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NATIONAL OIL SERVICES OF FLORIDA

National Oil Services of Florida

Division of  
International Petroleum Corp.

FLD 065 680 613

Plant City

Accompanies permit application

HO 29-87589

(see permit file)

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Materials

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1.0 DESCRIPTIONSOUTH WEST DISTRICT  
TAMPA1.1 DESCRIPTION OF FACILITY OPERATIONS

The primary operation of the facility consists of the dispatching of vehicles and equipment for waste oil pickup services to the petroleum industries in general. The facility buys used or waste oil directly from facilities that generate it and facilities that do not generate it such as collectors, processors, brokers and distributors (See Table 3 for Company Buying Standard). The land is presently used as a tank farm for the handling, treating and storage of raw, in process and finished oils. The raw or unprocessed oils include and are limited to: automotive crankcase oil, diesel crankcase oil, hydraulic fluid, industrial lubricants, PCB free transformer oils, and virgin fuel No.'s 2, 4, 5 and 6 oils. It specifically excludes solvents, metal working oils, rolling oils, sludges and tank bottoms from tank cleaning. These oils are provided both as supply to other facilities and sold as fuel. None is burned at this facility. Some are further processed to demetalize and dewater and then blended at the facility for burner fuel. None is disposed of as a waste.\* None is used, sold or provided to another facility for use as a non-fuel. Certain oils are blended as number 4 and 5 oil for flots for the phosphate industry.

\* Hazardous wastes have been disposed of by Rollins, Waste Control Systems and Chemical Waste Management, Inc.

1.1.1 SUMMARY OF OPERATIONS

International Petroleum Corporation collects, transports, treats and stores waste oils according to State and Federal regulations as a hazardous waste management facility. It then renders the waste non-hazardous by demetalizing and blending it to ASTM, NBS, and API specifications on site. It thereby recycles the waste oil as burner fuel and flots products equivalent to or superior to virgin fuels and sells them off-site.

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Hazardous Waste

PROCESS FLOW OUTLINE

Oil is collected from approximately 6000 locations like service stations, trucking terminals, auto dealerships, airports etc. It is unloaded into receiving day tanks. It is processed after certification by detailed analytical analyses. It is then stored in master batches until it is sold or transferred to other facilities. The process is detailed below. The oil collected during each day by our trucks is delivered to the plant usually after 3:00 P.M. daily. The oil collected is pumped from each truck into receiving tanks. The daily deliverable capacity of the receiving tanks seldom exceeds 25,000 gallons. The combined volume of these tanks is 60,000 gallons. This is more than adequate for day to day operation. This projects to annual sales of approximately 5 - 6 million gallons per year. The collected oil is allowed to settle overnight in the receiving tanks. The following morning the water that has settled out of the oil is pumped from the receiving tanks to the water holding tank. The remaining oil is transferred to the treat tank via the hot oil heat exchanger. The oil is heated to a temperature of 150° - 160° F. during passage through the heat exchanger and held at 140° - 150° F. in the treat tank. A proprietary set of regents are added to the hot oil. These chemicals drastically reduce the solubility of the metals in the oil and break emulsions. These metals include the wear metals like cadmium, chromium, and iron; the additive metals like barium, calcium, zinc, magnesium and sodium; and the lead from oils used in gasloine engines. As the water decreases in the oil the metallic salts grow into larger crystals which are then removed through a clarifier by polymeric addition. The heat imparted causes any residual water to drop out. As new oil is added to the treat tank, the older dry demetalized oil is pumped into various tanks in the farm for batch blending according to its specifications. A flow diagram of process occurs in Figure D2A.

As it accumulates, the finished product is pumped into storage tanks and then it is transferred to rail tank cars or transports and shipped. The water holding tank also serves as a reservoir for oil-water separation. It also serves as a spill receiver in that

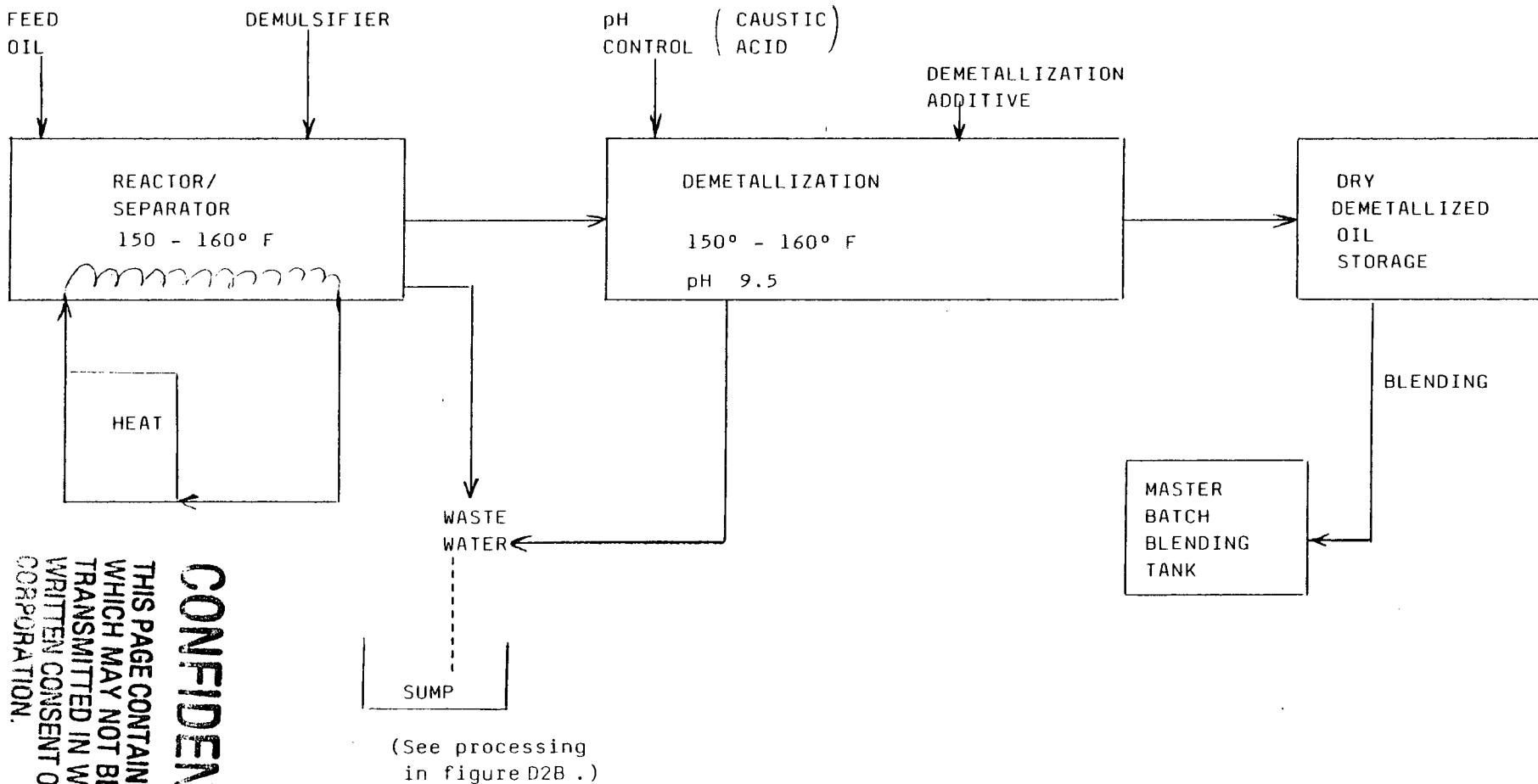


FIGURE D2A I.P.C. PROCESS DIAGRAM, 20,000 GALLON BATCH PROCESS

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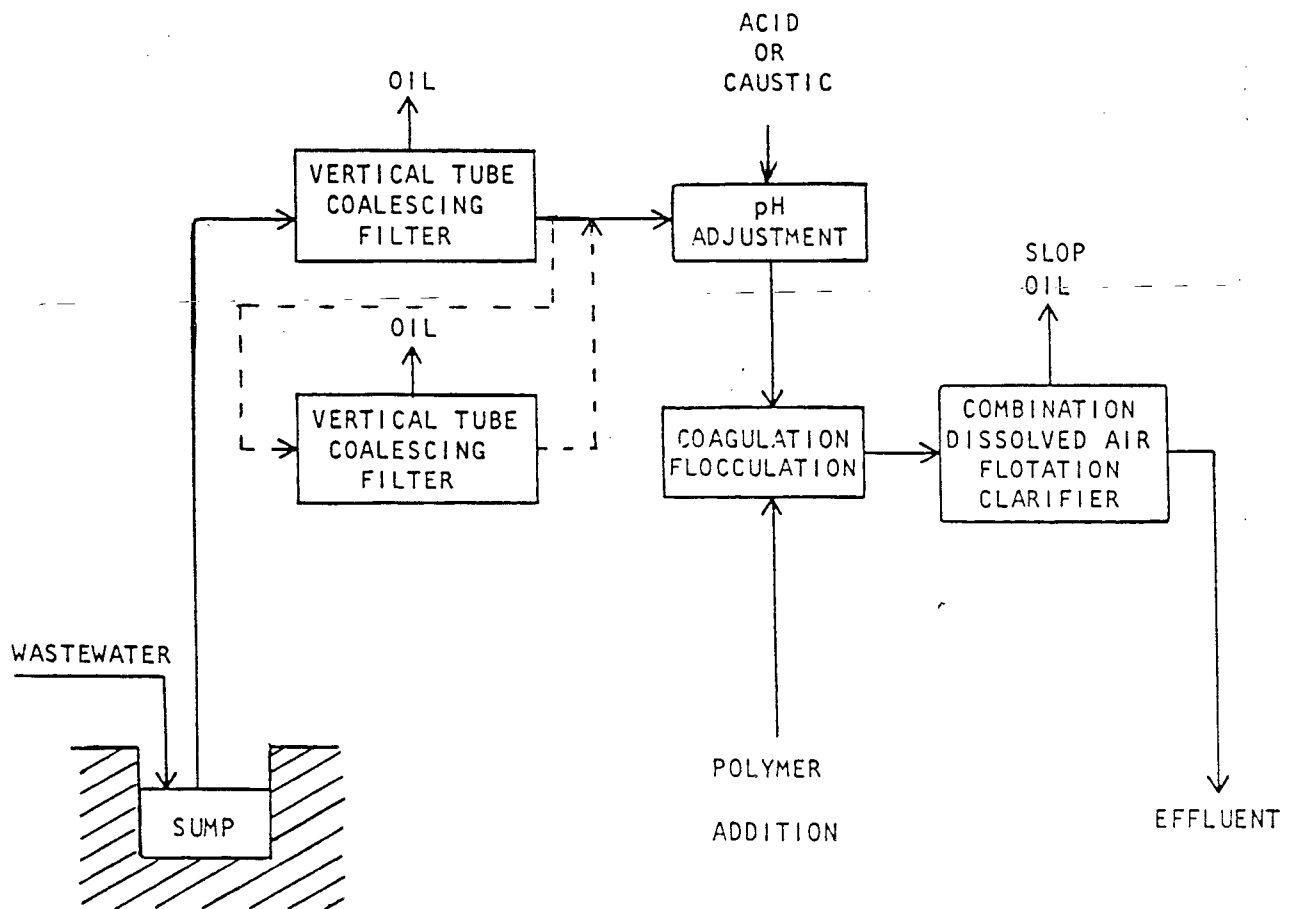


Figure D2B Schematic of aqueous treatment system.

