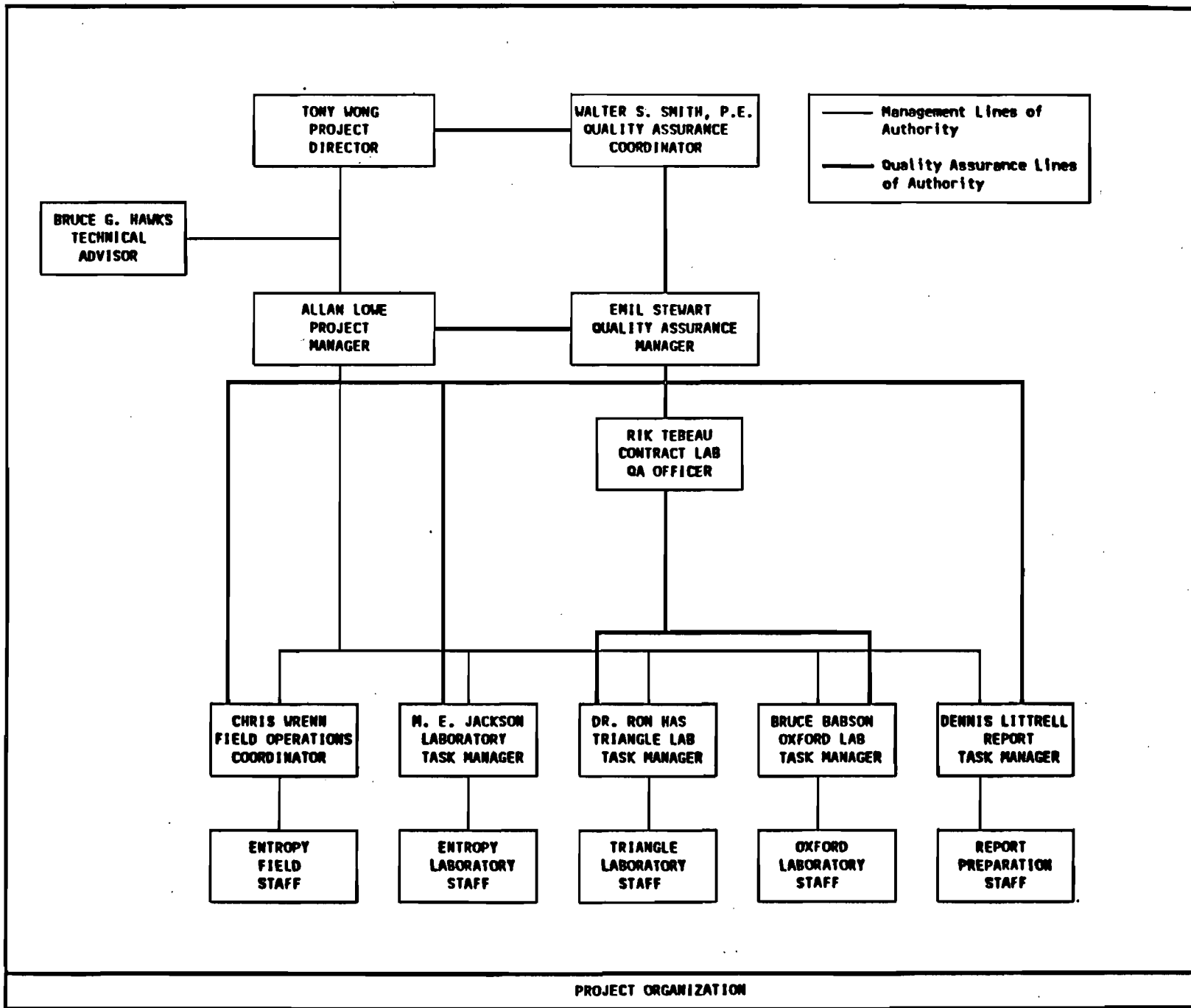
[To be specified.]

4.4 Report Preparation

[To be specified.]

4.5 Technical Personnel

Resumes of personnel who will be involved in this project will be included in Appendix C at a later date.



QUALITY ASSURANCE OBJECTIVES

The overall measurement objective is to determine the effectiveness of the system in achieving thermal destruction of the hazardous constituents in the test burn wastes. Numerical QA objectives for accuracy and precision of the sampling system calibration, sample preparation, and analysis procedures can be established based on previous experience in applying comparable procedures to a variety of complex sample matrices.

The QA objectives for analytical accuracy and precision for this test burn sampling and analysis program are consistent with general laboratory practices. The accuracy objectives will be quantitatively determined for the volatile POHCs in the stack gas. This will be achieved by performance of the VOST audit if required by the regulatory agencies. Accuracy for the other parameters can be quantitatively determined if supplied with performance (check) samples by the regulatory agencies. The quality assurance objective for completeness is to obtain analytical results for at least 75% of the VOST samples and at least 90% of the remaining samples collected during each performance test.

The representativeness of gas samples collected by EPA Method 5 type trains will be ensured by their collection at $\pm 10\%$ isokinetic sampling conditions. VOST collection (single point sampling) will consist of four 40-minute samples collected during a 160 minute test period for each test burn. Each 40-minute sample will be collected at a constant sampling rate (approximately 1/2 liter per minute). Bag samples collected for EPA Method 3 analysis are multi-point and constant rate. Each of these samples, therefore, represents the average composition of the stack gas during each test run that the sample is collected. Process samples will be collected in accordance with the methods described in Sampling and Analysis Methods for Hazardous Waste Incineration (First Edition), a guideline document prepared for the EPA by Arthur D. Little, Inc. Each sample container will be rinsed thoroughly with waste prior to isolating the sample.

All data will be reported in terms of mass analyte per unit volume of sample matrix. Results from the analyses of all samples for a given matrix will be reported in the same units. Rates of POHCs input to the system and exhausted from the stack will be stated in common units (e.g., pounds per hour) to enable calculation of DREs. The analytical approach used to demonstrate that the data will be suitable in obtaining four nines DRE is presented in Appendix A.

A list of QA objectives for all measurements, field and laboratory, are presented in Table 5-1.

**TABLE 5-1
QUALITY ASSURANCE OBJECTIVES**

<u>SAMPLE</u>	<u>PARAMETER</u>	<u>PRECISION*</u>	<u>ACCURACY**</u>	<u>COMPLETENESS</u>
Raw Mill Feed	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
	POHCs	≤20% RPD	-	>90%
Raw Mill Product	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
	POHCs	≤20% RPD	-	>90%
Kiln Feed	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
	POHCs	≤20% RPD	-	>90%
Preheater Discharge	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
	POHCs	≤20% RPD	-	>90%
Kiln Baghouse Catch	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
	POHCs	≤20% RPD	-	>90%
Clinker Dust	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
	POHCs	≤20% RPD	-	>90%
Clinker	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
	POHCs	≤20% RPD	-	>90%

- continued next page -

**TABLE 5-1 (Cont'd)
QUALITY ASSURANCE OBJECTIVES**

<u>SAMPLE</u>	<u>PARAMETER</u>	<u>PRECISION*</u>	<u>ACCURACY**</u>	<u>COMPLETENESS</u>
Solid Waste Fuel	Caloric Value	≤12% RPD	-	>90%
	Specific Gravity	≤11% RPD	-	>90%
	pH	≤10% RPD	-	>90%
	Ash	≤15% RPD	-	>90%
	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
	POHCs	≤20% RPD	-	>90%
	Tire Derived Fuel/Used Oil			
Coal To Mill	Caloric Value	≤12% RPD	-	>90%
	Specific Gravity	≤11% RPD	-	>90%
	Ash	≤15% RPD	-	>90%
	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
Coal To Kiln	Caloric Value	≤12% RPD	-	>90%
	Specific Gravity	≤11% RPD	-	>90%
	Ash	≤15% RPD	-	>90%
	Metals	≤18-25% RPD	-	>90%
	Chlorine	≤20% RPD	-	>90%
	Sulfur	≤18-25% RPD	-	>90%
	POHCs	≤20% RPD	-	>90%
Main Stack	Particulate		-	>90%
	HCl	≤10%RPD	-	>90%
	Metals	≤20%RPD	-	>90%
	POHCs	-	±50%RE	≥75%***
	Volatile	-	-	≥75%***
	PCDD/PCDF	-	-	>90%
	Semivolatile	-	-	>90%
	Opacity	-	-	-

*
**

RPD - Relative Percent Difference

RE - Relative Error

Three out of the four samples collected for each test run will be analyzed

SAMPLING PROCEDURES

Tables 6-1 through 6-4 summarize all sampling and analytical procedures to be used during the program. Following are descriptions of the process and stack sampling methods to be performed.

6.1 Process Sampling Methods

6.1.1 Process (Raw Mill Feed, Raw Mill Product, Preheater Feed, Preheater Discharge, Baghouse Catch, Clinker Dust and Clinker)

All process samples will be taken at normal locations. Grab samples, approximately 500 mL, will be taken at the intervals specified in Table 6-1. These grab samples, for each condition, will be composited in a five gallon bucket. At the end of each condition, the composite sample will be well mixed and split into two samples. One sample will be sent to the laboratory for required analysis and the other will be stored as a backup. The backup is kept in case of breakage in shipment or handling. FM&M personnel will be responsible for the collection of samples only. Program management personnel will be responsible for the proper splitting of samples, labelling and packaging for shipment.

6.1.2. Solid Hazardous Waste Fuel

Solid waste fuel will be sampled by Florida Mining and Materials personnel at the processing facility, prior to the performance test. These samples will be composited per batch. The containers will be marked with a batch code for tracking during the test.

6.1.3. Tire Derived Fuel/Used Oil

During each run of Condition 2, three tires will be collected by FM&M personnel, one each at the beginning, middle and end. This will yield 9 tires per the condition for sampling. Each tire will have a cross section taken (using a hand saw). This will include the head. These nine samples will be frozen using liquid nitrogen and the resulting residue and metal bead will be analyzed.

Used oil will be collected by FM&M personnel in precleaned sample containers supplied by the laboratory. The sampler will be trained in proper collection technique. Grab samples will be collected hourly from sample taps on the discharge side of the feed pumps and composited on a per run basis. The composite samples will be analyzed for metals, physical parameters and POHCs, where required.

**TABLE 6-1
CONDITION I
SAMPLES AND ANALYTICAL PROCEDURES**

<u>SAMPLES TO BE TAKEN</u>	<u>SAMPLE FREQUENCY</u>	<u>SAMPLING METHOD</u>	<u>ANALYSIS PARAMETER*</u>	<u>ANALYTICAL METHOD*</u>	<u>LABORATORY***</u>
Raw Mill Feed	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Raw Mill Product	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Kiln Feed	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Limestone and Sand	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Kiln Baghouse Catch	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Clinker	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL

**TABLE 6-1 (Cont'd)
CONDITION I
SAMPLES AND ANALYTICAL PROCEDURES**

<u>SAMPLES TO BE TAKEN</u>	<u>SAMPLE FREQUENCY</u>	<u>SAMPLING METHOD</u>	<u>ANALYSIS PARAMETER*</u>	<u>ANALYTICAL METHOD*</u>	<u>LABORATORY***</u>
Mill Scale	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Staurolite	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Solid Waste Fuel	Grab	Batch	Presampled	Caloric Value ASTM D240-76	GEL
			Viscosity	ASTM D2161, 2983	GEL
			Specific Gravity	APHA 213E	GEL
			pH	EPA 150.1	GEL
			Ash	ASTM D808	GEL
			Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
			Coal to Mill	Hourly/Condition	Grab
			Viscosity	ASTM D2161, 2983	GEL
			Specific Gravity	APHA 213E	GEL
			pH	EPA 150.1	GEL
			Ash	ASTM D808	GEL
			Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL

TABLE 6-1 (Cont'd)
CONDITION I
SAMPLES AND ANALYTICAL PROCEDURES

<u>SAMPLES TO BE TAKEN</u>	<u>SAMPLE FREQUENCY</u>	<u>SAMPLING METHOD</u>	<u>ANALYSIS PARAMETER*</u>	<u>ANALYTICAL METHOD*</u>	<u>LABORATORY***</u>
Coal to Kiln	Hourly/Condition	Grab	Caloric Value	ASTM D240-76	GEL
			Viscosity	ASTM D2161, 2983	GEL
			Specific Gravity	APHA 213E	GEL
			pH	EPA 150.1	GEL
			Ash	ASTM D808	GEL
			Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Main Stack	2 hours/run	EPA M5/Draft M26	Particulate	EPA M5	Entropy
	2 hours/run 160 min./run	EPA Draft Metals SW-846-0030	HCl	Ion Chromatography	GEL
			EPA Draft Metals		GEL
	3 hours/run 1 hour/run	EPA M23 EPA M9	POHCs	SW-846-5040	TLI
			Volatile PICS	SW-846 5040	TLI
	3 hours/run 3 hours/run	EPA M3A	Semivolatile PICS	SW-846 8270	TLI
			Opacity	EPA M9	-
	3 hours/run 3 hours/run 3 hours/run 3 hours/run	EPA M3A	Oxygen	EPA M3A	Entropy
			Carbon Dioxide		
			Sulfur Dioxide	EPA M6C	Entropy
Nitrogen Oxide			EPA M7E	Entropy	
Carbon Monoxide			EPA M10	Entropy	
3 hours/run	EPA M25A	Total Hydrocarbons	EPA M25A	Entropy	

* Metals - antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, silver, and Thallium.

** SW-846 Analytical methods from "Test Methods for Evaluating Solid Wastes," September, 1986.

*** Entropy Environmental, Inc.

GEL - General Engineering Laboratories, Inc.

TLI - Triangle Laboratories, Inc.

**TABLE 6-2
CONDITION II
SAMPLES AND ANALYTICAL PROCEDURES**

<u>SAMPLES TO BE TAKEN</u>	<u>SAMPLE FREQUENCY</u>	<u>SAMPLING METHOD</u>	<u>ANALYSIS PARAMETER*</u>	<u>ANALYTICAL METHOD*</u>	<u>LABORATORY***</u>
Raw Mill Feed	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Raw Mill Product	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Kiln Feed	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Limestone and Sand	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Kiln Baghouse Catch	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Clinker	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL

**TABLE 6-2 (Cont'd)
CONDITION II
SAMPLES AND ANALYTICAL PROCEDURES**

<u>SAMPLES TO BE TAKEN</u>	<u>SAMPLE FREQUENCY</u>	<u>SAMPLING METHOD</u>	<u>ANALYSIS PARAMETER*</u>	<u>ANALYTICAL METHOD*</u>	<u>LABORATORY***</u>
Mill Scale	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Staurolite	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Solid Waste Fuel	30 min./run	Grab	Caloric Value	ASTM D240-76	GEL
			Viscosity	ASTM D2161, 2983	GEL
			Specific Gravity	APHA 213E	GEL
			pH	EPA 150.1	GEL
			Ash	ASTM D808	GEL
			Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Coal to Mill	Hourly/Condition	Grab	Caloric Value	ASTM D240-76	GEL
			Viscosity	ASTM D2161, 2983	GEL
			Specific Gravity	APHA 213E	GEL
			pH	EPA 150.1	GEL
			Ash	ASTM D808	GEL
			Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL

**TABLE 6-2 (Cont'd)
CONDITION II
SAMPLES AND ANALYTICAL PROCEDURES**

<u>SAMPLES TO BE TAKEN</u>	<u>SAMPLE FREQUENCY</u>	<u>SAMPLING METHOD</u>	<u>ANALYSIS PARAMETER*</u>	<u>ANALYTICAL METHOD*</u>	<u>LABORATORY***</u>
Coal to Kiln	Hourly/Condition	Grab	Caloric Value	ASTM D240-76	GEL
			Viscosity	ASTM D2161, 2983	GEL
			Specific Gravity	APHA 213E	GEL
			pH	EPA 150.1	GEL
			Ash	ASTM D808	GEL
			Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Main Stack	2 hours/run	EPA M5/Draft M26	Particulate	EPA M5	Entropy
			HCl	Ion Chromatography	GEL
	2 hours/run 160 min./run	EPA Draft Metals SW-846-0030	EPA Draft Metals		GEL
			POHCs	SW-846-5040	TLI
			Volatile PICS	SW-846 5040	TLI
	3 hours/run	EPA M23	PCDD/PCDF	SW-846 8290X	TLI
			Semivolatile PICS	SW-846 8270	TLI
	1 hour/run	EPA M9	Opacity	EPA M9	-
	3 hours/run	EPA M3A	Oxygen	EPA M3A	Entropy
			Carbon Dioxide		
	3 hours/run	EPA M6C	Sulfur Dioxide	EPA M6C	Entropy
	3 hours/run	M7E	Nitrogen Oxide	EPA M7E	Entropy
3 hours/run	EPA M10	Carbon Monoxide	EPA M10	Entropy	
3 hours/run	EPA M25A	Total Hydrocarbons	EPA M25A	Entropy	

* Metals - antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, silver, and Thallium.

** SW-846 Analytical methods from "Test Methods for Evaluating Solid Wastes," September, 1986.

*** Entropy Environmental, Inc.

GEL - General Engineering Laboratories, Inc.

TLI - Triangle Laboratories, Inc.

**TABLE 6-3
CONDITION III
SAMPLES AND ANALYTICAL PROCEDURES**

<u>SAMPLES TO BE TAKEN</u>	<u>SAMPLE FREQUENCY</u>	<u>SAMPLING METHOD</u>	<u>ANALYSIS PARAMETER*</u>	<u>ANALYTICAL METHOD*</u>	<u>LABORATORY***</u>
Raw Mill Feed	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Raw Mill	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Kiln Feed	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Limestone and Sand	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Kiln Baghouse Catch	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Clinker	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL

**TABLE 6-3 (Cont'd)
CONDITION III
SAMPLES AND ANALYTICAL PROCEDURES**

<u>SAMPLES TO BE TAKEN</u>	<u>SAMPLE FREQUENCY</u>	<u>SAMPLING METHOD</u>	<u>ANALYSIS PARAMETER*</u>	<u>ANALYTICAL METHOD*</u>	<u>LABORATORY***</u>
Mill Scale	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Staurolite	Hourly/Condition	Grab	Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Solid Waste Fuel	Batch	Grab Presample	Caloric Value	ASTM D240-76	GEL
			Viscosity	ASTM D2161, 2983	GEL
			Specific Gravity	APHA 213E	GEL
			pH	EPA 150.1	GEL
			Ash	ASTM D808	GEL
			Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
			Coal to Mill	Hourly/Condition	Grab
Viscosity	ASTM D2161, 2983	GEL			
Specific Gravity	APHA 213E	GEL			
pH	EPA 150.1	GEL			
Ash	ASTM D808	GEL			
Metals	SW-846 6010 or 7041-7841	GEL			
Chlorine	IC EPA 300.0	GEL			
Sulfur	SW-846 6010	GEL			
POHCs	SW-846 8240 or 8010/8020	GEL			

**TABLE 6-3 (Cont'd)
CONDITION III
SAMPLES AND ANALYTICAL PROCEDURES**

<u>SAMPLES TO BE TAKEN</u>	<u>SAMPLE FREQUENCY</u>	<u>SAMPLING METHOD</u>	<u>ANALYSIS PARAMETER*</u>	<u>ANALYTICAL METHOD*</u>	<u>LABORATORY***</u>
Coal to Kiln	Hourly/Condition	Grab	Caloric Value	ASTM D240-76	GEL
			Viscosity	ASTM D2161, 2983	GEL
			Specific Gravity	APHA 213E	GEL
			pH	EPA 150.1	GEL
			Ash	ASTM D808	GEL
			Metals	SW-846 6010 or 7041-7841	GEL
			Chlorine	IC EPA 300.0	GEL
			Sulfur	SW-846 6010	GEL
			POHCs	SW-846 8240 or 8010/8020	GEL
Main Stack	2 hours/run	EPA M5/Draft M26	Particulate	EPA M5	Entropy
			HCl	Ion Chromatography	Entropy
	2 hours/run 160 min./run	EPA Draft Metals SW-846-0030	EPA Draft Metals		GEL
			POHCs	SW-846-5040	TLI
			Volatile PICS	SW-846 5040	TLI
	3 hours/run	EPA M23	PCDD/PCDF	SW-846 8290X	TLI
			Semivolatile PICS	SW-846 8270	TLI
	1 hour/run	EPA M9	Opacity	EPA M9	-
	3 hours/run	EPA M3A	Oxygen	EPA M3A	Entropy
			Carbon Dioxide		
	3 hours/run	EPA M6C	Sulfur Dioxide	EPA M6C	Entropy
3 hours/run	M7E	Nitrogen Oxide	EPA M7E	Entropy	
3 hours/run	EPA M10	Carbon Monoxide	EPA M10	Entropy	
3 hours/run	EPA M25A	Total Hydrocarbons	EPA M25A	Entropy	

* Metals - antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, silver, and Thallium.

** SW-846 Analytical methods from "Test Methods for Evaluating Solid Wastes," September, 1986.

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6.2 Main Stack Sampling Methods

6.2.1 Sampling Point Determination - EPA Method 1

The number and location of the sampling or traverse points will be determined according to the procedures outlined in EPA Method 1.

6.2.2 Flue Gas Velocity and Volumetric Flow Rate - EPA Method 2

The flue gas velocity and volumetric flow rate will be determined according to the procedures outlined in EPA Method 2. Velocity measurements will be made using type S pitot tubes conforming to the geometric specifications outlined in EPA Method 2. Accordingly, each pilot tube is assigned a coefficient of 0.84. Differential pressures will be measured with manometers or magnehelic gauges of the appropriate range. Effluent gas temperatures will be measured with chromel-alumel thermocouples equipped with a digital temperature indicator.

6.2.3 Flue Gas Moisture Content - EPA Method 4

The flue gas moisture content will be determined in conjunction with each EPA Method 5 type train and according to the sampling and analytical procedures outlined in EPA Method 4. The impingers will be connected in series and will contain reagents as described below. The impingers will be contained in an ice bath in order to assure condensation of the moisture in the flue gas stream. Any moisture that is not condensed in the impingers is captured in the silica gel, therefore all moisture can be weighed and entered into moisture content calculations.

6.2.4 Flue Gas Composition and Molecular Weight - EPA Method 3A

EPA Method 3A will be used to determine the oxygen (O₂) and carbon dioxide (CO₂) concentrations in the flue gas stream. Concentration measurements will be made using instrumentation and test procedures that conform with the requirements of EPA Method 3A. An extractive sampling system will provide a continuous, conditioned sample (i.e., free of water vapor and particulate matter) to the analyzers. The following analyzers are used for the measurements of O₂ and CO₂.

A Teledyne Model 320P-4 will be used to determine the concentration of O₂ in the flue gas stream. This instrument utilizes Teledyne's patented micro-fuel cell, which consumes O₂ from the atmosphere surrounding the measuring probe. The consumption of O₂ generates a proportional electrical current. This current is then amplified and provides a signal output which corresponds to full scale measurement ranges of either 0 to 10% and 0 to 25% O₂.

An ACS (Fuji) Model 3300 Non-Dispersive Infrared (NDIR) Analyzer continuously monitors the CO₂ concentration in the flue gas stream. The theory of operation for this analyzer is based on the principle that CO₂ gas has a unique absorption line spectrum in the

infrared region. The instrument consists of an infrared light source, a chopper, a measuring cell, and a detector. The infrared light beam emitted by the source passes through the measuring cell filled with a continuously flowing gas sample. The light beam is partially absorbed or attenuated by the gas species of interest in this cell before reaching the front chamber of the detector. Both the front and rear chambers of the sealed detector are filled with a reference gas. The difference in the amount of light absorbed between the front and rear chambers is dependent on the concentration of the gas species of interest within the sample measuring cell and creates a pressure differential between the two chambers. This pressure difference is then observed as gas flow by the micro-flow sensor located in a channel communicating the two chambers. The resulting AC signal from the micro-flow sensor is rectified, amplified, and linearized into a DC voltage signal for output. The analyzer full scale measurement is 0 to 20% for CO₂.

6.2.5 Sulfur Dioxide - EPA Method 6C

The concentration of sulfur dioxide (SO₂) in the flue gas stream will be measured continuously using a Western Research Model 721A SO₂ analyzer. The basic analytical principle involves quantitative measurement of the absorption of UV radiation by SO₂ molecules. The analyzer uses a single light source which emits an appropriate wavelength illuminating the sample cell through which the sample is continuously passed. Narrow band optical filters are rotated through the beam, and the radiation passed is detected by a single photodetector. The signal pulses from the photodetector are separated by a demultiplexer into a measuring channel and a reference channel. A log ratio amplifier computes the logarithm of the ratio of the input signals, producing a signal that is proportional to the concentration of SO₂. The SO₂ analyzer will be operated on the 0-250 ppm SO₂ or the 0-2500 ppm SO₂ range.

6.2.6 Nitrogen Oxides - EPA Method 7E

The concentration of nitrogen oxide (NO_x) in the flue gas stream will be measured with a Beckman Model 951A NO/NO_x analyzers. The analyzer automatically and continuously determines the concentration of nitrogen oxide (NO) or oxides of nitrogen (NO_x) in a flowing gas mixture. The analytical technique is chemiluminescence. In the determination of NO_x, the sample is routed through a converter where the NO₂ within the sample is dissociated to form NO. The sample is then passed through a reaction chamber where the NO is quantitatively converted to NO₂ by gas phase oxidation with molecular ozone produced within the analyzer. In this reaction, the NO₂ molecules are elevated to an electronically excited state and immediately reverted to a non-excited ground state. This reversion is accompanied by emission of photons, which impinge on a photomultiplier detector and generate a low level DC current. The current is then amplified and used to drive a front panel meter and a data recorder.

The NO_x concentration seen by the instrument includes the contributions of both the NO in the sample and the NO resulting from the dissociation of the NO₂ in the sample.

6.2.7 Carbon Monoxide - EPA Method 10

The carbon monoxide (CO) concentrations will be determined in accordance with EPA Method. 10. The sample gas to the carbon monoxide analyzer will pass through layered Ascarite-II and Drierite tubes to remove carbon dioxide and any residual water vapor, respectively.

The Fuji Model 3300 non-dispersive infrared (NDIR) analyzers are used to continuously measure the CO₂ and CO each have a unique absorption line spectrum in the infrared region. Each instrument consists of an infrared light source, a chopper, a measurement cell, and a detector. The infrared light beam emitted by the source passes through the measuring cell, which is filled with a continuously flowing gas sample. The light beam is partially absorbed or attenuated by the gas species of interest in this cell before reaching the front chamber of the detector. Both the front and rear chambers of the sealed detector are filled with a reference gas. The difference in the amount of light absorbed between the front and rear chambers is dependent on the concentration of the gas species of interest within the sample measurement cell; a pressure differential is thus created between the two chambers. The pressure difference is then observed as gas flow by the micro-flow sensor is rectified, amplified, and linearized into a DC voltage signal for output.

To ensure data accuracy of the Fuji CO analyzer, a set of four calibration gases will be injected through the reference Method 10 sampling system before the compliance testing to demonstrate the linearity of the analyzer. These four gases will consist of purified compressed zero air, CO at 20% of span (low), CO at 50% of span (mid) and CO at 90% of span (high). All gases will be documented traceable to National Bureau of Standards Reference Materials. The output from the Fuji 3300 CO analyzer, as well as the output from the Fuji 3300 CO₂ removed from the sample gas by the ascarite as determined by the Method 3A analysis results.

6.2.8 Total Gaseous Organic - EPA Method 25A

The total gaseous hydrocarbon concentration (THC) will be determined in accordance with the procedures outlined in EPA Method 25A. A continuous sample of the flue gas will be delivered into either a Ratfisch Model RS55 or a Beckman Model 402 hydrocarbon analyzer.

This instrument operates on the principle of flame ionization, a phenomena in which combustion of hydrocarbons results in the generation of carbon ions. This process yields a current directly proportional to the carbon content of the molecules in the gas stream. The current is converted to a voltage output and recorded continuously on a strip chart, as well as a computerized data acquisition system.

The instrument is calibrated using EPA Protocol Number 1, National Bureau of Standards concentrations of propane in nitrogen (N₂). Sample gas to the THC analyzers will pass through a filtering system and a sample conditioning system to remove particulate matter

and water vapor, respectively. The dry-basis concentration of THC is then reported in ppmv as propane.

The instrument is calibrated using National Bureau of Standards concentrations of propane in nitrogen (N₂). The wet-basis concentration of THC is then reported in ppmv as propane.

Calibration and linearity checks are performed through the entire sampling system before and after each test period. "Zero drift" checks are made after each run to adjust the concentration bias due to drift. All parts of the sampling train are heated to a temperature of at least 250°F.

6.2.9 Particulate and Hydrogen Chloride - EPA Proposed Method 26/EPA Method 5*

The sampling and analytical procedures outlined in the EPA Proposed Method 26 and EPA Method 5 in combination with Methods 1 through 4 described above will be used to determine hydrogen chloride (HCl) and particulate emissions. The sampling train impinger system will contain 100 mL of 0.1N sulfuric acid in each of the first two impingers, 100 mL of 0.1N sodium hydroxide in the third impinger, and 200 grams of silica gel in the fourth impinger. A knockout impinger may be necessary depending on source conditions. The nozzle and probe liner will be constructed of borosilicate glass or quartz. The filter holder will be constructed of borosilicate glass with a Teflon frit filter support and a sealing gasket. A quartz-fiber or Teflon mat filter will be used. Nozzles, probe liners, and filter holders will be rinsed thoroughly prior to testing. In all cases, particulate runs will be performed within ± 10% of 100% isokinetic conditions.