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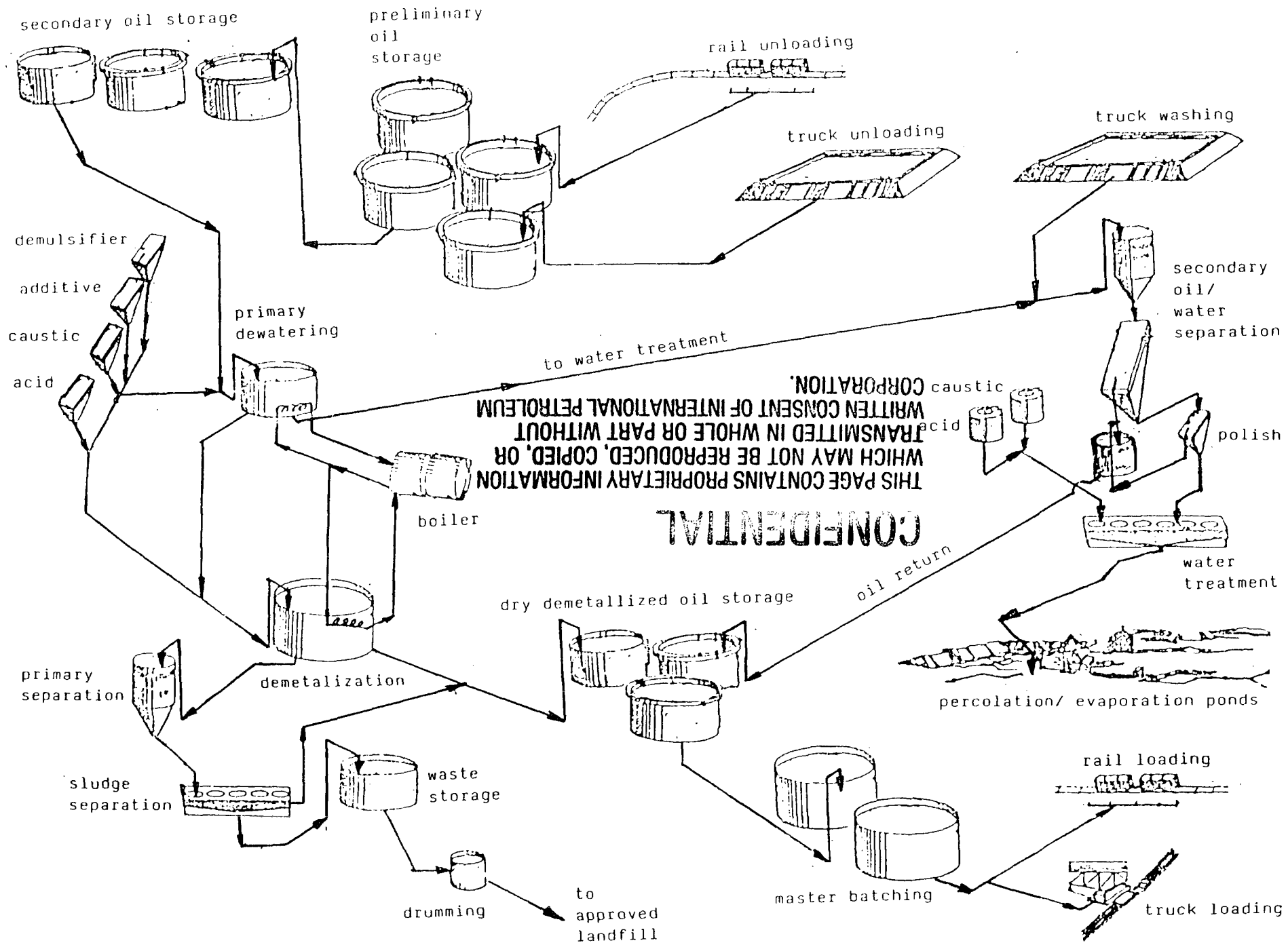
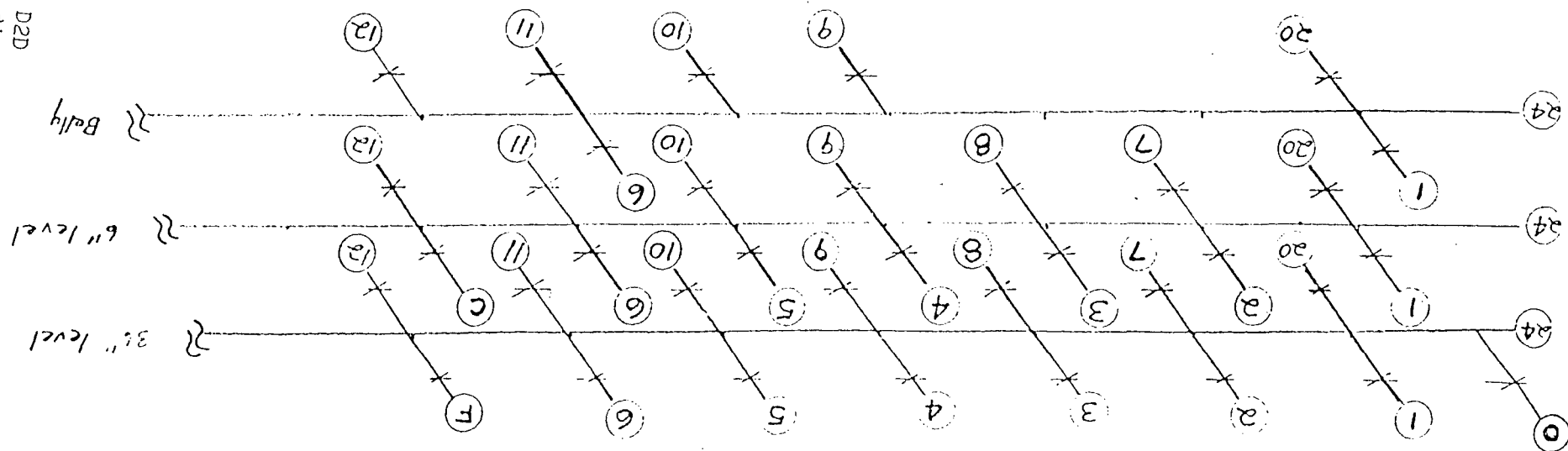


Figure D2C  
Process flow sheet

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Figure D2D  
piping diagram for tank Farm A



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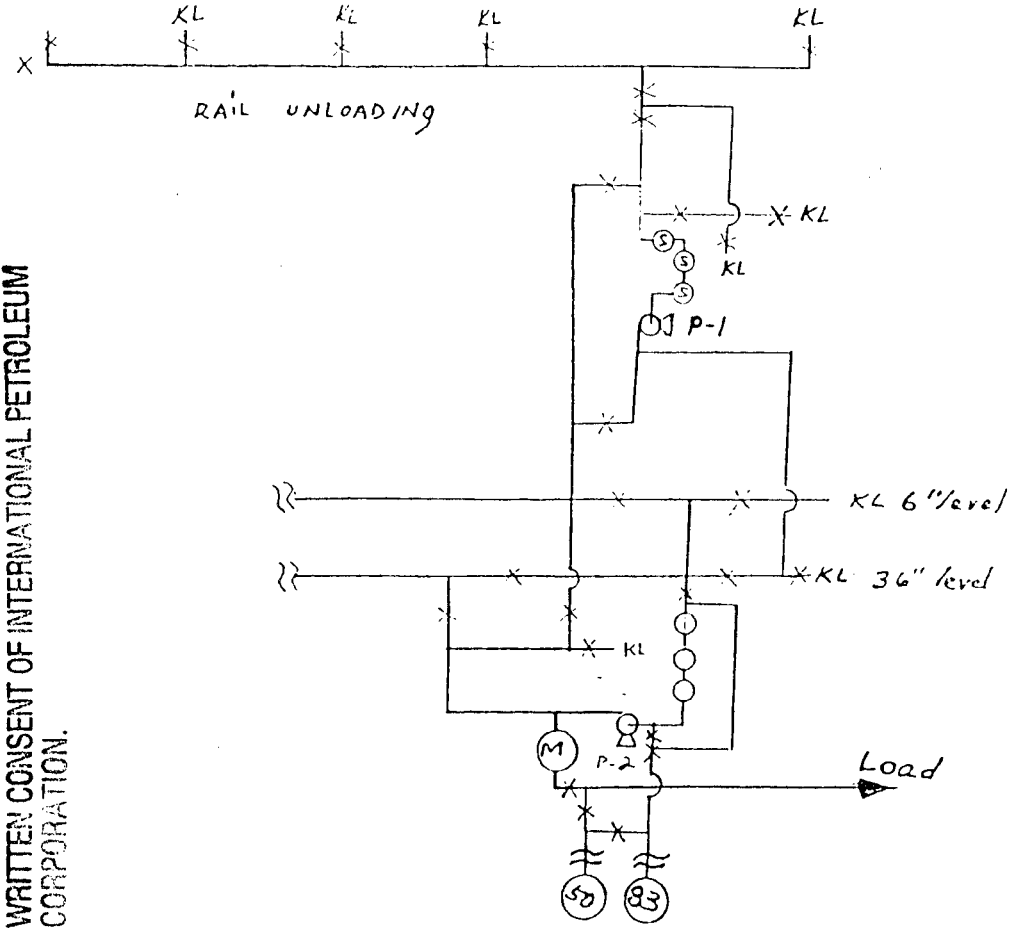