The sulfuric acid solution will be analyzed using ion chromatography techniques for HCl. Duplicate analyses will be performed on 10% of the samples and a reagent blank. The sulfuric acid and sodium hydroxide solutions will be analyzed using ion chromatography for the following cations: NH₄⁺, Fe²⁺, Fe³⁺, Al³⁺, Ba²⁺, Ca²⁺, K⁺, Mg²⁺, and Na⁺. EPA Method 5 analytical procedures will be used for the determination of particulates. METCO Environmental routinely retains particulate samples following analysis. Samples are placed in specifically identified containers, and stored in a controlled temperature and humidity environment.

6.2.10 Trace Metals - Multi Metal Sampling Train

EPA Draft Multi-Metal Sampling Train will be used to collect the following metals: antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, silver and thallium. This train is operated in the same manner as a regular EPA Method 5 sampling train. Pretest preparations, preliminary determinations, and leak check

*This method is the same as described in Methods Manual for Compliance with BIF Regulations.

procedures will be those outlined in EPA Method 5. The average sampling rate for each run will be within $\pm 10\%$ of 100% isokinetic conditions. Each test run will be a minimum of two (2) hours in duration and will sample a minimum of 60 dry standard cubic feet. Borosilicate glass or quartz probe liners and nozzles will be used to avoid possible contamination. Figure 6-1 presents a schematic of the EPA MMTL sampling train.

The reagents placed in the impingers will be as follows: 100 mL of 5% HNO_3 /10% H_2O_2 in each of the first two impingers, the third will contain, 100 mL of acidic KMnO_4 , the fourth will remain empty, and the fifth will contain 200 grams of silica gel. In addition, a low metals quartz filter, Pallflex 2500 QAT-UP, will be used. The filter will be placed between the probe exit and the first impinger.

The front half of the sampling train will be rinsed with 0.1 N nitric acid to recover the metals. After impinger adsorbing solution recovery, all sample exposed components will be rinsed into appropriate containers with 0.1 N nitric acid. Surgical gloves will be used during recovery.

The condensate catch determination will be performed according to EPA Method 4 procedures. SW-846 Methods (atomic absorption (AA)) will be used to determine metal concentrations. The filter, filter rinses, and the first three impinger catches and rinses will be analyzed for metals listed. The KMnO_4 impinger reagent and rinse will be analyzed for mercury using cold vapor AA. Duplicate analyses will be performed on 10% of all MMTL samples. In addition a MMTL field, reagent, and filter blank will be analyzed. Spikes will also be added to determine metals recovery efficiencies.

6.2.11 POHCs - SW-846 Method 0030

SW-846 Method 0030 (Volatile Organic Sampling Train (VOST)) will be used to measure emissions of the designated POHCs. The VOST system is designed to draw effluent flue gas through a series of sorbent traps. The first trap will contain Tenax resin, and the second will contain a section of Tenax followed by a section of activated charcoal. The Tenax trap will be preceded by a water-cooled condenser arranged so that condensate will drain vertically through the Tenax. The VOST system is depicted schematically in Figure 6-2.

The Tenax resin will be Soxhlet extracted with methanol for 16 hours, dried in an oven at 100°C , and then packed in sampling tubes. The tubes will be placed in a Nutech VOST tube conditioner and conditioned with nitrogen at 180°C for twelve hours. The conditioned tubes, accompanied by charcoal tubes will be placed in Ziploc bags and then stored in a freezer.

Sampling will take place for 160 minutes per test run; three test runs will be performed per test condition. Each pair of traps will be replaced every 40 minutes so that four pairs of traps will be collected per run, twelve pairs per condition. Three of the four pair of VOST tubes will be analyzed for each run to represent a two hour test run. The fourth will be held for backup and analyzed if one of the first three cannot be analyzed. The VOST will be operated at a sampling rate of 0.5 liter per minute (20 liters per sample). VOST sampling procedures will strictly adhere to SW-846 Method 0030.

Analysis for the POHCs will follow the guidelines of SW-846 Method 5040, a method that has been proposed for volatile organic compounds that are amenable to the purge-trap-desorb (P-T-D) technique. The pair of traps for each sample will be analyzed separately. The condensate for each sample will also be analyzed. The average of the three samples will be reported for each test run. The organic contents will be thermally desorbed from the samples into water using a carrier gas. The desorbed compounds will then be purged from the water and collected on an analytical trap containing Tenax and charcoal. The compounds are then back-purged off the trap into the GC/MS. Helium will be the carrier gas in all cases.

VOST Breakthrough. According to the EPA, breakthrough is present if the catch on the second tube exceeds 30% of the first tube and is above 75 ng. It should be stated that Entropy will follow the guidelines and procedures specified in EPA's SW-846 Method 0030 (VOST). However, Entropy cannot accept responsibility should EPA invalidate results collected according to EPA's suggested guidelines and procedures.

VOST Sorbent Cartridge Blanks. Extra sorbent cartridges will be taken to the sampling site to serve as field and trip blanks. One pair of VOST tubes, designated as a field blank, will be placed in the sampling train, leak checked, and recovered during each day of the testing series. The field blank will be capped and stored for transport in the same manner as the sample-exposed cartridges. The field blanks will be analyzed by the same method as the actual samples. An additional pair of tubes, designated as a trip blank, will be transported to and from the field and otherwise treated as the other cartridges, except that the caps will not be removed. The trip blank will not be analyzed unless the field blanks suggest possible contamination.

Preventing contamination of the sorbent during transport or in the environment surrounding the hazardous waste facility is an important Quality Assurance consideration. Entropy has developed procedures for sample collection and transport which go beyond the requirements of the VOST sampling method. Entropy's system isolates the VOST from the surrounding atmosphere during sampling, effectively preventing sorbent contamination and preserving sample integrity. Blank analyses are consistently below detectable limits, confirming the effectiveness of this procedure.

6.2.12 Polychlorinated Dibenzo-P-Dioxins/Polychlorinated Dibenzofurans and - Proposed EPA Method 23

Proposed EPA Method 23 (M23) will be used to measure emissions of the polychlorinated dibenzo-p-dioxins/polychlorinated dibenzofurans. The M23 sampling train includes a cartridge of XAD-2 resin to capture hydrocarbons that pass through the particulate filter. The sample gas is cooled by a water-cooled condenser prior to encountering the XAD cartridge. The following sections describe each component in the M23 sampling train.

Sampling Train Description. A schematic of a typical M23 sampling train is shown in Figure 6-3. Flue gas is pulled from the stack through a stainless steel nozzle and a borosilicate glass probe. Particulate matter is removed from the gas stream by means of a glass fiber filter (EPM2000) housed in a glass filter holder maintained at $248 \pm 25^{\circ}\text{F}$. The sample gas passes through a water-cooled condenser and into the XAD sorbent trap for removal of the organic constituents. The condenser and XAD trap are arranged in a manner that allows the condensate to drain vertically through the XAD trap. A chilled impinger train will be used to remove water from the flue gas and a dry gas meter will be used to measure the sample gas flow. Sealing greases will not be used on the sample train.

XAD Preparation

The XAD resin is purchased precleaned from Supelco. Care will be taken to ensure that the resin is kept at temperatures below 120°F before and after sample collection to prevent resin decomposition. The sorbent trap will be charged with 20 to 30 grams of the precleaned resin.

The period of time between charging the trap and use in the field will be minimized and will not be allowed to exceed 14 days.

Glassware Preparation. All glass parts of the M23 sample train including the sorbent trap glassware, will be precleaned prior to sampling according to the procedures listed:

1. Soak all glassware in hot soapy water (Alconox) at 50°C or higher
2. H₂O rinse, three (3) times
3. Distilled/deionized H₂O rinse, three (3)times
4. Pesticide grade acetone rinse, three (3)times
5. Pesticide grade methanol/methylene chloride rinse, three (3) times
6. Bake at 450°F for 2 hours.
7. Cap glassware with clean glass plugs or methylene chloride rinsed aluminum foil.

Clean glassware will be capped with precleaned foil or glass plugs until sample train assembly. Following sample recovery, the glassware will be reused at the same sampling location.

Sample Train Operation

The sample train will be operated according to Proposed EPA Method 23. Special attention will be given to the following Quality Control checks: The entire sample train will be leak tested to ensure that leakage does not exceed the lesser of a) 4 percent of the average sampling rate, or b) 0.02 cfm. The probe exit temperature will be maintained above 248°F, and the filter compartment will be maintained at 248°F ± 25°F during sampling. Gas entering the sorbent module will be maintained at or below 68°F. Isokinetic sampling will be maintained within ± 10 percent of 100. Stored resin will be kept below 120°F at all times.

Sample Recovery

Recovery of the M23 samples and assembly of the sample trains will be conducted in an environment free from uncontrolled dust, such as a lab or vehicle if a lab is not available. The sampling train will first be rinsed with acetone and methylene chloride, then toluene, and finally with acetone (which will be discarded).

Sample containers from a typical M23 test run will include:

Container Number 1	Filter(s)
Container Number 2	Rinses of nozzle, probe, and front half of filter holder
Container Number 3	XAD cartridge and resin
Container Number 4	Knockout impinger contents (if used)
Container Number 5	First, second, and third impinger contents
Container Number 6	Rinses of back half of filter holder, transfer line, condenser, and knockout impinger
Container Number 7	Rinses of first, second, and third impingers
Container Number 8	Silica gel

Blanks of each solvent lot used at the test site will also be saved for potential analysis. All sample containers containing water will be extracted for analysis by the designated lab within 14 days after sample collection. It should be noted that depending on the particulate loading and/or the flue gas moisture content, the actual number of containers from each sample run may vary.

Sample Train Blanks

A field blank will be recovered during each operating condition of the testing program. A field blank consists of a complete M23 sampling train (probe, filter, transfer line, condenser, XAD trap, and impinger set) which is assembled as though to collect a sample, but flue gas is not pulled through the train. The field blank train will be leak checked the same number of times as a sampling train used during a test run. The train will be placed at the sampling location for the duration of one test run. The train is then returned to the laboratory and disassembled for recovery using the same procedure used to recover actual samples. The field blank will be obtained using a train that has previously been used to collect at least one actual sample from the test site.

Method 23 Analysis

The XAD traps, filters, and impinger solutions will be analyzed for PCDD/PCDF according to SW-846 Methods 8290X and 8270, respectively. All extracts from one (1) test run will be combined and volume reduced to 1mL resulting in one (1) extract per test run from which an aliquot will be taken for analysis.

6.2.13 Opacity - EPA Method 9

The procedures outlined in EPA Method 9 will be used for the determination of the stack plume opacity. Entropy will place a certified, experienced visible emissions observer on site to record plume opacity for a minimum of one hour per test condition, unless otherwise directed.

6.2.14 Hexavalent Chromium

EPA Draft Method. An EPA Research & Development Sampling Train will be used to collect hexavalent chromium (Cr^{+6}). This train is operated similar to a regular EPA Method 5 sampling train. Pretest preparations, preliminary determinations, and leak check procedures will be those outlined in EPA Method 5. The average sampling rate for each run will be within $\pm 10\%$ of 100% isokinetic conditions. Each test run will be two (2) hours in duration and will sample a minimum of 60 dry standard cubic feet with a lower detection limit of approximately four nanograms/DSCM. To avoid possible contamination, all components which come in contact with samples will be made of glass or Teflon.

The sampling train will consist of four impingers. The contents of the impingers will be as follows: 150 ml 0.1N KOH in the first impinger, 75 ml of 0.1N KOH in the second, 75

ml of 0.1N KOH in the third, the fourth will contain 200 grams of silica gel. A recirculation line is used to constantly wash the probe interior with KOH. Its purpose is to capture hexavalent chromium before it converts to trivalent chromium. Following sampling, a post-test nitrogen purge is also used as a safeguard against conversion of hexavalent chromium to trivalent. The sample train is rinsed into appropriate containers with DI water. Immediately following sample recovery the reagent is pressure filtrated to remove insoluble matter which could cause hexavalet conversion. Sample analysis for Cr^{+6} is determined by ion chromatography coupled with a post column reactor (IC/PCR). Afield blank will also be analyzed and if there is a problem with the analytical results, the reagent blanks will also be analyzed.

SAMPLE CUSTODY

Sample custody procedures for this program are based on EPA recommended procedures (Quality Assurance Handbook for Air Pollution Measurement Systems: Volume 3, Stationary Source-Specific Methods (1977)). Since samples will be analyzed by one or more laboratories as well as in the field, the custody procedures emphasize careful documentation of sample collection and field analytical data and the use of chain of custody records for samples being transported. The procedures which will be used are discussed below.

7.1 Field Sampling Operations

The project manager will be responsible for ensuring that proper custody and documentation procedures are followed for the field sampling and field analytical efforts. He will be assisted in this effort by key sampling personnel involved in sample recovery.

Samples are collected, transported, and stored in clean containers which are constructed of materials inert to the analytical matrix such as glass jars. Only containers which allow air tight seals are used. Amber glass jars are employed when containers are needed to inhibit photochemical reactions.

All sampling data, including information regarding sampling times, locations, and any specific considerations associated with sample acquisition will be recorded on preformatted data sheets. The field sampling data form for an isokinetic type sampling train is shown in Figure 7-1. The analytical data form for an isokinetic type sampling train is shown in Figure 7-2.

Following sample collection, all samples will be given a unique alphanumeric sample identification code as shown in Figure 7-3. Actual sample code could vary slightly depending upon sample location and type. Figure 7-4 is the chain of custody sheet which will accompany all isokinetic type reagent boxes used during the testing program.

Process samples (feed scrubber, ash, etc.) that are collected during testing will be packed for shipment to the appropriate laboratories and process sample chain of custody forms (Figure 7-5) will be completed for each shipment box. The chain of forms and a letter specifying the treatment of each sample will also be enclosed in the sample shipment container. Samples to be combined and analyzed immediately, extracted and stored for later analysis, or simply stored as is, will be clearly stated in this letter. The purpose of the letter will be to ensure that the correct samples are combined, that samples containing moisture will be analyzed on an as-received basis, and that samples with a limited shelf life (those containing water) are extracted within the allowable time of 7 to 10 days after collected.

Samples are stored for transport from the lab to the field to the lab in storage boxes constructed in a fashion which minimizes movement and thus prevents breakage of containers. For example, boxes used for transporting glass containers have foam inserts

with form-fitting cutouts. Sample transport boxes are always locked except when in use. Vans containing equipment and samples are locked whenever they are left unattended.

VOST traps are placed in culture tubes which also contain activated charcoal. These tubes are then placed in Ziploc bags which also hold open charcoal tubes. VOST traps will be kept cold during transportation and storage.

A daily activity log will be maintained by the project supervisor. This will be an informal log used to record various types of information, such as minor problems which arise, sketches of sampling locations, names and phone numbers of plant contacts, daily activity summaries, etc.

7.2 Analytical Operations

Analytical operations will be performed on-site in the laboratory as well as in the Entropy Laboratory. The analytical data form to be used for on-site fixed gas (molecular weight) determinations using Orsat analyzer is included in Figure 7.1 and Figure 7.2. Samples analyzed by outside laboratories are transported with a Request-For-Analysis form as shown in Figure 7-6. This form will list sample identifications, analytical parameters, sample matrices, anticipated date of results, and other relevant information necessary to ensure the appropriate analyses are performed and to document the progress of the samples.

Figure 7-3

JOB IDENTIFICATION NUMBER: _____

JOB NAME: _____

JOB LOCATION: _____

I	-	MS	-	M5/26	-	1	-	Sample Fraction
<u>Operating Conditions</u>		<u>Sampling Location</u>		<u>Sample Type</u>		<u>Run Number</u>		
Condition I		MS - Main Stack		M5/26		1,2,...		
Condition II		MS - Main Stack		M23				Front Wash Filter
Condition III				VOST				XAD Tube
		MMTL						Condensate
		M3A						Impingers
		M6C						Solvent Rinse
		M7E						
		M10						
		Process Samples						
		RMF - Raw Mill Feed						
		RMP - Raw Mill Products						
		KF - Kiln Feed						
		KBC - Kiln Baghouse Catch						
		SHWF - Solid Hazardous Waste Fuel						
		C - Coal						
		T - Tires						

CALIBRATION PROCEDURES AND FREQUENCY

8.1 Sampling Equipment

Entropy's sampling equipment is calibrated according to the criteria specified in the reference method being employed. In addition, Entropy follows the guidelines set forth in the "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods". Dry gas meters, orifices, nozzles, and pitot tubes are calibrated in accordance with this document.

The range of the calibration is specified for all environmental measurements to encompass the range of probable experimental values. This approach ensures that all results are based upon interpolative analyses rather than extrapolative analyses. Calibrations are designed to include, where practical, at least four measurement points evenly spaced over the range. This practice minimizes the probability that false assumptions of calibration linearity will be made. In addition, it is common practice to select, when practical, at least one calibration value which approximates the levels anticipated in the actual measurement.

Typically, calibration frequency is dictated by the need to demonstrate the stability of the calibration value over the course of measurements. Dry gas meters undergo a pretest calibration check on site.

Following the test program, calibrations are checked on all relevant items of sampling equipment to ensure the validity of data collected in the field according to the procedures outlined in Section 11. The calibration performance criteria for the sampling equipment are presented in Table 8-1. Sampling equipment calibration forms are presented in Figures 8-1 through 8-10.

8.2 Analytical Instruments

Calibration materials are employed which ensure calibration accuracy. Materials are prepared according to prescribed procedures. For example, depending on the end use of the data, gaseous calibration standards reflect either traceability to National Institute of Standards and Technology Standard Reference Materials (NIST SRMs) or traceability to in-house primary standards (prepared as part of an established methodology). To ensure that all calibration standards prepared in-house have quality levels that in turn ensure meeting the data quality goals of all measurement programs, the stack test company uses high quality reagents, volumetric devices, balances, etc. When adequate calibration materials cannot be prepared in-house, necessary materials (either NIST SRMs or materials possessing NIST SRM Traceability) are acquired from outside sources. The analytical balance used for gravimetric determination is checked each day of use with Class S weights. The procedure is described in "Quality Assurance Handbook for Air Pollution Measurement Systems," Volume III.

ANALYTICAL PROCEDURES

Tables 6-1 through 6-3 summarize all sampling and analytical procedures to be used during the program. Table 9-1 listed the sample matrix, analytical parameter, analytical method, expected detection limits, and the recommended holding times.

**TABLE 9-1
ANALYTICAL PROCEDURES**

<u>Matrix</u>	<u>Parameter</u>	<u>Analytical Method</u>	<u>Detection Limits</u>	<u>Holding Time</u>
Raw Mill	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	28 days (Hg)-180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	28 days
	Sulfur POHCs	SW-846 6010 SW-846 8240 or 8010/8020	1 ppm 10-1000 ppb	180 days 14 days
Raw Mill Product	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	28 days (Hg)-180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	28 days
	Sulfur POHCs	SW-846 6010 SW-846 8240 or 8010/8020	1 ppm 10-1000 ppb	180 days 14 days
Kiln Feed	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	28 days (Hg)-180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	28 days
	Sulfur POHCs	SW-846 6010 SW-846 8240 or 8010/8020	1 ppm 10-1000 ppb	180 days 14 days
Limestone and Clay	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	28 days (Hg)-180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	28 days
	Sulfur POHCs	SW-846 6010 SW-846 8240 or 8010/8020	1 ppm 10-1000 ppb	180 days 14 days

**TABLE 9-1 (Cont'd)
ANALYTICAL PROCEDURES**

<u>Matrix</u>	<u>Parameter</u>	<u>Analytical Method</u>	<u>Detection Limits</u>	<u>Holding Time</u>
Mill Scale	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	28 days (Hg)-180days
	Chlorine	IC EPA 300.0	0.1 wt.%	28 days
	Sulfur POHCs	SW-846 6010 SW-846 8240 or 8010/8020	1 ppm 10-1000 ppb	180 days 14 days
Kiln Baghouse	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	28 days (Hg)-180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	28 days
	Sulfur POHCs	SW-846 6010 SW-846 8240 or 8010/8020	1 ppm 10-1000 ppb	180 days 14 days
Clinker	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	28 days (Hg)-180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	28 days
	Sulfur POHCs	SW-846 6010 SW-846 8240 or 8010/8020	1 ppm 10-1000 ppb	180 days 14 days
Staurolite	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	28 days (Hg)-180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	28 days
	Sulfur POHCs	SW-846 6010 SW-846 8240 or 8010/8020	1 ppm 10-1000 ppb	180 days 14 days
Fly Ash	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	28 days (Hg)-180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	28 days
	Sulfur POHCs	SW-846 6010 SW-846 8240 or 8010/8020	1 ppm 10-1000 ppb	180 days 14 days
Used Oil/TDF	Caloric Value	ASTM D240-76	Negative	-
	Viscosity	ASTM D2161, 2983	-	-
	Specific Gravity	APHA 213E	-	-
	pH	EPA 150.1	-	Upon receipt
	Ash	ASTM D808	0.1 wt%	-
	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	-
	Sulfur	SW-846 6010	1 ppm	-
	POHCs	SW-846 8240 or 8010/8020	1 ppm	-

**TABLE 9-1 (Cont'd)
ANALYTICAL PROCEDURES**

<u>Matrix</u>	<u>Parameter</u>	<u>Analytical Method</u>	<u>Detection Limits</u>	<u>Holding Time</u>
Solid Hazardous Waste Fuel	Caloric Value	ASTM D240-76	Negative	-
	Viscosity	ASTM D2161, 2983	-	-
	Specific Gravity	APHA 213E	-	-
	pH	EPA 150.1	-	Upon receipt
	Ash	ASTM D808	0.1 wt%	-
	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	180 days
	Chlorine	IC EPA 300.0	0.1 wt.%	-
	Sulfur	SW-846 6010	1 ppm	-
	POHCs	SW-846 8240 or 8010/8020	1 ppm	-
	Coal to Mill	Caloric Value	ASTM D240-76	Negative
Specific Gravity		APHA 213E	-	-
Ash		ASTM D808	0.1 wt%	-
Metals		SW-846 6010 or 7041-7841	0.2-10 ppm	180days
Chlorine		IC EPA 300.0	0.1 wt.%	-
Sulfur		SW-846 6010	1 ppm	-
POHCs		SW-846 8240 or 8010/8020	1 ppm	-
Coal to Kiln		Caloric Value	ASTM D240-76	Negative
	Specific Gravity	APHA 213E	-	-
	Ash	ASTM D808	0.1 wt%	-
	Metals	SW-846 6010 or 7041-7841	0.2-10 ppm	180days
	Chlorine	IC EPA 300.0	0.1 wt.%	-
	Sulfur	SW-846 6010	1 ppm	-
	POHCs	SW-846 8240 or 8010/8020	1 ppm	-
	Main Stack	Particulate	EPA M5	0.1mg
HCl		Ion Chromatography	0.2 ug/mL	28 days
Metals		EPA Draft Metals	0.001-0.017 ug/g	180 days
POHC		SW-846 5040	5-15 ng	14 days
Volatile		SW-846 5040 PCDD/PCDF	5-15 ng SW-846 8290X	14 days 0.02-0.1 ng14 days
Semivolatile		SW-846 8270	5 ug	14 days
Opacity		EPA Method 9	-	-
Benzene		SW846 5040	5-15 ng	14 days

DATA REDUCTION, VALIDATION, AND REPORTING

10.1 Data Reduction

Extreme care will be exercised to ensure hand recorded data is written accurately and legibly. Additionally, the use of prepared data recording forms, conveniently formatted, is an important aid to verify that all necessary data items are recorded. The collected field and laboratory data will be reviewed by the analyst and the Project Manager.

Entropy will reduce and validate all of the sampling and analytical data that is collected. The sampling data will include flow measurements, calibration, etc. Each laboratory will reduce all analytical results prior to their submission to Entropy. The analytical data will be used to determine concentrations and emission rates of the compounds of interest.

Data reduction follows guidelines published in EPA Reference Methods, where applicable, and by guidance documents where EPA Reference Methods are not available. Validated computer programs will be used to calculate all reported values.

10.2 Data Validation

Following reduction of the data, Entropy will review and validate the results, because this test involves collection of data by various individuals from field sampling activities to laboratory analyses. Data validation and coordination are very important. The data from field sampling activities and from laboratory analyses are reviewed for completeness and comparability. A cursory review of the completed data sheets would indicate missing data necessary for accomplishing the objectives of the test. The Project Manager will review the field data and the calculated results. The Project Manager and the laboratory quality assurance officer will perform the same function for analytical data.

Another useful technique to aid in data validation is the analysis of duplicate samples. Duplicate analyses are employed for the precision determination of the laboratory.

10.3 Data Reporting

The testing company's reports cover compliance testing, continuous monitoring performance testing, guarantee testing and a variety of site specific studies. Entropy will present DRE's as well as the concentrations and emission rates for the compounds of interest. DRE's will be reported as percentages, concentrations as ppm or gr/dscf, and emission rates as pounds per hour. Particulate results will be reported in grains/DSCF, corrected to 7% oxygen. An example Table of Contents is presented in Appendix B.

10.4 Report Contents

Copies of the test report will be submitted after the test series has been completed. Results reported will include, but not be limited to, emission rates and concentrations of particulates and gaseous pollutants, and ash or residue determinations, any liquid stream

constituents determinations, and any other type of data requested by the client. This report will also include a list of all personnel present during testing, summary of results, descriptions of test procedures used, a description of the source and its operation during testing, test locations drawings, example calculations, raw field data and equipment calibrations.

INTERNAL QUALITY CONTROL CHECKS

Specific quality control (QC) procedures will be followed to ensure the continuous production of useful and valid data throughout the course of this test program. The QC checks and procedures described in this section represent an integral part of the overall sampling and analytical scheme. Strict adherence to prescribed procedures is quite often the most applicable QC check. A discussion of both the sampling and analytical QC checks that will be utilized during this program is presented below.

11.1 Equipment Inspection and Maintenance

Each item of field test equipment is assigned a unique, permanent identification number. An effective preventive maintenance program is necessary to ensure data quality. Each item of equipment returning from the field is inspected before it is returned to storage. During the course of these inspections, items are cleaned, repaired, reconditioned, and recalibrated where necessary. Each item of equipment transported to the field for this test program is inspected again before being packed to detect equipment problems which may originate during periods of storage. This minimizes lost time on the job site due to equipment failure. Occasional equipment failure in the field is unavoidable despite the most rigorous inspection and maintenance procedures. For this reason, Entropy can quickly respond to the job site replacement equipment for all critical sampling train components.

11.2 Equipment Calibration

New items for which calibration is required are calibrated before initial field use. Equipment whose calibration status may change with use or time is inspected in the field before testing begins and again upon return from each field use. When an item of equipment is found to be out of calibration, it is repaired and recalibrated or retired from service. All equipment is periodically recalibrated in full, regardless of the outcome of these regular inspections.

Calibrations are conducted in a manner, and at a frequency, which meets or exceeds U.S. EPA specifications. Entropy follows the calibration procedures outlined in the EPA Methods, and those recommended within the Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III (EPA-6000/4-77-027b, August, 1977. When these methods are inapplicable, Entropy uses methods such as those prescribed by the American Society for Testing Materials (ASTM).

Data obtained during calibrations are recorded on standardized forms, which are checked for completeness and accuracy by the quality assurance director or the quality assurance manager. Data reduction and subsequent calculations are performed using Entropy's own computer facilities. Calculations are checked at least twice for accuracy. Copies of calibration forms are included in the test or project reports.

Emissions sampling equipment requiring calibration includes pitot tubes, pressure gauges, thermometers, dry gas meters, and barometers. The following sections elaborate on the calibration procedures followed by Entropy for these items of equipment.

A: Pitot Tubes. All Type S pitot tubes used by Entropy, whether separate or attached to a sampling probe, are constructed in-house. Each new pitot is inspected in accordance with the geometry standards contained in EPA Method 2. All Type S pitot tubes, constructed and positioned according to these standards, will have a coefficient of 0.84 ± 0.02 . This coefficient will not change as long as the pilot tube is not damaged.

Each pitot tube is inspected visually upon return from the field. If a cursory inspection indicates damage or raises doubt that the pitot remains in accordance with the EPA geometry standards, the pitot tube is refurbished as needed and recalibrated.

B: Differential Pressure Gauges. Some meter consoles used by Entropy is equipped with 10-inch water column (W.C.) inclined-vertical manometers. Fluid manometers do not require calibration other than leak checks. Manometers are leak checked in the field prior to each test series, and again upon return from the field.

Most of Entropy's meter consoles are equipped with Magnehelic differential pressure gauges. Each set of gauges is calibrated initially over its full range, 0-10 inches W.C. After each field use, the calibration of the gauge set is checked against an inclined manometer at the average delta p encountered during the test. If the agreement is within ± 5 percent, the calibration is acceptable.

C. Impinger Thermometer Prior to the start of testing, the thermometer used to monitor the temperature of the gas leaving the last impinger is compared with a mercury-in-glass thermometer which meets ASTM E-1 No. 63F specifications. The impinger thermometer is adjusted if necessary until it agrees within 2°F of the reference thermometer. If the thermometer is not adjustable, it is labeled with a correction factor.