Figure D2E P&amp;ID -- Tank Farm A

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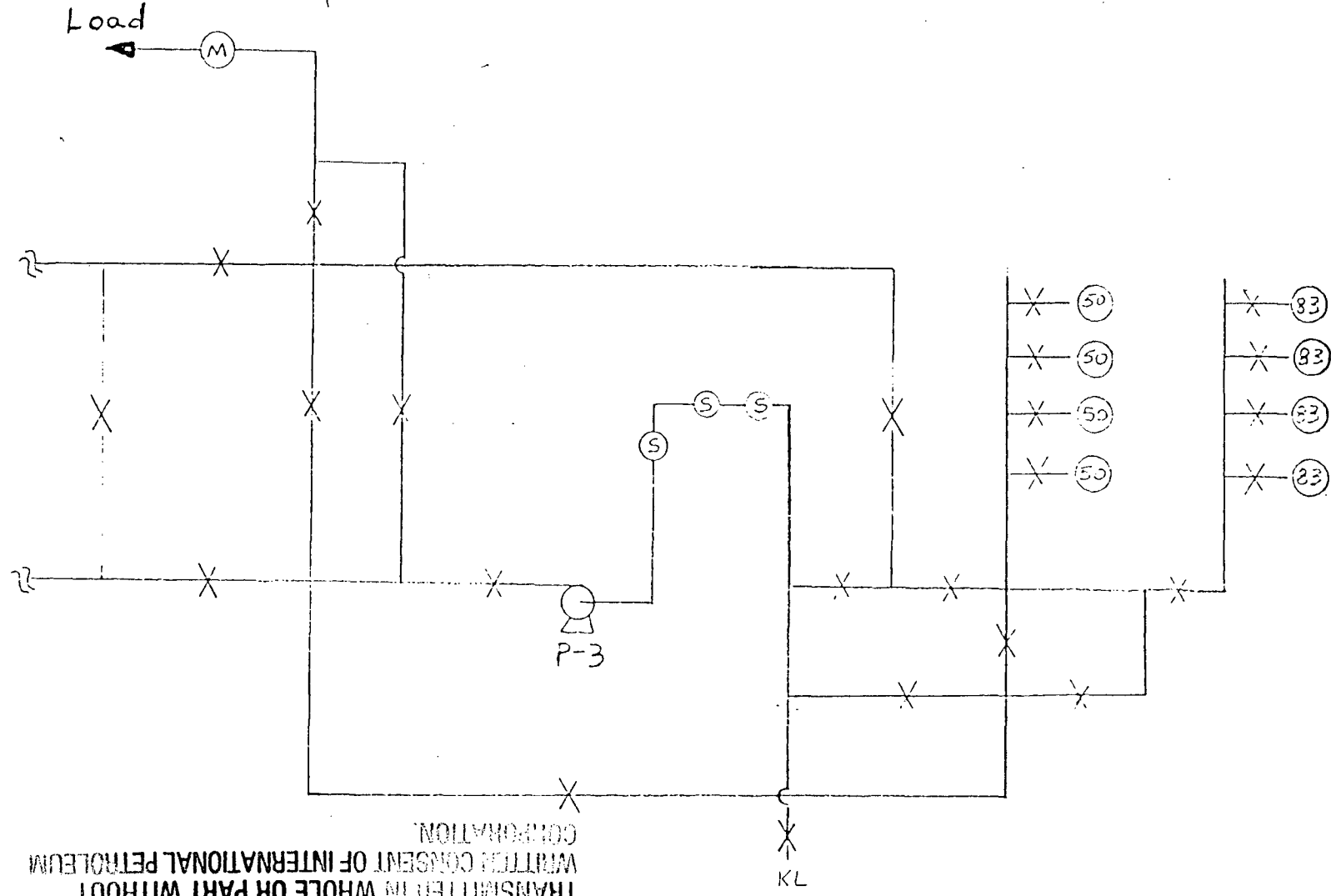


Figure D2F  
P & ID Tank Farm B

unlikely event. Under normal operations it never exceeds the half-filled condition. After the residual oil is separated from the water by an API separator, the water is directed to waste treatment. The separated oil is returned to the process. See Figure D2B for a schematic of the aqueous treatment system.

### 1.3 PROCESSING & BULK STORAGE TANKS

All tanks are either of riveted and welded or all welded steel construction. All are ultrasonically inspected at least annually and visually inspected daily and repaired as needed to prevent rupture or other failures. All tanks are more than 20 meters (65 feet) from the facility's property line.

All tanks and tank to tank connections are hard piped and comply with ASTM & API standards. Adequate valving provides isolation of tanks and/or hard piping in case of repairs, maintenance or leaking. All facilities are padlocked closed except when in service for oil transfer.

The areas around the tanks are diked to contain any releases that could occur as a result of overflow or rupture. Figure 5 lists potential release points, accident type, control and tank size. The volumetric capacity of each contained area is sufficiently large to contain the entire contents of the largest tank located in the respective area as well as to provide at least 10 - 12 inches of freeboard. The 10 - 12 inch freeboard allows for an abnormally heavy rainfall that could conceivably occur coincidentally. The freeboard is adequate to also protect releases from any wind or wave action. The dike height is 4 feet above grade. The diked area surrounds all tanks.

The base underlying all the tanks is concrete free from cracks with a central sump and a 4' high cement block wall surrounding the pad and is sufficient to contain all discharges and accumulated precipitation until the collected material is detected, processed and

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## TANK FARM B

Tank Farm B contains two (2) tanks. Each tank has a capacity of 210,000 gallons. Each tank has welded seams and meets API Standard Code 650 for oil storage. The tanks were ultrasonically tested before the tanks were placed in service. They were placed in service in 1983 and have a 50 year projected life.

The tanks are sited on concrete pads with four (4) foot high reinforced concrete walls capable of containing 25 percent of the volume of a tank. In the unlikely event of a tank rupture and spill the containment area will hold one forth of the volume of each of the two tanks. A 400 gpm pump is located in the contained area in addition to the two (2) 400 gpm pumps located in tank farm A. These pumps are capable of removing the remaining capacity of the ruptured tank in 2.2 hours.

Additionally, the site is sloped south-southeast. Any overflow of oil from the four foot high dike would flow toward tank farm A and could be pumped from the unloading sump. As an additional back up, the two percolation pond each can hold 125,000 gallons. This combined configuration assures no release ~~will leave the property~~ or be released to the environment.

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FIGURE 5 Potential Spills--Prediction and Control

<u>Source</u>	<u>Major Type of Failure</u>	<u>Maximum Spill Gallons</u>	<u>Secondary Containment</u>
Above ground tank 1 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 1 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 2 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 2 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 3 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 3 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 4 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 4 Vertical	Tank Rupture	15,000	Concrete Retaining Wall



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FIGURE 5 CONTINUED  
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Major Type of Failure		Maximum Spill	Secondary
Source	Failure	Gallons	Containment
Above ground tank 5 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 5 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 6 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 6 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 7 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 7 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 8 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 8 Vertical	Tank Rupture	15,000	Concrete Retaining Wall

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FIGURE 5 CONTINGENCY

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<u>Source</u>	<u>Major Type of Failure</u>	<u>Maximum Spill Gallons</u>	<u>Secondary Containment</u>
Above ground tank 9 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 9 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 10 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 10 Vertical	Tank rupture	15,000	Concrete Retaining Wall
Above ground tank 11 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 11 Vertical	Tank Rupture	20,000	Concrete Retaining Wall
Above ground tank 12 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 12 Vertical	Tank Rupture	20,000	Concrete Retaining Wall

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FIGURE 5 CONTINUED

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Source	Major Type of Failure	Capacity, Gallons	Containment
Above ground tank 13 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 13 Vertical	Tank Rupture	24,000	Concrete Retaining Wall
Above ground tank 14 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 14 Vertical	Tank Rupture	14,000	Concrete Retaining Wall
Above ground tank T-150 Vertical	Tank Rupture	210,000	Concrete Retaining Wall
Above ground tank T-83 Vertical	Tank Rupture	210,000	Concrete Retaining Wall
Total Capacity		648,000	

removed. The dike surrounding the treatment tank rises to a height  $4\frac{1}{2}$  feet above grade. It will accommodate the 24,000 gallon capacity of the tank again with enough freeboard (10% excess) to allow for an unusually heavy coincidental rainfall and eliminate release to the atmosphere by wind or wave action.

#### 1.4 PROCESS AND TRANSFER OPERATIONS

The operating design of the facility provides a closed hardline tank to tank and overflow system. Thus, discharges are virtually limited to tank rupture, pumping operations, piping or hose ruptures on pump suction side.

#### 1.5 TRUCK UNLOADING AND TANK CAR LOADING

Truck unloading into receiving day tanks is conducted via hose to the suction of Pump P-1 or P-2 and thence by hardline into the respective day tanks. Each day tank has a capacity of slightly more than 10,000 gallons. They are provided with an interconnecting overflow line. Maximum daily collections are not expected to ever exceed 25,000 gallons. Further, each previous day's collection is processed before subsequent collections are transferred into these receiving tanks. Consequently, overflow spills are not ever expected to occur.

Accidental releases during making or breaking of the hose connections to the trucks are controlled by drip troughs and/or containers positioned at the truck discharge site. Additionally, a special discharge hose line fitting is provided to permit air displacement of liquid in the hose line following emptying of the truck and closure of the truck discharge valve.

Prior to loading any tank car, a careful check is made to be sure that the tank car discharge valve is closed and the cap on the car's discharge line is securely in place and sealed.

Truck unloading and tank car loading are also continuously monitored by the plant operator to forestall any unexpected event that could result in an accidental release.

The base of the truck unloading area is concrete free of cracks or gaps. It is sloped to drain into a sump and operated to drain and remove liquids resulting from leaks, spills or precipitation. The sump has sufficient capacity to contain at least 10% of the volume of the largest containers unloaded. All spilled or leaked material and accumulated precipitation is removed from the sump in a timely fashion as necessary to prevent overflow. It is pumped into a tank and managed as a hazardous waste.

#### 1.6 OPERATING MAINTENANCE PROCEDURE

All valves permitting flow to or from the internal plant system are padlocked in the closed position when not specifically used for waste oil transfer operations. Furthermore, all the final outlets and hose connections are covered by dust caps which remain padlocked in place. It is mandatory for discharge free operation that all of the above valves and lines are unlocked immediately before use and immediately closed and locked when no longer in service.

All material transfers or product movement operations are continuously monitored by the plant operators or other appropriately trained personnel until such transfers or operations are completed. When unloading, all tank trucks are positioned so that the tank discharge valve is always over a drip-trough. This procedure catches any drippage when the dust covers are removed prior to connecting the pump suction hose to the tank truck outlet.