D. Dry Gas Meter Thermometer The thermometer used to measure the temperature of the metered gas sample is checked prior to each field trip against an ASTM mercury-in-glass thermometer. The dry gas meter thermometer is acceptable if the values agree within $\pm 5.4^\circ\text{F}$. Thermometers not meeting this requirement are adjusted or labeled with a correction factor.

E. Flue Gas Temperature Sensor All thermocouples employed by Entropy for the measurement of flue gas temperatures are calibrated upon receipt. Initial calibrations are performed at three points (ice bath, boiling water, and hot oil). An ASTM mercury-in-glass thermometer is used as a reference. The thermocouple is acceptable if the agreement is within 1.5 percent (absolute) at each of the three calibration points.

Before and after each field use, the reading from the flue gas thermocouple/potentiometer combination is compared with an ASTM mercury-in-glass reference thermometer at ambient conditions. If the two agree within ± 1.5 percent (absolute), the thermocouple and potentiometer are considered to be in proper working order.

F. Dry Gas Meter and Orifice Two procedures are used to calibrate the dry gas meter and orifice simultaneously. The full calibration is a complete laboratory procedure used to obtain the calibration factor of the dry gas meter. Full calibrations are performed over a wide range of orifice settings. A simpler procedure, the posttest calibration, is designed to check whether the calibration factor has changed. Posttest calibrations are performed after each field test series at an intermediate orifice setting (based on the test data) and at the maximum vacuum reached during the test.

Entropy uses, as a transfer standard, a dry gas meter that is calibrated annually against a spirometer. During the annual calibration, triplicate calibration runs are performed at seven flow rates ranging from 0.25 to 1.40 cfm.

G. Dry Gas Meter Each metering system receives a full calibration at the time of purchase and a posttest calibration after each field use. If the calibration factor, Y, deviates by less than five percent from the initial value, the test data are acceptable. If Y deviates by more than 5 percent, the meter is recalibrated and the meter coefficient (initial or recalibrated) that yields the lowest sample volume for the test runs is used.

EPA Method 5 requires another full calibration anytime the posttest calibration check indicated that Y has changed by more than 5 percent. Standard practice at Entropy is to recalibrate the dry gas meter anytime Y is found to be outside the range of 0.97 to 1.03.

H. Orifice An orifice calibration factor is calculated for each flow setting during a full calibration. If the range of values does not vary by more than 0.15 in. H₂O, the arithmetic average of the values obtained during the calibration is used.

I. Barometer Each field barometer is adjusted before each test series to agree within ± 0.1 inches of a reference aneroid barometer. The reference barometer is checked weekly against the station pressure value (corrected for elevation difference) reported by the National Weather Service station at the Raleigh-Durham airport, approximately 2.5 miles from Entropy's location.

**TABLE 11A
STACK GAS SAMPLING
SUMMARY OF PERFORMANCE TEST
METHODS**

Analysis	Sampling Method	Cond. 1	Cond. 2	Cond. 3
Main Stack				
Particulate	M5	+	+	+
Volatile Organics - Benzene	EPA SW-846-0030(VOST)		+	+
PCDD/PCDF and PAH	EPA Draft Method 23		+	+
TCB ⁺	EPA Draft Method 23	+		
SF-6 ⁺	Modified Method 18	+		
Metals	EPA Draft Multi Metals Train	+	+	+
Hydrogen Chloride	EPA SW846-0050 [*]	+	+	+
Sulfur Dioxide	M6C	+	+	+
Oxygen	M3A	+	+	+
Carbon Dioxide	M3A	+	+	+
Carbon Monoxide	M10	+	+	+
Oxides of Nitrogen	M7E	+	+	+
Visible Emissions	M9	+	+	+
Total Hydrocarbons	M25A	+	+	+
Chromium (VI)	EPA Draft Method	+		+

* Entropy will use a 0.3 micron filter which is allowed in the method and will quantify all cationic interferences as defined in this method. Appendix D contains correspondence from USEPA on these issues.

+ TCB is 1,2,4 Trichlorobenzene which is the DE-POHC. Sulfur hexafluoride as a POHC is directly measured by GC on Stack Gas.

11.3 Sampling Quality Control Procedures

The following pretest QC checks will be conducted:

- All sampling equipment will be thoroughly checked to ensure clean and operable components.
- Equipment will be inspected for possible damage from shipment.
- The oil manometer used to measure pressure across the Type S pitot tube will be leveled and zeroed.
- The number and location of the sampling traverse points will be checked before taking measurements.
- The temperature measurement system will be visually checked for damage and operability by measuring the ambient temperature prior to each traverse.

In addition to the general QC procedures listed above, QC procedures specific to each sampling method will also be incorporated into the sampling scheme. These method specific procedures are discussed below.

A: Sampling Train QC Checks. The following QC procedures will be emphasized:

Prior to Start of Tests

- Keep all cleaned glassware and sample train components sealed until train assembly.
- Assemble the sampling trains in an environment free from uncontrolled dust.
- Visually inspect each sampling train for proper assembly.
- Perform pretest calculations to determine the proper sampling nozzle size.

Prior to Testing

- Visually inspect the sampling nozzle.
- Visually inspect the Type S pitot tube.
- Leak check each leg of the Type S pitot tube.
- Leak check the entire sampling train.

During Testing

- Readings of temperature and differential pressure will be taken at each traverse point.
- All sampling data and calculations will be recorded on preformatted data sheets.
- All calibration data forms will be reviewed for completeness and accuracy.
- Any unusual occurrences will be noted during each run on the appropriate data form.
- The project supervisor will review sampling data sheets daily during testing.
- Properly maintain the roll and pitch axis of the Type S pitot tube and the sampling nozzle.

- Leak check the train before and after if any component change takes place.
- Conduct additional leak checks if the sampling time exceeds 4 hours.
- Maintain the probe, filter, and impingers at the proper temperature.
- Maintain ice in the ice bath at all times.
- Make proper readings of the dry gas meter, delta P and delta H, temperature, and pump vacuum during sampling at each traverse point.
- Maintain isokinetic sampling within $\pm 10\%$ of 100%.

After Testing

- Visually inspect the sampling nozzle.
- Visually inspect the Type S pitot tube.
- Leak check each leg of the Type S pitot tube.
- Leak check the entire sampling train.

B: QC for Volumetric Air Flow Rate Determinations.

Flue Gas Velocity

Data required to determine the flue gas velocity will be collected using the methodology specified in EPA Method 2. Quality control procedures are as follows:

- Visually inspect the Type S pitot tube before and after sampling.
- Leak check both legs of the pitot tube before and after sampling.
- Check the number and location of the sampling traverse points before taking measurements.

Flue Gas Molecular Weight

Samples to be used for determination of flue gas molecular weight will be collected using the integrated sampling technique specified in EPA Method 3.

Quality control will focus on the following procedures:

- The sampling train will be leak checked before and after each run.
- A constant sampling rate will be used in withdrawing a sample.
- The sampling train will be purged prior to sample collection.
- The sampling port will be properly sealed to prevent air leakage.

Moisture Content

The moisture content of the gas stream will be determined using the technique specified in EPA Method 4.

The following QC checks will be performed:

- The sampling train will be leak checked before and after each run.
- Ice will be maintained in the ice bath throughout each run.

11.4 Analytical Quality Control Procedures

All analyses for this program will be performed using accepted laboratory procedures in accordance with the specified analytical protocols. Adherence to prescribed QC procedures will ensure data of consistent and measurable quality. Analytical QC will focus upon the use of control standards to provide a measure of analytical precision and accuracy. Also, specific acceptance criteria are defined for various analytical operations including calibrations, control standard analyses, drift checks, blanks, etc. Minimum Quality Assurance Measurements are presented in Table 11-1. The following general QC procedures will be incorporated into the analytical effort:

- The on-site project manager will review all analytical data and QC data on a daily basis for completeness and acceptability.
- Analytical QC data will be tabulated using the appropriate charts and forms on a daily basis.
- Copies of the QC data tabulation will be submitted to the quality assurance manager following the completion of the test program.
- All hard copy raw data (i.e., chromatograms, computer printouts, etc.) will be maintained in organized files.

Specific analytical QC procedures for the Orsat analyzer are listed below.

- The analyzer will be leveled and the fluid levels zeroed prior to use.
- The analyzer will be leak checked prior to use.
- The analyzer will be thoroughly purged with sample prior to analysis.
- The analyzer will be checked by analyzing an ambient air sample.

Hydrogen Chloride Analytical QC Checks are listed below.

- Lab (reagent) blanks will be analyzed for every 10 samples or at least one per matrix.
- Duplicates are analyzed for each sample and should agree within 10%.

**TABLE 11-1
MINIMUM QUALITY ASSURANCE MEASUREMENTS**

<u>SAMPLE</u>	<u>PARAMETER</u>	<u>BLANKS*</u>	<u>PERCENT DUPLICATES**</u>	<u>SURROGATE MATRIX SPIKES</u>
Raw Mill Feed	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Raw Mill Product	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Kiln Feed	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Limestone and Clay	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Mill Scale	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Kiln Baghouse Catch	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Clinker	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Used Oil/TDF	Caloric Value	1 MB	10%	-
	Viscosity	1 MB	10%	-
	Specific Gravity	1 MB	10%	-
	pH	1 MB	10%	-
	Ash	1 MB	10%	-

TABLE 11-1 (Con't)
MINIMUM QUALITY ASSURANCE MEASUREMENTS

<u>SAMPLE</u>	<u>PARAMETER</u>	<u>BLANKS*</u>	<u>PERCENT DUPLICATES**</u>	<u>SURROGATE MATRIX SPIKES</u>
	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Solid Hazardous Waste	Caloric Value	1 MB	10%	-
	Viscosity	1 MB	10%	-
	Specific Gravity	1 MB	10%	-
	pH	1 MB	10%	-
	Ash	1 MB	10%	-
	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Coal	Caloric Value	1 MB	10%	-
	Specific Gravity	1 MB	10%	-
	Ash	1 MB	10%	-
	Metals	1 MB	10%	-
	Chlorine	1 MB	10%	-
	Sulfur	1 MB	10%	-
	POHCs	1 MB	10%	100%
Main Stack	Particulate	Acetone	-	-
	HCl	RB	100%	-
	Metals	FB, RB, FLB	10%	-
	POHCs	FB, TB	-	100%
	Volatile PICS	FB, TB	-	100%
	PCDD/PCDF	FB	-	100%
	Semivolatile PICS		FB	-100%
	Opacity	-	-	-

* MB - Method Blank
 FB - Field Blank
 TB - Trip Blank
 RB - Reagent Blank
 FLB - Filter Blank

** Percent duplicates corresponds to all performance test samples collected.

PERFORMANCE AND SYSTEMS AUDITS

Performance and systems audits are routine elements of all Entropy QA/QC Programs. System audits are always conducted by test team leaders prior to departure for the sampling site. These audits entail the use of equipment check lists, previously completed by the Project Manager and Project Supervisor, to assure the inclusion of all materials and equipment necessary for the sampling task.

The laboratory staff conducts system audits of the laboratory on a regular basis and reports to the Quality Assurance Manager as conditions require. In addition to these system audits, performance audits (if applicable) of the laboratory are while processing samples for analysis. Audit materials typically include materials available from the EPA or materials specially prepared by the QAC.

Relative to the emissions sampling associated with this project, a total system audit technique is not presently available. However, an on-site qualitative inspection and review of each sampling system will be performed by the Project Manager. This systems audit will include observation and recording of the following activities:

- 1) Sample train assembly and leak checks
- 2) Posttest leak checks and sample train disassembly
- 3) Sample recovery, labeling, and storage for shipment

Entropy will perform any audit requested by the regulatory agencies.

PREVENTIVE MAINTENANCE

Well maintained equipment is a very important factor in assuring the quality and completeness of measurement data. Measurements conducted in the field are more sensitive to the degree of equipment maintenance than are measurements conducted within a laboratory setting, because the former are often performed in an uncontrolled dynamic environment.

Entropy minimizes the potential impact of equipment malfunction on data completeness through two complementary approaches. First, an in-house equipment maintenance program is part of routine operations. The maintenance program's strengths include:

- (a) trained technicians experienced in the details of equipment maintenance and fabrication,
- (b) adequate spare parts inventory, and
- (c) the availability of tools and specialized equipment.

The second approach is based upon equipment redundancy. Backup equipment, spare parts, and tools are included on the materials transported to the field for each sampling task. This approach allows the sampling team to respond to equipment breakage or malfunction in a timely fashion, minimizing the quantity of lost data.

For field equipment, preventive maintenance schedules are based on the results of routine inspections and on accumulated experience. Maintenance schedules for major analytical instruments (e.g., balances, gas chromatographs) follow manufacturer's recommendations.

SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The experimental design allows data to be grouped into sets that can be analyzed to evaluate data precision and accuracy. The general tendency and dispersion of data is assessed by reviewing the following parameters.

14.1 Precision

Precision is used to demonstrate the reproducibility of analytical results obtained using the same method. This precision will be evaluated by calculating the mean, relative standard deviation, and relative percent difference.

14.1.1 Arithmetic Mean

The mean of a series of replicate measurements offers an average value for the measurement. The mean is calculated as:

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n}$$

where:

n = number of replicate measurements, and
 X_i = value of each individual measurement.

14.1.2 Relative Standard Deviation

The relative standard deviation (RSD) of a series of measurements offers an assessment of the degree of dispersion of the measurements as a percentage of the mean value. The percent RSD is calculated as:

$$\% \text{ RSD} = (s/\bar{X}) \times 100\%$$

where:

S = standard deviation

$$S = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n-1}}$$

14.1.3 Relative Percent Difference

The relative percent difference (RPD) will be calculated for duplicate analyses by the following equation:

$$\text{RPD} = \frac{X_1 - X_2}{(X_1 + X_2)/2} \times 100\%$$

14.2 Accuracy

Accuracy will be determined from Audit or Performance Evaluation samples if provided by the EPA or regulatory agency. Accuracy will be measured by calculating the percent relative error between the value obtained from the analysis and the actual known value. The percent relative error is determined by the following equation:

$$\% \text{ Relative Error} = \frac{\text{value} - \text{actual}}{\text{actual}} \times 100\%$$

14.3 Completeness

Completeness is determined from the number of samples analyzed and the number of samples collected. Completeness is calculated using the following equation:

$$\text{Completeness} = \frac{\text{number of samples analyzed}}{\text{number of samples collected}}$$

Table 5-1 lists the target precision, accuracy, and completeness objectives for various parameters expected for this project.

CORRECTIVE ACTION

Any samples not collected according to the methods will be considered for voiding. Any equipment found to be out of calibration or not operating properly will be repaired or replaced before additional measurements are made.

If weakness or problems become apparent during system or performance audits, corrective action will be initiated immediately. Examples of corrective actions which might be taken are recalibration of instruments using freshly prepared calibration standards, and replacement of reagents that give unacceptable blank values.