The drip-trough accumulations are never allowed to remain overnight. Unless required earlier, the pump of the last truck each day is used to empty the drip-trough accumulations during the time the truck is being transferred.

Upon completing the removal of oil from each truck, the truck discharge valve is closed and the air bleed valve on the suction line opened. The pump is then allowed to continue running for 1 or 2 minutes to empty the hose and pipe lines of any liquid transferred.

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Then the appropriate pump discharge valve is closed. Complete inspection of all fluid transfer lines and valves are made daily by the plant operator and the manager individually and at separate times. Any indication of leakage at joints valve bonnets, packing glands, etc. is corrected immediately.

Upon completion of transfers into tank cars, the liquid transfer valve on the suction side of the pump is closed. At the same time, the air bleed valve on the suction line is opened. The pump again is allowed to run for 1 or 2 minutes to clear the lines of any liquid. Then the appropriate pump discharge valve is closed and locked.

All valves not involved in any active transfer circuit must remain closed and locked. Careful check is made whenever a transfer circuit is changed, shut down or a new transfer circuit is started to be certain that only those valves involved in the circuit are opened and that all others are closed. This careful check is made each time before any pump is started. When pumping is finished all valves are locked closed after line clearance is completed.

Oil accumulated in the oil sump adjacent to the separator is pumped out daily or more often as required.

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## Solids/Oil/Water Separator

The solids/oil/water separator is a General Electric model VPS-4. It is a mild steel tank with coal tar epoxy coated surfaces and is unaffected by most corrosive chemicals.

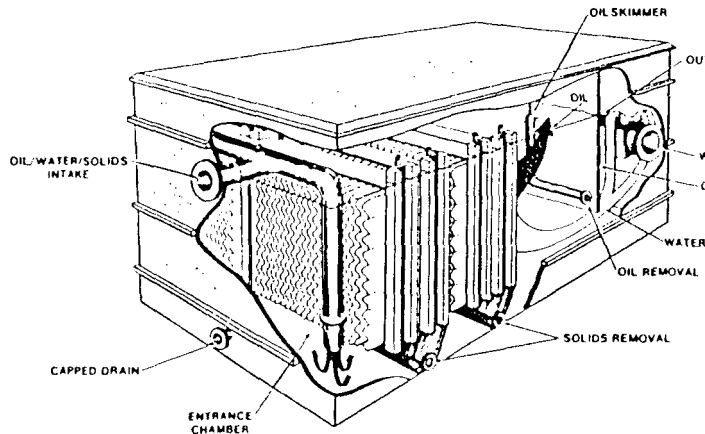
The plates are assembled in modules with one quarter inch spacing to handle high solids and oil content. The unit has two adjustable gravity-flow oil slummers designed for ten percent oil input concentrations. The unit can process 60-500 gpm at a nominal 125 gpm at a maximum oil content of effluent of less than 15 ppm. A description is found in Figure D2G.

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# How They Work

Figure D2G



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General Electric separators use gravity for the removal of solids and combine two processes for the removal of oil, gravity and the coalescing action of the plates.

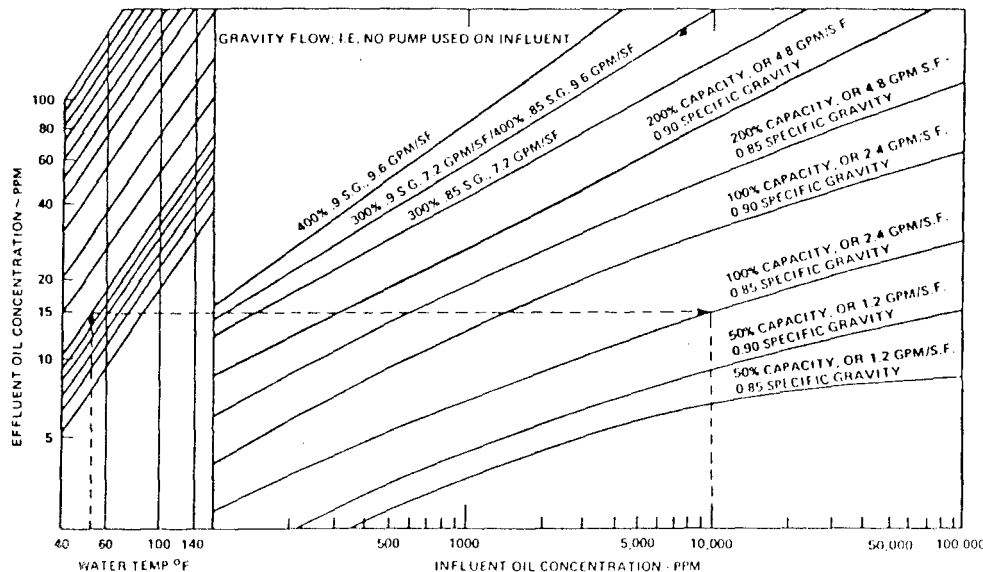
When the wastewater enters a GE separator, the flow rate is reduced to a range of 1 to 4 feet per minute. Most of the solids and oil (by volume) are deposited or removed in the entrance chamber. The remaining solids and oil uniformly enter the vertical plate section where solids and oil droplets as small as 5 microns are removed.

The plates provide laminar flow stability. This feature, plus the coalescing action of the plates, provides an ideal environment for gravity separation. The vertically oriented plates provide collection surfaces for the falling solids and the rising oil. The collected solids drop freely to the bottom of the hopper where they can be removed through drains. The oil coalesces on the plates, and, by gravity, flows to the surface.

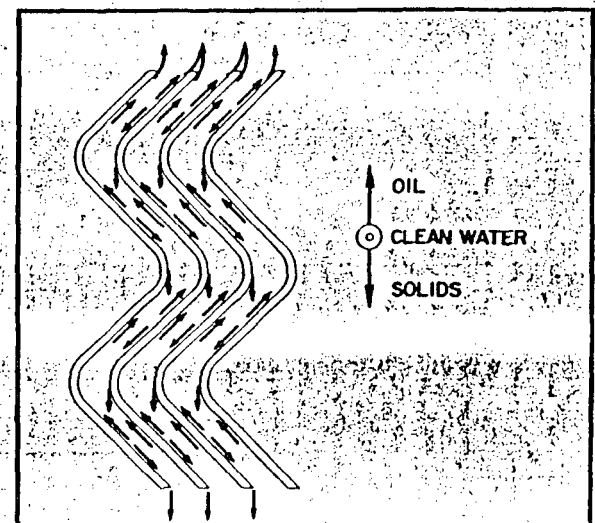
The plates are corrugated to provide a large surface area per unit volume for increased separator efficiency. The plates can process wastes at temperatures to 212°F, withstand pH levels from 2 to 12, and are unaffected by most common mineral acids.

Solids/Oil/Water Separator

Influent VS Effluent Oil Concentration



Solids/Oil Removal Flow





## DEMETALLIZATION

The process re-refines used oil by first chemically precipitating and physically filtering the metal compounds from the oil. The demetallized and deashed oil is then blended to Federal Burner Fuel Specification VV-F-815C. The following steps will be carried out in sequence. These are separate steps. The feed stock product from the demetallization section is essentially pure hydrocarbon stock free from metal and solvent adulteration.

A detailed process design schematic is given in Drawing No. 1. As shown in Drawing 1, the used oil is unloaded into a sump and transferred by pump to the waste oil feed tank. The waste oil is pumped from the feed tank, to the first of three demetallizing reactors.

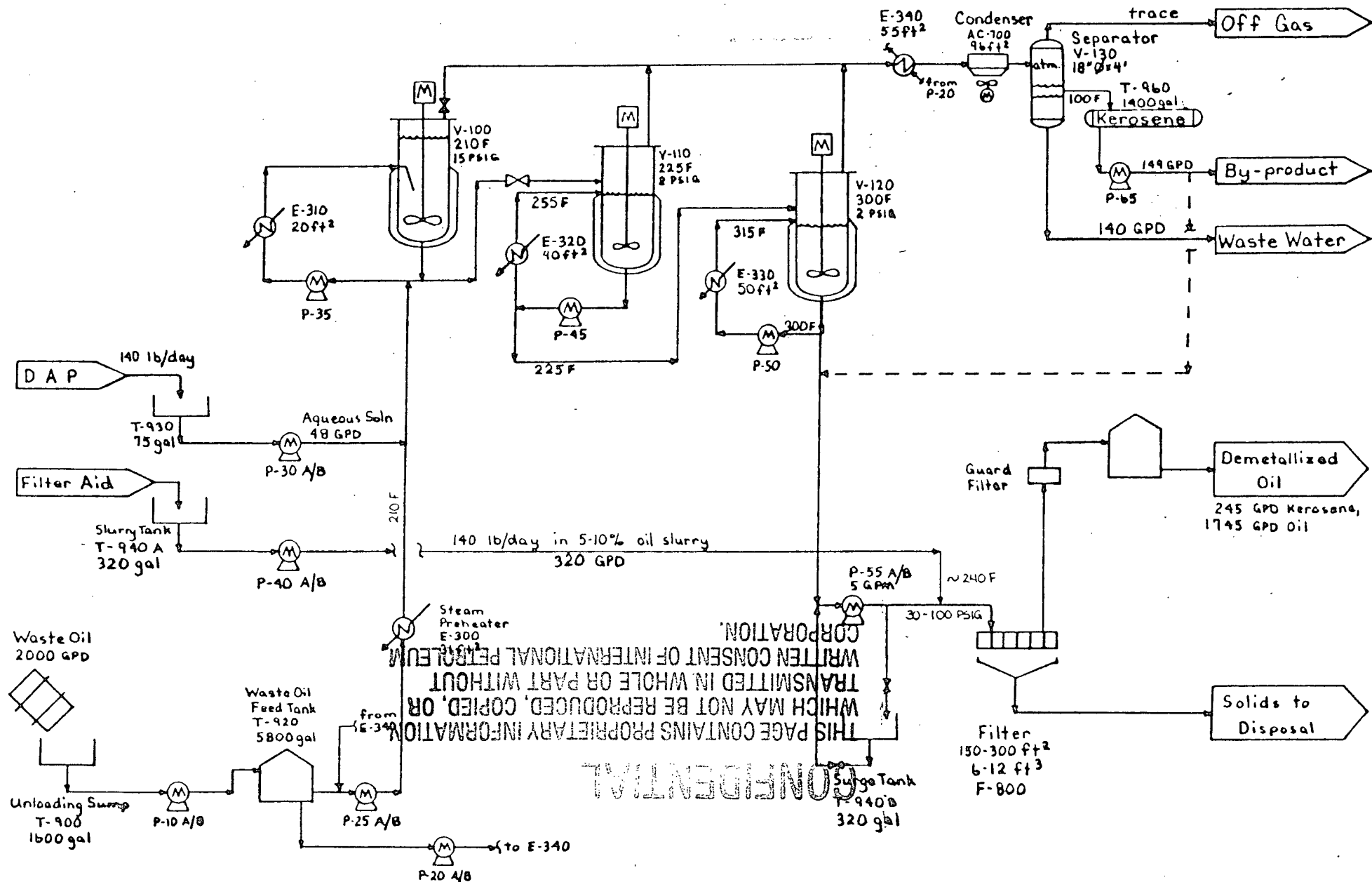
This raw waste oil contains water, a kerosene type material which is a dilution from gasoline and #2 diesel oil, carbon particles and dissolved metal compounds. These metal compounds come from the motor fuel (lead) or were added to the fresh lube oil as detergents, dispersants, anti-wear agents, anti-oxidants or rust inhibitors (calcium, barium, lead, zinc). The metal portions of these organo-metal compounds are precipitated as insoluble phosphates by reacting the hot oil with an aqueous ammonium phosphate. The required amount varies from 0.5 to 1.5 weight per cent of the waste oil feed. It is proportional to the ash content of the waste oil. The original water in the oil, which varies considerably, (but is generally in the 5 to 10 weight per cent range, plus the 2 to 3 weight per cent added with the DAP solution) is boiled out of the oil between 225 to 300°F and about 1 or 2 atmospheres of pressure #2 and #3. At the same time part of the diluent kerosene in the waste oil (usually in the 5 to 15 weight per cent range) is also boiled off. The steam and kerosene vapors are then condensed. The solids originally present in the waste oil are removed by filtration after adding about 1 weight per cent diatomaceous earth (D.E.) as a filter aid. The oil is filtered at about 300° F. This demetallizing step reduces the ash content of the oil feed which is normally in the 1 to 2 weight per cent range to less than 0.1 weight per cent ash. The oil will

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spend about one hour and forty-five minutes in this process step and will be demetallized to less than 50 ppm lead, 1 ppm arsenic, chromium and cadmium.

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Drawing 1 Demetallizing Section  
 (Flows are for 8 hr. day)

## WASTE ANALYSIS PLAN

Each lot of material considered by I.P.C. is analyzed whether it is utilized or not. Further each lot disposed of by the facility is analyzed to characterize it. All lots are analyzed according to Mil-F-859E.

## LABORATORY ANALYSIS

Laboratory analyses for organic and inorganic constituents are conducted for all major process and waste streams associated with the recycling including both the fuel feed and processed oils. Generalized schematics of the characterizations performed are shown in Figure A-1. Selected properties of the representative oils are determined either in-house or by appropriate subcontractors. The analytical protocols used are provided in the following pages. These analytical methods are specific to the instrumentation, reagents and procedural modifications are routinely used in the process and waste characterization. A summary of test methods occurs in Tables 1 and 2. A Company Buying Standard occurs in Table 3.

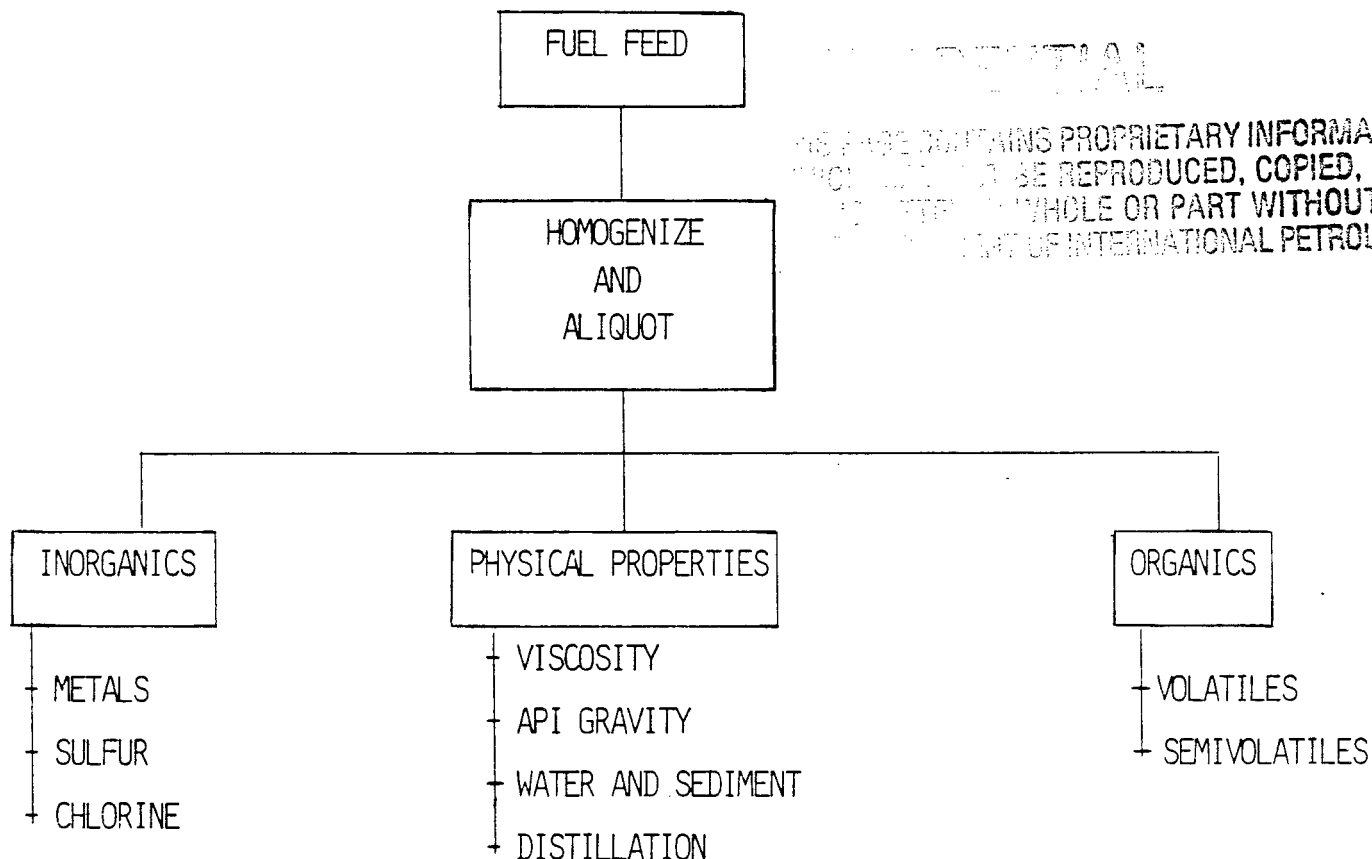


Figure A-1. Oil characterization.

Table 1. Test Method

<u>TEST</u>	<u>ASTM*</u>
Flash Point	D93
Pour Point	D97
Water and Sediments (1-4 grade fuels)	D1796
Water (5 & 6 grade fuels)	D95
Viscosity	D86 or D445
Gravity, API	D287
Sulfur	D1552, D129, D1551 or D2662

Table 2. Summary of oil analyses

<u>Property of Test</u>	<u>Oil</u>
Viscosity (SUS @ 40° C)	90 - 850
API Gravity (60° F)	10 - 34
Water % by Volume	0 - 10
BS & W % by Volume	0 - 11
Flashpoint (°F)	140 - 700
Heating Value (BTU/lb)	13000 - 19600
Chloride (% by wt)	< 0.1
Sulfur (% by wt)	0.1 - 2.1
Zinc ppm	10 - 1089
Chromium ppm	0 - 20
Calcium ppm	2 - 2200
Barium ppm	tr - 3650
Phosphorous ppm	0 - 50
Silica ppm	20 - 90
Iron ppm	tr - 1200
Aluminum ppm	tr - 150
Lead ppm	0 - 1000
Vanadium ppm	4 - 150

\* Analysis methods for organics and metals used by I.P.C. are found in 40 CFR 261 Appendix III.

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#### WASTE GENERATION

Wastes that are generated at the facility are the result of washing the outside surface of truck-tractor units, empty tank trailer and other equipment; precipitation and residual water dropped from oil lots. Occasionally the interior of a tank trailer may be washed, but it is rarely done on site. Normally it is done by an outside cleaning contractor. The resulting water is collected at a sump which is contained within a concrete pad. It is then pumped to a steel tank for storage and processed through an API oil/water separator and disposed of after analysis to assure compliance with all existing industrial waste water criteria. In all cases, the identity of material that may be contained is known. Characteristics of the waste water are known on the basis of waste, source sampling and on the basis of manifests which accompany the transportation of the materials.

Since the waste water is mostly water, it is normally within acceptable discharge limits. In the unlikely event it is not, it is treated, solar evaporated or shipped to a hazardous waste disposal site properly equipped to handle the waste.

#### RECORD KEEPING AND ADMINISTRATION

Careful records including bills of lading, manifest and sources and identities of wastes are maintained. Sample and analysis results and a list of contaminants and their source are included with each shipment.

#### NEW LABORATORY

International Petroleum Corporation is presently building a new analytical facility to include automated GC, GCMS and a plazma emission. This will replace and / or compliment the analyses presently being done by P. E. La-Moreaux, Caleb Brett and Thornton Labs. By mid - 1985 all analytical work will be done in house.

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#### SAMPLING METHOD

Prior to disposal, representative samples of waste water are removed from the tank at various levels. These are then combined as a composite. Compatibility of the waste water is then determined. Its reaction or evolution of gas resulting from the addition of acid to a portion of the sample of pH less than 1 and the addition of soda ash to another portion of the sample until the pH of the mixture is approximately 13 is determined. On the basis of these observations; already knowing the chemical characteristics of the water, sufficient information regarding the waste is obtained for proper treatment and disposal.

#### FREQUENCY OF ANALYSIS

The contents of each storage tank are subjected to analytical tests and observations prior to disposal. Bench scale tests are performed each time a lot of waste water is disposed.