Should performance audits indicate that any methods are out of control, the Quality Assurance Coordinator and Project Manger will review the audit data and determine the appropriate action to be taken. Independent calibration checks will be used if necessary to resolve problems with an analytical system.

In all cases where standard procedures cannot resolve the problem, the QAC and the Project Manager will determine the appropriate action. The Project Manager has daily contact and detailed familiarity with the procedures. The QAC offers a broader background and more detached viewpoint. This combination helps in formulating quick and reliable solutions to various problems. The Project Manager will be responsible for initiating the action, and the QAC will be responsible for determining if this action has resolved the problem.

Entropy's handling procedure for sorbent traps, from blank checking to sample collection and analysis, are designed to eliminate contamination in field blank sorbent traps by limiting their exposure to contaminants in the ambient air. Corrective action will be taken if the field blanks are significantly different from the trip blanks. This comparison will indicate whether high levels in the field blank are due to contamination from exposure to ambient air or from degradation of the sorbent traps.

QA activities when criteria associated with routine activities were not met, and when methods were being evaluated:

1. replicate determination, to enable estimates of the precision of sampling and analysis phases;
2. split samples, in order to assess either the quality of the sample preparation phase or to acquire an independent check of the measurement result;
3. spiked samples, in order to assess the influence of matrix effects;
4. checks of reagent quality; and
5. checks of the adequacy of calibration standards.

QUALITY ASSURANCE REPORTS TO MANAGEMENT

Quality assurance reporting and documentation are important elements of final project reports. Reports provided to agency and industrial clients contain sections which (a) describe QA/QC activities and criteria relevant to the measurements reported, (b) provide results from the application of these activities, and (c) address the impact of these results on the measurements reported. The QA procedures used in this test program generate sufficient documentation to indicate data quality. QA results will be included in the final report. At a minimum the following will be addressed in the final report:

- (a) Precision
- (b) Recovery (VOST accuracy)
- (c) Blanks
- (d) Sampling and analysis equipment calibration
- (e) Cylinder gas audits and on-site equipment audits
- (f) Spike composition verification and recovery checks

All evidence of the execution of the QA guidelines is reviewed by management. In addition, during regular meetings of the project managers, all aspects of the project are discussed, including the QA of each task. Entropy's emission test and laboratory groups participate in audits by EPA's Quality Assurance Division, and Entropy's QA officer makes independent audit checks.

APPENDIX A

DRE CALCULATIONS

[TO FOLLOW]

APPENDIX B

EXAMPLE REPORT TABLE OF CONTENTS

[TO FOLLOW]

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INTRODUCTION

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DISCUSSION OF RESULTS

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SAMPLING AND ANALYTICAL PROCEDURES

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- A. Location of Sampling Points
- B. Source Emissions Calculations
- C. Calibration Data
- D. Field Testing Data
- E. Analytical Data
- F. Oxides of Nitrogen Monitor and Carbon Monoxide Monitor Data
- G. Sulfur Dioxide Monitor and Total Hydrocarbons Monitor Data
- H. Oxygen Monitor and Carbon Dioxide Monitor Data
- I. Visible Emissions Data
- J. Plant Operational Data
- K. Chain of Custody
- L. Resumes of Test Personnel

Appendix B

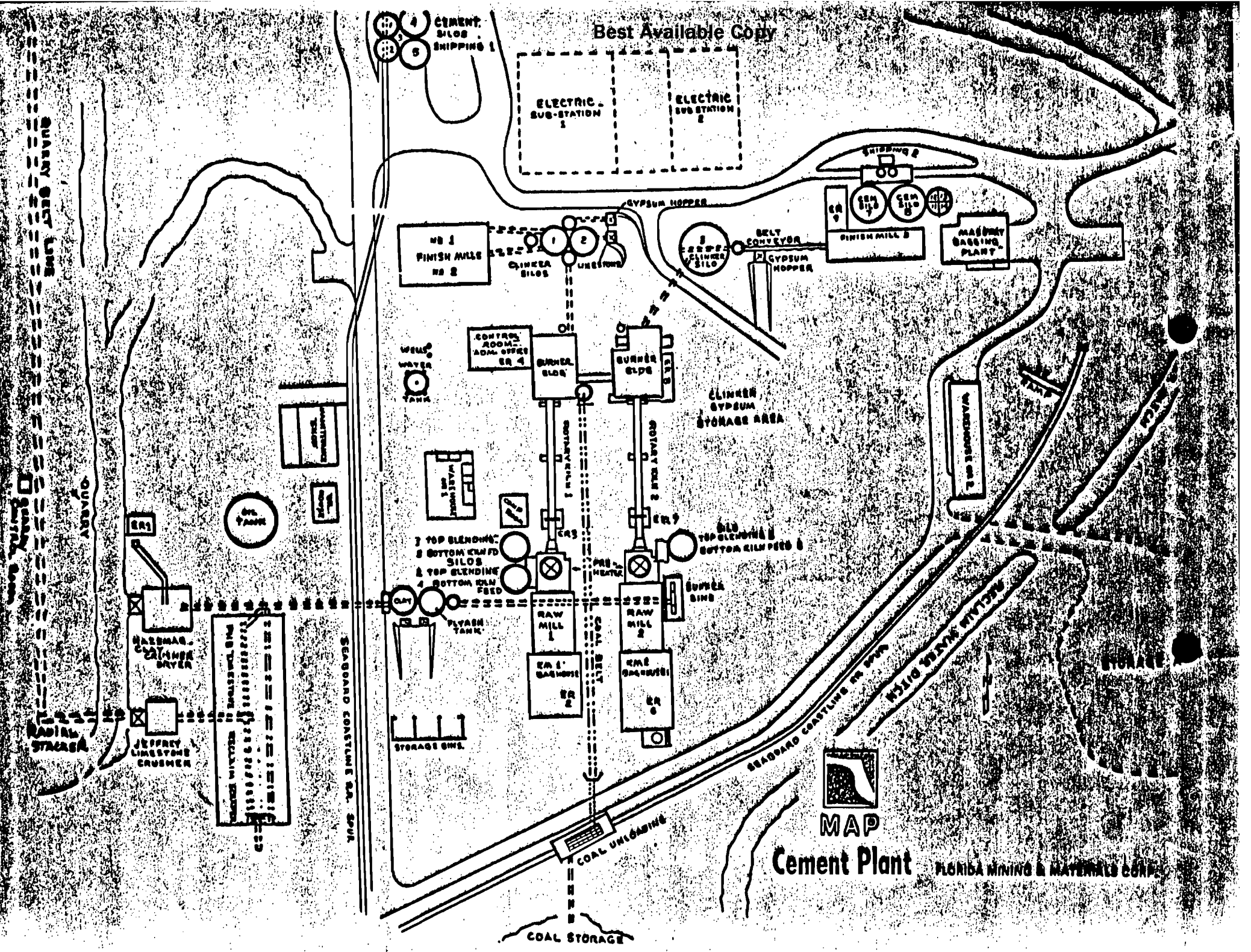
Kiln Process Flow Sheet

**Performance Test Plan
Florida Mining & Materials
Brooksville, Florida**

February 22, 1991

**Review Draft for Hernando County
Review and Advisory Committee**

Best Available Copy



CEMENT
SILOS
SHIPPING 1

ELECTRIC
SUB-STATION 1

ELECTRIC
SUB-STATION 2

NO 1
FINISH MILLS
NO 2

CONTROL
ROOM
ADM. OFFICE
ER 4

BURNER
BLOCK

BURNER
BLOCK

CLINKER
GYPSUM
STORAGE AREA

FINISH MILL 1

MASONRY
BAGGING
PLANT

7 TOP BLENDING
8 BOTTOM KILN
SILOS

RAW
MILL 1

RAW
MILL 2

5 TOP BLENDING
6 BOTTOM KILN
SILOS

HAZEMAN
CRAWLER
DRYER

JERRY
LIMESTONE
CRUSHER

WATER
TANK

SEABOARD COASTLINE R.R. SPUR

CLAY
TANK

STORAGE
SILOS

COAL
UNLOADING

COAL STORAGE



MAP

Cement Plant FLORIDA MINING & MATERIALS CORP.

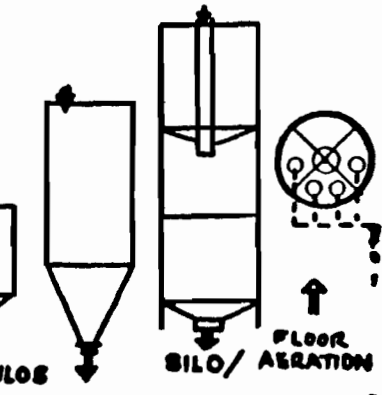
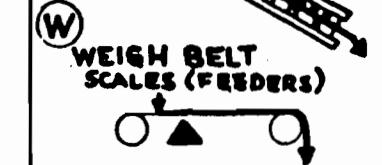
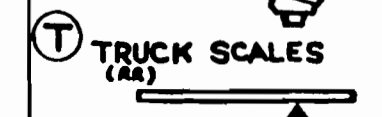
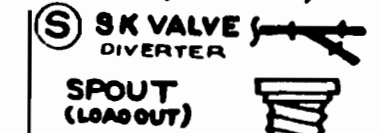
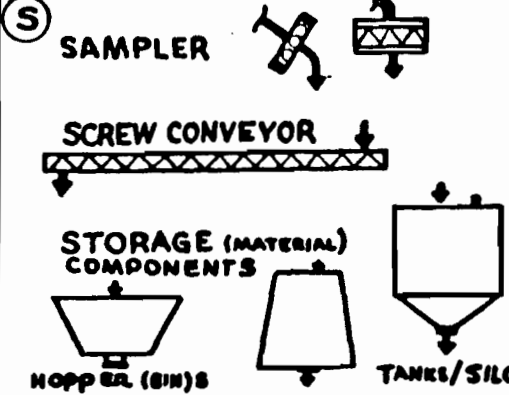
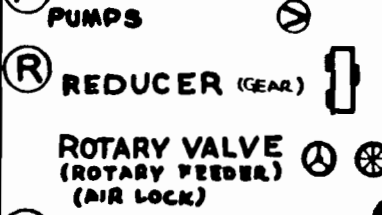
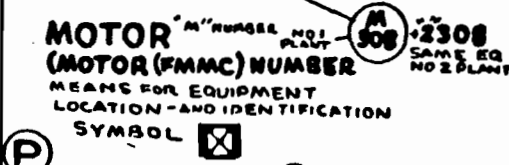
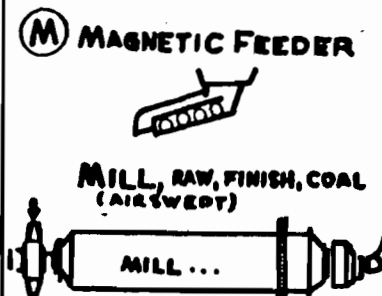
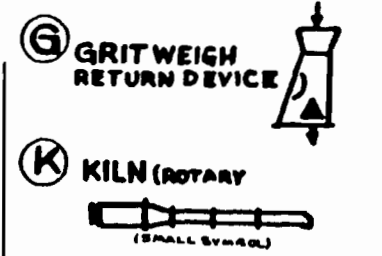
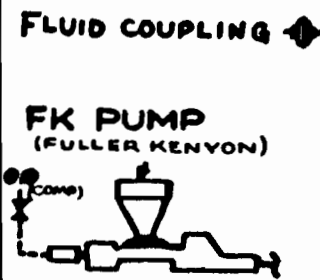
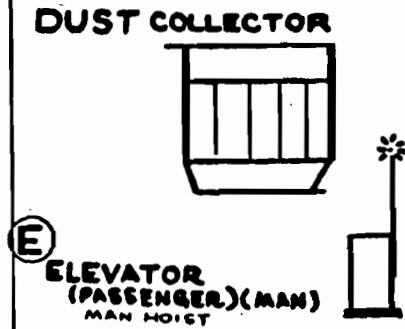
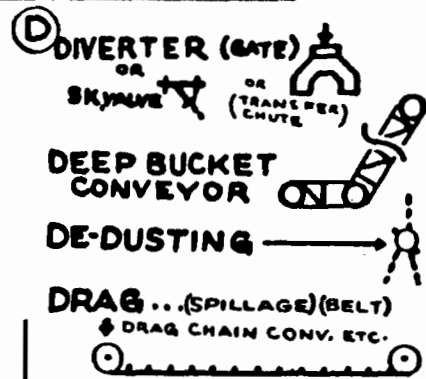
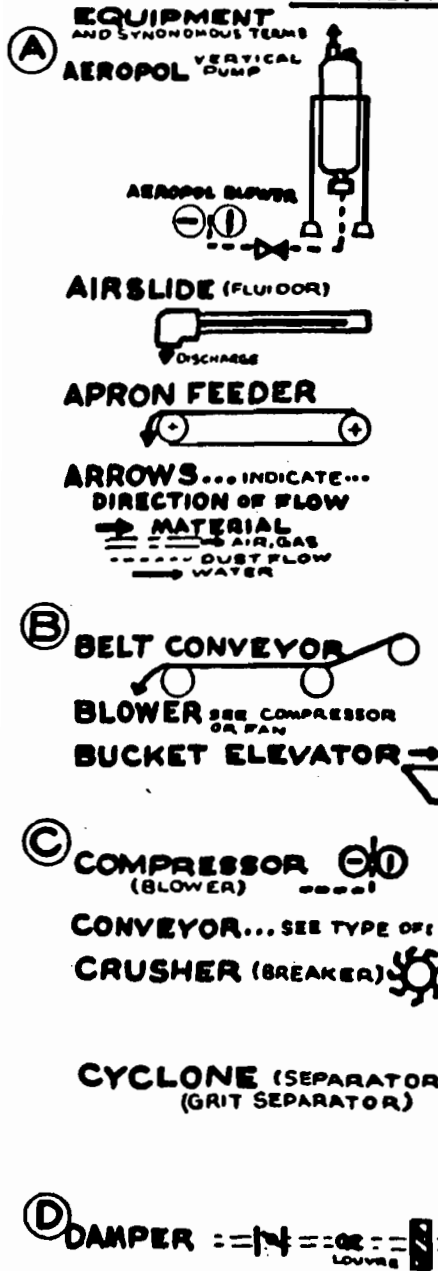
R E F E R E N C E G U I D E
 F L O R I D A M I N I N G A N D M A T E R I A L S " M " (M O T O R) N U M B E R S

<u>PLANT NO 1</u>	<u>PLANT NO 2</u>
000	000
QUARRY, LIMESTONE MINING AND CONVEYING, QUARRY	
100	100
PRIMARY CRUSHING AND DRYING, CLAY	
200	2200
RAW MATERIALS TRANSPORT AND STORAGE, PHB	
300	2300
RAW MATERIAL GRINDING SYSTEM	
400	2400
RAW MEAL BLENDING AND STORAGE	
500	2500
PREHEATER, KILN FEED SYSTEM	
600	2600
COUNTERFLOW PREHEATER	
700	2700
ROTARY KILN AND COOLER	
800	2800
FUEL SYSTEM, OIL, COAL SUPPLY AND FIRING	
900	2900
GRATE COOLER SYSTEM	
1000.....	3000
KILN, MILL AND COOLER BAG FILTER SYSTEMS	
1100.....	3100
CLINKER, GYPSUM, FLYASH STORAGE AND TRANSPORT SYSTEMS ..	
1200.....	3200
CEMENT GRINDING SYSTEM	
1300.....	3300
CEMENT TRANSPORT, STORAGE AND LOADOUT SYSTEMS	
1400.....	3400
SERVICES... PLANT AIR SYSTEMS	
	3500
	MASONRY BAGGING SYSTEM.....
1900.....	SHOP EQUIPMENT
2000	MOBILE EQUIPMENT

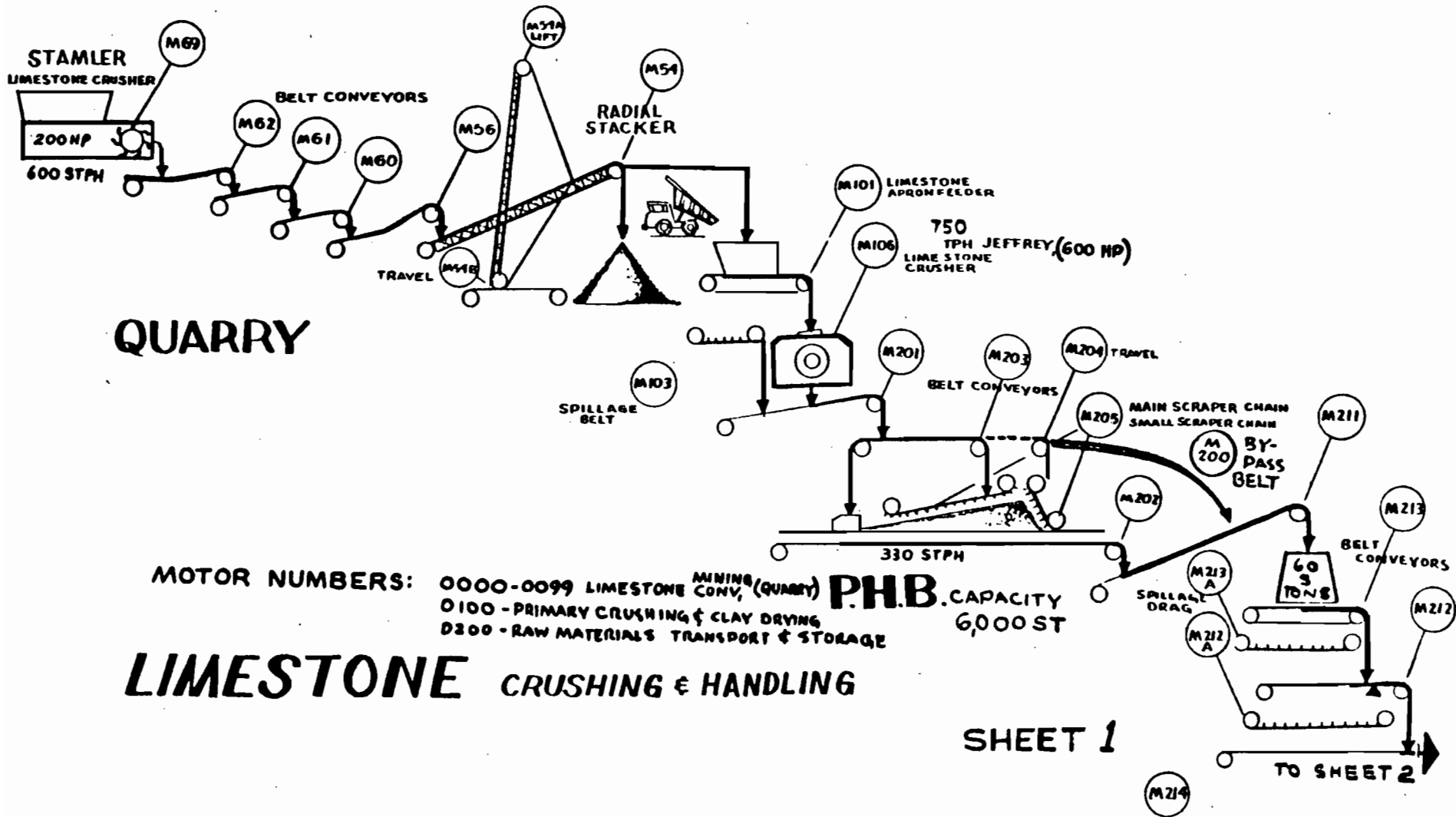
SYMBOLS

FOR

SCHMATIC DIAGRAMS F.M.M.C., BROOKSVILLE CEMENT PLANT (1984)



SHEET 1



QUARRY

MOTOR NUMBERS: 0000-0099 LESTONE MINING CONV. (QUARRY)
 0100 - PRIMARY CRUSHING & CLAY DRYING
 0200 - RAW MATERIALS TRANSPORT & STORAGE

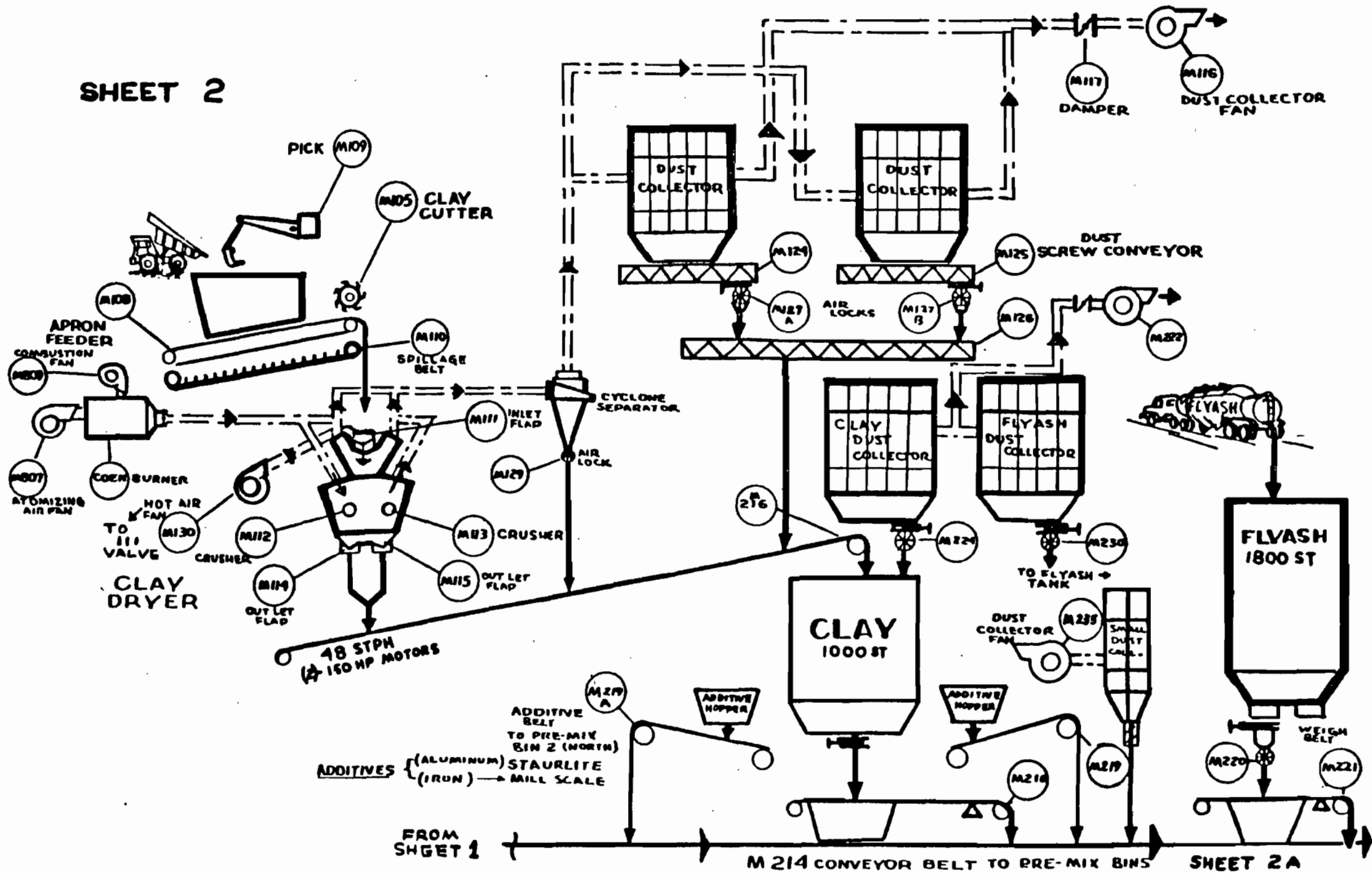
P.H.B. CAPACITY 6,000 ST

LIMESTONE CRUSHING & HANDLING

SHEET 1

M214

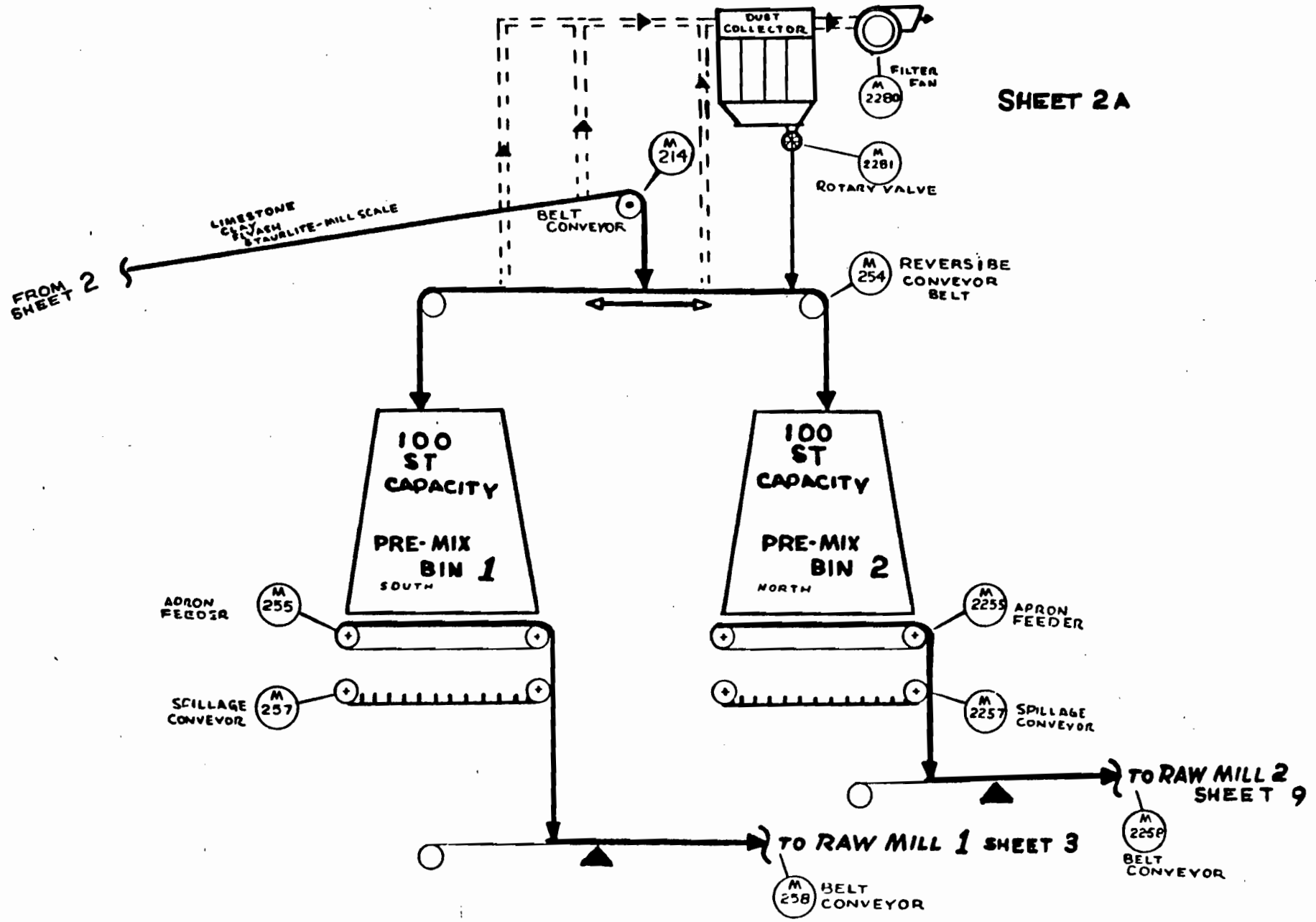
SHEET 2



CLAY, ADDITIVES HANDLING-STORAGE

SHEET 2

MOTOR NUMBERS: 0100 PRIMARY CRUSHING/CLAY DRYING
 0200 RAW MATERIALS TRANSPORT/STORAGE
 0800 OIL FIRING (DRYING SYSTEM)