#### PARAMETERS ANALYZED

Prior to disposal, the following observations and analyses are made on the waste water:

1. Water Solubility
2. Flashpoint
3. Odor
4. Color
5. Number of Phases
6. TSS and TDS
7. Viscosity
8. Volume
9. pH, Temp., Conductivity
10. Reactivity with Air and Water
11. Oil and Grease
12. Heavy Metals including Lead and Cadmium
13. Pesticides/herbicides including Lindane, Methoxychlor, Toxaphene, 2, 4D, 245TP Silvex and Endrine

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## BURNER FUEL OIL SPECIFICATIONS

Fuel oils found in commerce exhibit a range of values for each specified property, as do the recycled oils which may be used separately or blended with them. The first general set of fuel oil classifications in the U.S. was issued by the National Bureau of Standards in 1929. They were the first industrywide attempt to classify fuels according to significant properties. These standards, based on a joint conference of consumers, burner manufacturers, and general interest groups held on January 9, 1929, were issued as Commercial Standard (CS) 12-29. This standard was revised in 1933, 1934, 1938, and 1940, with the final revision issued in 1948 as CS 12-48, and remained in effect until 1966, at which time it was withdrawn. These first standards established the presently used fuel oil grades.

At the present time, the following sets of specifications are, or in the recent past have been, used for burner fuel oils:

ASTM D 396-76, Standard Specification for Fuel Oils

VV-F-815C, Federal Specification, Fuel Oil, Burner

MIL-F-859E, Military Specification, Fuel Oil, Burner

These specifiers are the primary specifications in current use by IPC and are summarized in Table 4.

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TABLE 4. SUMMARY OF SPECIFICATIONS CONTAINED IN  
ASTM METHOD NO. D 396.

Test procedures	Fuel oil grades (number)					
	1	2	4	light <sup>5</sup>	heavy <sup>5</sup>	(Bunker C) <sup>6</sup>
Flash point, °F (°C)	100 (38) or legal <sup>a</sup>	100 (38) or legal	130 (55) or legal	130 (55) or legal	130 (55) or legal	140 (60) or legal
Pour point, °F (°C)	0 (-18)	20 (-7)	20 (-7)	--	--	--
Water and sediment, volume %	0.05	0.05	0.50	1.00	1.00	2.00
Carbon residue on 10% bottoms weight %	0.15	0.35	--	--	--	--
Ash, weight %	--	--	0.10	0.10	0.10	--
Distillation temp. °F (°C)						
10% point (max)	420 (215)	--	--	--	--	--
90% point (min)	--	540 (282)	--	--	--	--
(max)	550 (288)	640 (338)	--	--	--	--
Kinematic viscosity cSt						
At 100 °F (38 °C)						
Minimum	1.4	2.0	5.8	>26.4	>65	--
Maximum	2.2	3.6	26.4	65	194	--
At 122 °F (50 °C)						
Minimum	--	--	--	--	(42) <sup>b</sup>	92
Maximum	--	--	--	--	(81)	638
API gravity, degrees, (min)	35	30	--	--	--	--
Copper strip corro- sion (max) (ASTM specifications at 50° C; Federal speci- fications at 100 °C)	No. 3	No. 3	--	--	--	--
Sulfur, weight % (max)	0.5 or legal	0.5 or legal	legal	legal	legal	legal
Aniline point °F (°C) (Federal specifications only)	report	report				

<sup>a</sup> "Legal" refers to applicable laws or regulations setting maximum sulfur levels and minimum flash point levels.

<sup>b</sup> Viscosity values in parentheses are for information only and not necessarily limiting.

#### CHARACTERISTICS OF RECYCLED OILS USED AS BURNER FUEL

Used petroleum oils recycled for use as burner fuel oils are obtained from a variety of sources. These sources include automotive garages and service stations, truck and taxi fleets, military installations, industrial plants and manufacturing facilities of all types, and waste-water treatment plants. The major types of used petroleum oils which are recycled into burner fuel oils are listed in Table 5. Also listed are some of the additives and contaminants which are typical of these oils.

In addition to the additives and contaminants listed in Table 5, there have been many analyses of used oils and waste oils, primarily for inorganic constituents. Reported analyses are found in Table 6.

Those values determined by IPC for organic and inorganic are found in Table 6A and 6B. References are provided to establish the analytical validity of parameters both analyzed for and methods used. The actual procedures used by IPC or independent consultants are also described in detail.

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TABLE 5. LUBRICATING OIL ADDITIVES AND CONTAMINANTS  
LIKELY TO BE FOUND IN USED OILS

<u>Used oil type</u>	<u>Typical kinds of additives</u>	<u>Primary source &amp; type of typical contaminants</u>
Motor oils	<u>Antiwear</u> : zinc organo-dithiophosphates; organic phosphorus compounds.	<u>Gasoline</u> : lead and lead compounds; halogens.
	<u>Corrosion inhibitors</u> : sulfonates; amine phosphates; organic phosphites.	<u>Additives</u> : zinc and zinc compounds; phosphorus compounds, etc.
	<u>Detergents and dispersants</u> : calcium, magnesium, barium, and zinc sulfonates, phosphonates, and phenates; alkenyl succinimides; acrylic polymers.	<u>Combustion products</u> : polynuclear aromatics; water; oxygenated compounds; sludge; varnish.
	<u>Viscosity index improvers</u> : isobutylene polymers; acrylate polymers.	<u>Fuel</u> : volatile liquid.
	<u>Pour-point depressants</u> : polymethacrylates; polyacrylamides.	<u>Contamination</u> : dirt; wear metals.
	<u>Antifoam agents</u> : silicones; synthetic polymers.	<u>Coolant leaks</u> : water, ethylene
Hydraulic and industrial oils	<u>Antioxidants</u> : zinc organo-dithiophosphates; hindered phenols; aromatic amines; sulfurized phenols.	
	Many of the above plus:	Many of the above plus:
	<u>Extreme pressure additives</u> : organosulfur compounds; organochlorine compounds; lead compounds; amines.	<u>Additives</u> : sulfur and sulfur compounds; chlorine and chlorinated compounds; fatty oils.
	<u>Emulsifiers</u> : surfactants; sulfonates; naphthenates; fatty acid soaps.	<u>Contamination</u> : metal particles.
	<u>Antiseptics</u> : alcohols; phenols; chlorine compounds.	<u>Solvents</u> : volatile liquid

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TABLE 6. SUMMARY OF REPORTED USED OIL ANALYSES (4-15)<sup>a</sup>

Property or test	Motor oils	Industrial oils <sup>b</sup>
Viscosity (SUS at 40 °C) <sup>c</sup>	87 to 837	143 to 330
API gravity (60 °F)	19.1 to 31.3	25.7 to 26.2
Water (% by volume) <sup>h</sup>	0.2 to 33.8 <sup>d</sup>	0.1 to 4.6
BS&W (% by volume)	0.1 to 42	NA <sup>e</sup>
Benzene insolubles (% by weight)	0.56 to 3.33	NA
Gasoline dilution (% by volume)	2.0 to 9.7	NA
Flash point (°F)	175 to 427	315 to 355
Heating value, Btu/lb	13,571 to 19,300	17,250 to 17,990
Ash, sulfated (% by weight)	0.03 to 6.43	3.2 to 5.9 <sup>f</sup>
Carbon residue (% by weight)	1.82 to 4.43	NA
Fatty oils (% by weight)	NA	0 to 60
Chlorine (% by weight)	0.17 to 0.47	< 0.1 to 0.83
Sulfur (% by weight)	0.17 to 1.09	0.54 to 1.03
Zinc (ppm) <sup>g</sup>	260 to 1,787	NA
Calcium (ppm)	211 to 2,291	NA
Barium (ppm)	9 to 3,906	NA
Phosphorus (ppm)	319 to 1,550	NA
Lead (ppm)	85 to 21,676	NA
Aluminum (ppm)	< 0.5 to 758	NA
Iron (ppm)	97 to 2,401	NA

<sup>a</sup> Most references did not provide data on all tests listed; therefore, data may be inconsistent between different tests.

<sup>b</sup> Very limited data were available for used industrial oils, on only 5 oils (4).

<sup>c</sup> SUS = Saybolt Universal Seconds

<sup>d</sup> One sample had a water content of 46.5 percent, but is considered an outlier.

<sup>e</sup> NA = data not available.

<sup>f</sup> Values for the industrial oils were stated to be for the regular ash, not sulfated.

<sup>g</sup> ppm (as the element) = 0.0001 weight percent or 10<sup>4</sup> ppm = 1 weight percent.

<sup>h</sup> BS&W = Bottom Settlements and Water

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TABLE 6A. DISPOSITION OF ELEMENTAL CONTAMINANTS IN PROCESS STREAMS

	Feedstock	Dematallized oil	Product oil
<u>Contaminant Weight</u> <u>(ug/g of feed)</u>			
Aluminum	8.3	<0.2	<0.1
Antimony	<0.3	1.0	<0.3
Arsenic	1.7	1.6	<0.5
Barium	150	2.2	0.06
Beryllium	<0.04	<0.01	<0.01
Boron	6.6	2.8	0.09
Cadmium	0.8	0.05	<0.02
Calcium	610	14	1.5
Chromium	3.4	<0.06	<0.06
Cobalt	0.2	<0.06	<0.06
Copper	13	2.3	<0.03
Iron	110	5.2	2.6
Lead	2,060	180	3.5
Magnesium	270	1.3	0.2
Manganese	13	0.04	0.08
Molybdenum	1.4	0.3	<0.03
Nickel	0.6	<0.1	<0.09
Selenium	<0.4	<0.4	<0.3
Silicon	22	<0.1	<0.09
Silver	<0.04	<0.02	<0.02
Sodium	59	89	<0.2
Strontium	0.1	<0.02	<0.02
Thallium	<1.7	<0.8	<0.7
Tin	2.2	2.1	<0.7
Titanium	2.2	0.02	<0.02
Vanadium	<0.2	<0.1	<0.09
Zinc	630	3.3	0.3

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TABLE 6B. DISPOSITION OF ORGANIC CONTAMINANTS IN PROCESS STREAMS

	Feedstock	Demetallized oil	Product oil
<u>Contaminant Weight</u> <u>(ug/g of feed)</u>			
Volatiles			
Trichlorofluoromethane	<20	<19	<17
Trichlorotrifluoroethane	<20	<19	<17
Methylene chloride	<20	<19	<17
1,1-Dichloroethane	<20	<19	<17
1,2-Dichloroethane	<20	<19	<17
t-1,2-Dichloroethylene	<20	<19	<17
Chloroform	<20	<19	<17
1,1,1-Trichloroethane	<20	<19	<17
Trichloroethylene	<20	<19	<17
Carbon tetrachloride	<20	<19	<17
Tetrachloroethylene	<20	<19	<17
Benzene	90	<19	<16
Toluene	670	260	160
Ethylbenzene	160	150	70
Semivolatiles			
Chlorobenzene	<20	<19	<17
Phenol	<10	<10	<10
Chlorophenol	<10	<10	<10
Dichlorobenzene	<10	<10	<10
Nitrobenzene	<10	<10	<10
Nitrophenol	<10	<10	<10
Naphthalene	280	210	95
2-Chloronaphthalene	<10	<10	<10
2,4,6-Trichlorophenol	<10	<10	<10
Acenaphthene	<10	<10	<10
N-nitrosodiphenylamine	<10	<10	<10
Hexachlorobenzene	<10	<10	<10
Phenanthrene/anthracene	130	110	71
Pyrene	<20	<15	<15
Benz(a)anthracene	<12	<10	<5
Benzo(a)pyrene	<10	<16	<6
4,4'-DDE	<20	<19	<17
PCBs	<1	<1	<1

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## INORGANIC ANALYSIS PROCEDURES

The procedures used for the determination of metals in oil, total chlorine and sulfur in oils are described in the following pages.

### PROCEDURE

Dry ashing procedure for the determination of metals in petroleum products

General ICAP method for trace element analysis

Sulfur in petroleum products

Chlorine in petroleum products

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## DRY ASHING PROCEDURE FOR THE DETERMINATION OF METALS IN PETROLEUM PRODUCTS

### SCOPE AND APPLICATION

This procedure may be used to prepare petroleum products for trace element analysis by AA or ICAP.

### SUMMARY OF METHOD

A 2-5 g portion of oil is ashed and the residue is taken up in nitric and hydrochloric acids. The resulting sample can then be analyzed by AA or ICAP for trace element content.

### REAGENTS

1. Baker "Instra-Analyzed"  $\text{HNO}_3$  and  $\text{HCl}$

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### APPARATUS

1. Muffle furnace
2. IR lamps
3. Platinum Crucibles, 15-ml capacity

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### PROCEDURE

1. Place 2-5 g of oil in a platinum crucible.
2. Place the sample under an IR lamp with the lamp at its highest position. Lower the lamp position gradually, do not allow the sample to boil. Leave the lamp at its lowest position until the sample appears ashed.
3. To complete ashing, place the sample in the muffle furnace and slowly increase the temperature (every half hour) from 100-600°C.
4. When the sample appears completely ashed, wash the ash into a 150 ml beaker using 2-3 ml 1:1  $\text{HNO}_3$ . Wash the platinum crucible with several portions of 1:1  $\text{HNO}_3$  and add the washings to the beaker. Continue this until approximately 10 ml of 1:1  $\text{HNO}_3$  has been added to the beaker. Rinse the crucible with several portions of de-ionized water and add the rinsings to the beaker. Add 2 ml of concentrated  $\text{HCl}$  and heat the sample gently for 15 minutes. Cool, transfer to a 100ml volumetric flask and dilute to volume. The sample may now be analyzed by AA or ICAP.

## GENERAL ICAP METHOD FOR TRACE ELEMENT ANALYSIS

### SCOPE AND APPLICATION

This method is designed to detect and quantify 27 elements in water and wastewater using Inductively Coupled Plasma. The elements detected by this procedure are shown in Table D4 .

TABLE D4 . DETECTION LIMITS AND WAVELENGTHS FOR ICP ANALYSIS

Element	Wavelength (nm)	Detection limit (ug/l)
Ag	328.0	1.0
Al	308.2	9.0
As	197.2	32
B	249.6 (second order)	4.0
Ba	493.4	0.6
Be	234.8	0.4
Ca	317.9	10
Cd	228.8 (second order)	0.8
Co	228.6	3.0
Cr	205.5 (second order)	2.6
Cu	324.7	1.6
Fe	259.9	3.4
Mg	279.0	20
Mn	257.6	0.4
Mo	202.0	2.2
Na	589.0	10
Ni	231.6 (second order)	4.8
Pb	220.3	16
Sb	206.8	15
Se	196.0 (second order)	22
Si	251.6	5.0
Sn	189.9	30
Sr	421.5	1.0
Ti	334.9	1.2
Tl	190.8 (second order)	41
V	292.4	5.4
Zn	213.8	0.8

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## INTERFERENCES

A variety of interferences may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

- Special interferences
- Physical interferences
- Chemical interferences

It is recommended that whenever a new or unusual sample matrix is encountered, the following tests should be performed:

- Serial Dilution--If the analyte concentration is at least a factor of 10 above the instrumental detection limit after dilution, an analysis of a dilution should agree within 5 percent of the original determination. If the values do not agree within 5 percent, a chemical or physical interference should be suspected.
- Spike Addition--the recovery of a spike addition added at a minimum level of 10X the instrumental detection limit should be recovered to within 90 to 110 percent. If not, a matrix effect should be suspected. The use of the method of standard addition will usually compensate for matrix effects. However, this technique does not detect coincident spectral overlap. If this is suspected, use of computerized compensation or comparison with an alternate method is recommended.
- Comparison with Alternate Method of Analysis-- Comparison with another analytical technique, such as Atomic Absorption Spectrophotometry may be helpful in detecting interferences.

## REAGENTS

1. Acids--All acids should be either J.T. Baker Ultrex or Instra-Analyzed grade.
  - a. Acetic acid
  - b. Hydrochloric acid, concentrated and (1+1). Prepare (1+1) HCl by adding 500 ml concentrated HCl to 400 ml deionized water and diluting to 1 liter.

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- c. Nitric acid, concentrated and (1+1). Prepare (1+1) nitric acid by adding 500 ml concentrated  $\text{HNO}_3$  to 400 ml deionized water and diluting to 1 liter.
2. Stock standard solutions, 1,000 ug/ml
- a. Aluminum--Dissolve 1.000 g of aluminum metal in an acid mixture of 4 ml of (1+1) HCl and 1 ml of conc.  $\text{HNO}_3$  in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask, add an additional 10 ml of (1+1) HCl and dilute to 1,000 ml with deionized water.
- b. Antimony--Dissolve 2.669 g K (SbO)  $\text{C}_4\text{H}_4\text{O}_6$  in deionized water, add 10 ml (1+1) HCl and dilute to 1,000 ml with deionized water.
- c. Arsenic--Dissolve 1.320 g  $\text{As}_2\text{O}_3$  in 100 ml of deionized, distilled water containing 0.4 g NaOH. Acidify the solution with 2 ml conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.
- d. Barium--Dissolve 1.516 g  $\text{BaCl}_2$  (dried at  $250^\circ\text{C}$  for 2 hrs) in 10 ml deionized water with 1 ml (1+1) HCl. Add 10.0 ml (1+1) HCl and dilute to 1,000 ml with deionized water.
- e. Beryllium--Dissolve 1.000 g of beryllium metal in a minimum volume of (1+1) HCl. Dilute to 1 liter with 1 percent (v/v) HCl.
- f. Boron Solution--Do not dry. Dissolve 5.716g anhydrous  $\text{H}_3\text{BO}_3$  in deionized water and dilute to 1,000 ml. Use a reagent meeting ACS specifications, keep the bottle tightly stoppered and store in a desiccator to prevent the entrance of atmospheric moisture.
- g. Cadmium--Dissolve 1.142 g CdO in a minimum volume of (1+1)  $\text{HNO}_3$ . Heat to increase rate of dissolution. Add 10.0 ml conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.
- h. Calcium--Suspend 2.498 g  $\text{CaCO}_3$ , which was dried at  $180^\circ\text{C}$  for 1 hr before weighing, in deionized water and dissolve cautiously with a minimum amount of (1+1)  $\text{HNO}_3$ . Add 10.0 ml conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.

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- i. Chromium--Dissolve 3.735g of potassium chromate in 1 liter of deionized water.
- j. Cobalt--Dissolve 1.000 g of cobalt metal in a minimum volume of (1+1)  $\text{HNO}_3$ . Add 10.0 ml (1+1)  $\text{HCl}$  and dilute to 1,000 ml with deionized water.
- k. Copper--Dissolve 1.252 g  $\text{CuO}$  in a minimum volume of (1+1)  $\text{HNO}_3$ . Add 10.0 ml conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.
- l. Iron--Dissolve 1.430 g  $\text{Fe}_2\text{O}_3$  in a warm mixture of 20 ml (1+1)  $\text{HCl}$  and 2 ml of conc.  $\text{HNO}_3$ . Cool, add an additional 5 ml of conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.
- m. Lead--Dissolve 1.599 g  $\text{Pb}(\text{NO}_3)_2$  in a minimum amount of (1+1)  $\text{HCl}$  and 2 ml of conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.
- n. Magnesium--Dissolve 1.658 g  $\text{MgO}$  in a minimum volume of (1+1)  $\text{HNO}_3$ . Add 10.0 ml conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.
- o. Manganese--Dissolve 1.000 g of manganese metal in a mixture of 10 ml conc.  $\text{HCl}$  and 1 ml conc.  $\text{HNO}_3$ , and dilute to 1,000 ml with deionized water.
- p. Molybdenum--Dissolve 2.043 g  $(\text{NH}_4)_2\text{MoO}_4$  in deionized water and dilute to 1,000 ml.
- q. Nickel--Dissolve 1.000 g of nickel metal in 10 ml hot conc.  $\text{HNO}_3$ , cool and dilute to 1,000 ml with deionized water.
- r. Selenium--Do not dry. Dissolve 1.727 g  $\text{H}_2\text{SeO}_3$  (actual assay 94.6%) in deionized water and dilute to 1,000 ml.
- s. Silicon--Fuse 2.139 g of silicon dioxide with 8 g of sodium hydroxide until a clear melt is obtained. Cool, dissolve the cake in 100 ml of 1:3 hydrochloric acid and make up to 1 liter.
- t. Silver--Dissolve 1.575 g  $\text{AgNO}_3$  in 100 ml of deionized water and 10 ml conc.  $\text{HNO}_3$ . Dilute to 1,000 ml with deionized water.
- u. Sodium--Dissolve 2.542 g  $\text{NaCl}$  in deionized water. Add 10.0 ml conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.