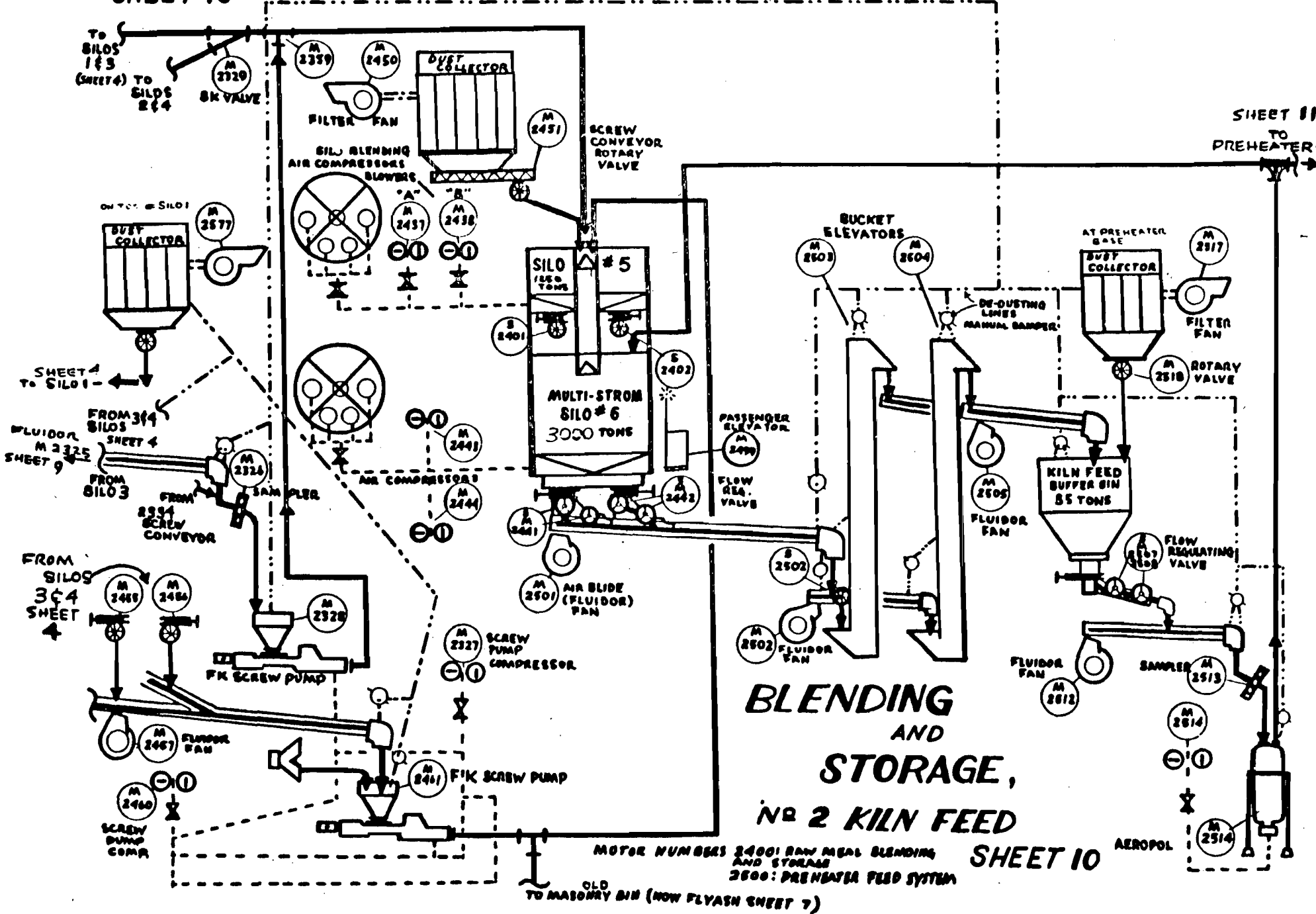
RAW MILL FEED STORAGE

PRE-MIX BINS
FEED TRANSPORT

MOTOR NUMBERS 0200: RAW MATERIALS TRANSPORT/STORAGE

SHEET 10

SHEET 11
TO PREHEATER



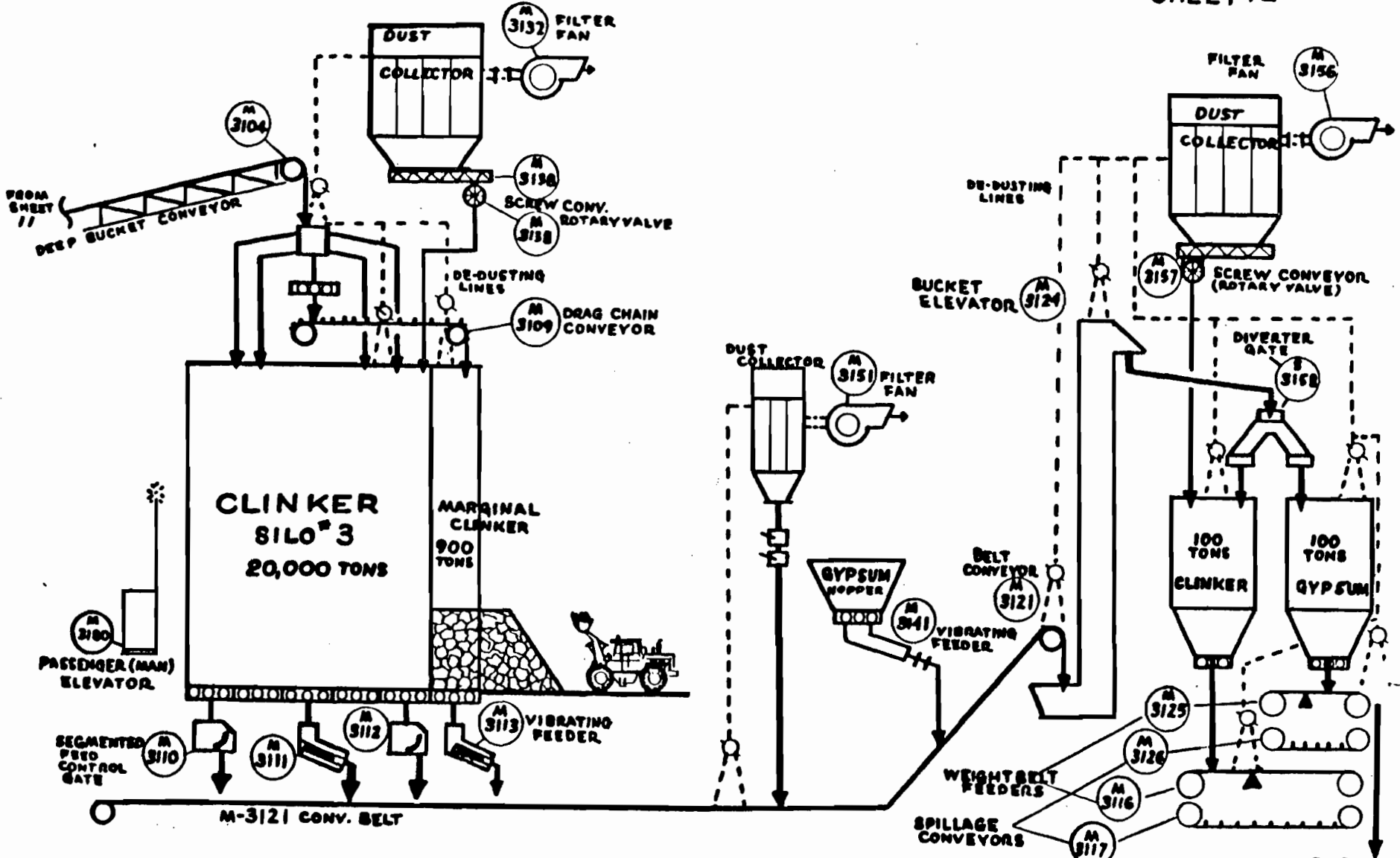
**BLENDING
AND
STORAGE,
NO 2 KILN FEED**

SHEET 10

MOTOR NUMBERS 2400: RAW MEAL BLENDING AND STORAGE
2500: PREHEATER FEED SYSTEM

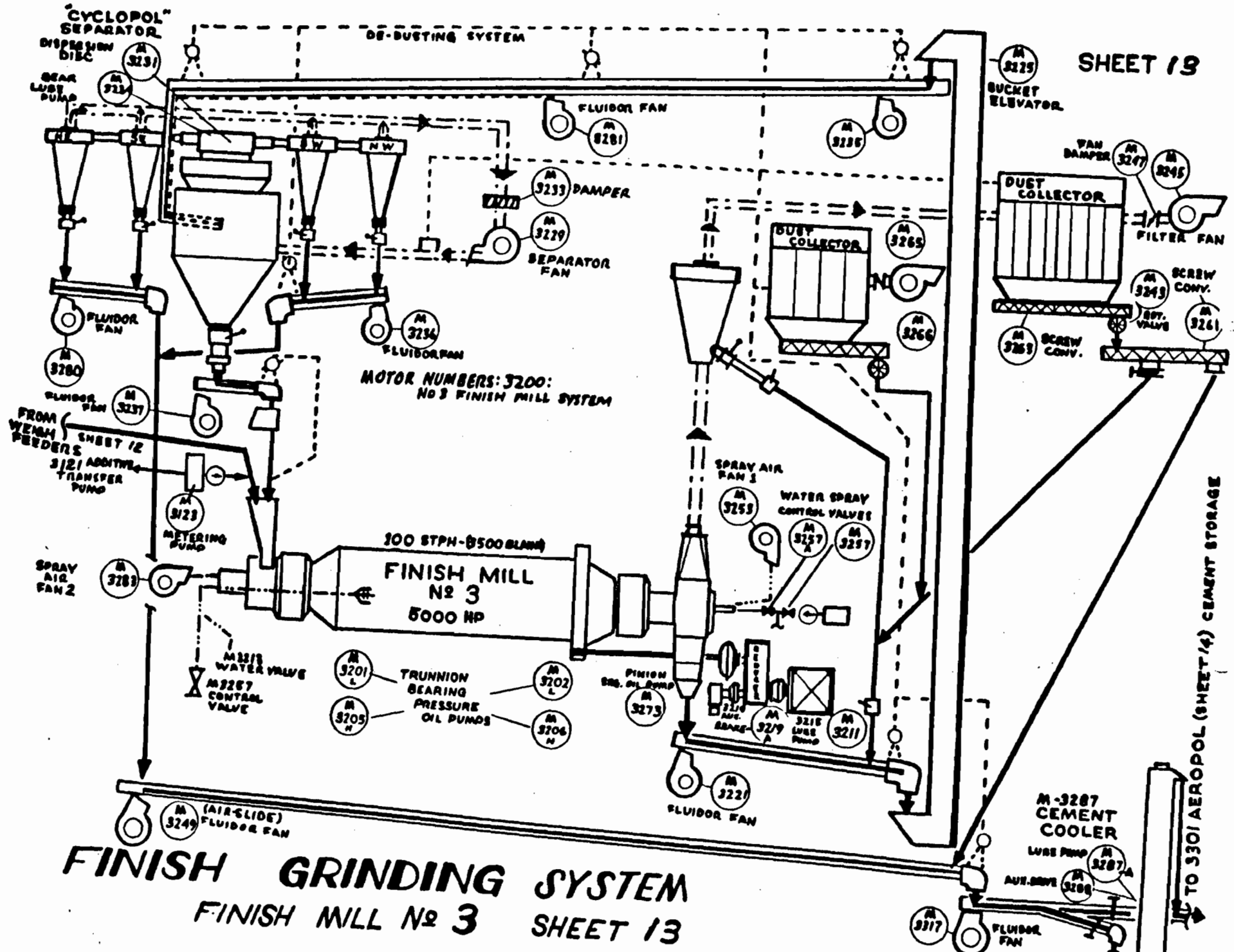
OLD TO MASONRY BIN (NOW FLYASH SHEET 7)

AEROPOL



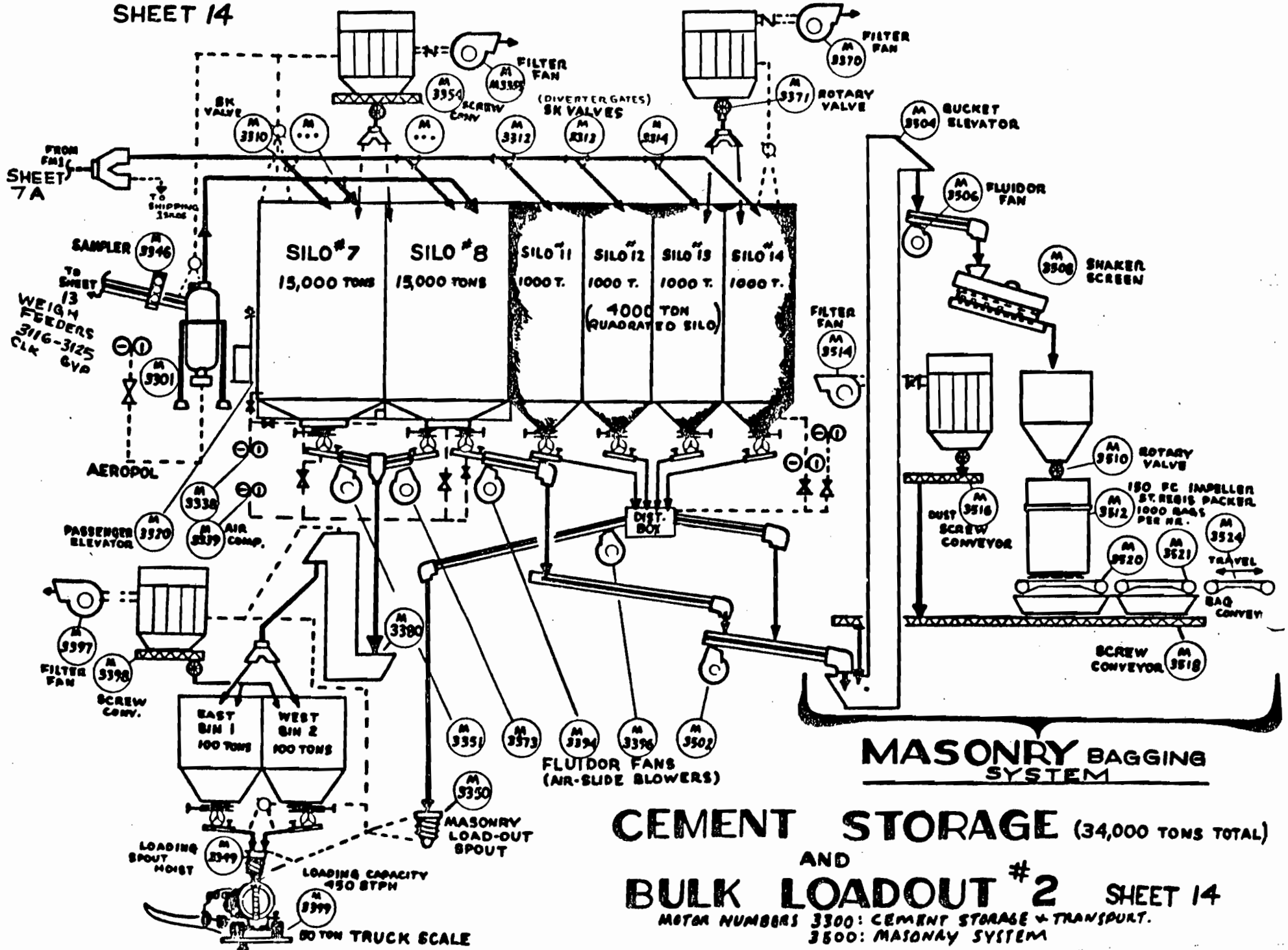
CLINKER TRANSPORT & STORAGE #2 KILN
SHEET 12

MOTOR NUMBERS 3100: CLINKER/GYP TRANSPORT/STORAGE
 NO 2 MILL, NO 3 FINISH MILL



FINISH GRINDING SYSTEM
FINISH MILL No 3 SHEET 13

TO 3301 AEROPOL (SHEET 14) CEMENT STORAGE



MASONRY BAGGING SYSTEM

CEMENT STORAGE (34,000 TONS TOTAL)
AND
BULK LOADOUT #2 SHEET 14
 MOTOR NUMBERS 3300: CEMENT STORAGE + TRANSPORT.
 3500: MASONRY SYSTEM