- v. Strontium--Dissolve 1.685g  $\text{SrCO}_3$  in 10 ml 1:1  $\text{HNO}_3$  and dilute to 1 liter with deionized water.
  - w. Thallium--Dissolve 1.303 g  $\text{TlNO}_3$  in deionized water. Add 10.0 ml conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.
  - x. Tin--Dissolve 1.000g of tin in 100 ml  $\text{HCl}$  (warm to  $60^\circ$  if necessary). Cool and dilute to 1 liter with (1+9)  $\text{HCl}$ .
  - y. Titanium--Dissolve 1.000g of titanium metal in 100 ml of (1+1)  $\text{HCl}$ . Cool and dilute to liter with (1+1)  $\text{HCl}$ .
  - z. Vanadium--Dissolve 2.297 g  $\text{NH}_4\text{VO}_3$  in a minimum amount of conc.  $\text{HNO}_3$ . Heat to increase rate of dissolution. Add 10.0 ml conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.
  - aa. Zinc--Dissolve 1.245 g  $\text{ZnO}$  in a minimum amount of dilute  $\text{HNO}_3$ . Add 10.0 ml conc.  $\text{HNO}_3$  and dilute to 1,000 ml with deionized water.
3. Mixed Calibration Standard Solutions--Prepare by combining appropriate volumes of the stock solutions in volumetric flasks. The final acid concentration should be 2 percent  $\text{HNO}_3$ . Prepare mixed standard V in a polyethylene flask. Mixed calibration standards are prepared fresh daily.

The following mixed calibration standards are normally prepared:

- Mixed Calibration Standard I--Blank, 2%  $\text{HNO}_3$
- Mixed Calibration Standard II--Fe, Al, Ba, Ni, Cr, Ti
- Mixed Calibration Standard III--Mg, Cu, Sb, As, Co, Pb, Mo, Se, Si, Tl, Be
- Mixed Calibration Standard IV--Ca, Cd, Ag
- Mixed Calibration Standard V--V, Sn, Sr, Mn, Zn, Na, B

#### APPARATUS

1. Jarrell-Ash Model 855 Atom Comp ICP
2. Liquid Argon
3. Beakers
4. Pipets
5. Volumetric flasks

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6. Hot plates
7. Watch glasses

#### QUALITY CONTROL REQUIREMENTS

Each group of 20 or fewer samples carried through the preparation and analysis procedures must include a blank, a spiked sample prepared from an EPA trace metals concentrate, and a duplicate.

After calibrating the instrument, analyze an EPA trace elements concentrate to verify the preparation of the mixed calibration standards. Enter the reported values in the instrument logbook.

#### PROCEDURE

1. Set up the instrument as specified in the Jarrell-Ash operating manual. Allow 30 minutes for warm-up prior to calibration.
2. Program the sample matrix into the computer.
3. Profile and calibrate the instrument using the mixed calibration standards. Flush the system with the calibration blank between each standard.
4. Analyze an EPA trace metals concentrate. The values obtained should be within 5 percent of the expected value. Enter the values in the instrument logbook.
5. Analyze the samples. Flush the system with the calibration blank between each sample.
6. If the method of standard addition is required, use the following procedure:

Two identical aliquots of the sample solution, each of volume  $V_x$ , are taken. To the first (labeled A) is added a small volume  $V_s$  of a standard analyte solution of concentration  $c_s$ . To the second (labeled B) is added the same volume  $V_s$  of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration  $c_x$  is calculated:

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$$c_x = \frac{S_B V_s c_s}{(S_A - S_B) V_x}$$

where  $S_A$  and  $S_B$  are the analytical signals (corrected for the blank) of solutions A and B, respectively.  $V_s$  and  $c_s$  should be chosen so that  $S_A$  is roughly twice  $S_B$  on the average. It is best if  $V_s$  is made much less than  $V_x$ , and thus  $c_s$  is much greater than  $c_x$ , to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results from this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.
2. The chemical form of the analyte added must respond the same as the analyte in the sample.
3. The interference effect must be constant over the working range of concern.
4. The signal must be corrected for any additive interference.

#### CALCULATIONS

1. Sample values are reported directly as concentration in ppm. All values should be blank corrected using the appropriate reagent blank.
2. If dilution was required, correct the sample values by the appropriate factor.
3. Data should be reported in ug/ml using up to three significant figures.

#### REFERENCE

1. "Inductively Coupled Plasma--Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes", Method 200.7, U.S. Environmental Protection Agency, Cincinnati, Ohio.

#### SULFUR IN PETROLEUM PRODUCTS

#### SCOPE AND APPLICATION

This method covers the determination of sulfur in petroleum products including

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lubricating oils containing additives; it is applicable to any petroleum product sufficiently low in volatility that it can be weighed accurately in an open sample boat.

#### SUMMARY OF METHOD

The sample is oxidized by combustion in a bomb containing oxygen under pressure. The sulfur, as sulfate in the bomb washings, is determined by ion chromatography.

#### SAMPLE COLLECTION AND PRESERVATION

No special precautions required.

#### INTERFERENCES

No known interferences at this time.

#### REAGENTS

1. Oxygen, free of combustible material and sulfur compounds, available at a pressure of 40 atmosphere.
2. Sodium Carbonate Solution--Dissolve 50 g of anhydrous  $\text{Na}_2\text{CO}_3$  in deionized water and dilute to 1 liter.
3. 1000 ug/ml Sulfate Solution--Dissolve 1.479 g of dried ( $105^\circ\text{C}$ )  $\text{Na}_2\text{SO}_4$  in deionized water and dilute to 1 liter.
4. Working Standard Solutions--Prepare, as a minimum, the following working conditions by dilution of the stock standard with deionized water: 5, 10 and 30 ug/ml.
5. Air--compressed.
6. Standard Eluent ( $0.003 \text{ M } \text{NaHCO}_3$  .  $0.024 \text{ M } \text{Na}_2\text{CO}_3$ )--Prepare by dissolving 1.0080 g  $\text{NaHCO}_3$  and 1.0176 g  $\text{Na}_2\text{CO}_3$  in deionized water and dilute to 4 liters.
7. Regeneration Solution ( $1\text{N } \text{H}_2\text{SO}_4$ )--Dilute 111 ml concentrated sulfuric acid to 4 liters with deionized water.

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## APPARATUS

1. Parr Bomb
2. Platinum sample cup
3. Platinum firing wire
4. White nylon or cotton sewing thread
5. Ignition circuit capable of supplying sufficient current to ignite the nylon or cotton thread without melting the wire.
6. Beakers (600 ml and 50 ml) and speedy vaps
7. Volumetric flasks, 100 ml
8. Hot plates
9. Dionex Ion Chromatograph
10. Plastic Syringe, 5-ml capacity
11. Millipore syringe filter unit 0.22  $\mu$ m
12. Recorder

## QUALITY CONTROL REQUIREMENTS

1. A method blank and an appropriate laboratory control sample such as NBS SRM 1619 must be analyzed with each group of samples.

## PROCEDURE

1. Preparation of bomb and sample:

Cut a piece of firing wire approximately 100 mm in length. Coil the middle section ( $\sim$ 20 mm) and attach the free ends to the terminals. Arrange the coil so that it will be above and to one side of the sample cup. Insert a nylon or cotton thread into the coil, of such length that one end extends into the sample cup. Place 10 ml  $\text{Na}_2\text{CO}_3$  solution in the bomb and by means of a rubber policeman wet the interior surface of the bomb, including the head, as thoroughly as possible. Weigh 0.8 g to 1.0 g sample into the sample cup.

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2. Addition of oxygen:

Place the sample cup in position and arrange the thread so that one end dips into the sample. Assemble the bomb and tighten the one end securely. Admit oxygen slowly (to avoid blowing the oil from the cup) until pressure is reached as indicated in the table below:

<u>Bomb Capacity (ml)</u>	<u>Min. Gage Pressure atm.</u>	<u>Max. Gage Pressure atm.</u>
300-350	38	40
350-400	35	37
400-450	30	32
450-500	27	29

3. Combustion:

Immerse the bomb in a cold water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample. Remove the bomb from the bath after immersion of at least 10 min. Release the pressure at a slow uniform rate such that the operation requires not less than 1 minute. Open the bomb and examine the contents. If traces of unburned oil or sooty deposits are found, discard the determination.

4. Collection of Sulfur Solution:

Rinse the interior of the bomb, the oil cup and the inner surface of the bomb cover with a fine jet of deionized water. Collect the washings in a 600-ml beaker which has a mark to indicate 75 ml. Remove any precipitate in the bomb by means of a rubber policeman. Wash the base of the terminals until the washings are neutral to a suitable indicator. Add 10 ml of saturated bromine water to the washings in the beaker. (The volume of the washings is normally in excess of 300 ml). Place the sample cup in a 50 ml beaker. Add

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5 ml of saturated bromine water, 2 ml of HCl and enough distilled water to cover the cup. Heat the contents of the beaker to just below its boiling point for 3 or 4 minutes and add to the beaker containing the bomb washings. Wash the sample cup and the 50-ml beaker thoroughly with deionized water. Remove any precipitate in the cup by means of a rubber policeman. Add the washings from the cup and the 50-ml beaker, and the precipitate, if any, to the bomb washings in the 600-ml beaker. Evaporate the combined washings to approximately 75 mls. Cool and dilute to volume in a 100 ml volumetric flask.

#### IC ANALYSIS

1. Set up the instrument according to the operator's manual.
2. Inject appropriate standards and record the peak heights. Analyze an EPA quality control sample to verify instrument calibration; the reported value should be within 5 percent of the expected value. Enter the results in the instrument logbook.
3. Inject the samples and record the peak heights. Make the necessary dilutions or adjustment of the MHO scale on the instrument to bring the sample into the linear range.
4. Calculate the regression data for the standards, enter the peak height value for each sample and record the calculated ppm value.
5. Calculate %S in the following manner:

$$\%S = \frac{\text{ug/ml SO}_4^= (\text{from curve}) \times \text{Dilution} \times \text{sample volume (ml)} \times 0.334}{\text{Sample weight (g)} \times 10,000}$$

#### REFERENCE

1. Standard Test Method for Sulfur in Petroleum Products (General Bomb Method) ANSI/ASTM D 129-64.

## CHLORINE IN PETROLEUM PRODUCTS

### SCOPE AND APPLICATION

This method covers the determination of chlorine in lubricating oils and greases, including new and used lubricating oils and greases containing additives, and in additive concentrates.

### SUMMARY OF METHOD

The sample is oxidized by combustion in a bomb containing oxygen under pressure. The chlorine compounds thus liberated are absorbed in a sodium carbonate solution and the amount of chloride present is determined by Ion Chromatography.

### SAMPLE COLLECTION AND PRESERVATION

Limit headspace of sample containers to preclude volatilization.

### INTERFERENCES

No known interferences at this time.

### REAGENTS

1. Oxygen--free of combustible material and halogen compounds available at a pressure of 40 atmosphere.
2. Sodium Carbonate Solution--Dissolve 50 g of anhydrous  $\text{Na}_2\text{CO}_3$  in deionized water and dilute to one liter.
3. 1000 ug/ml Chloride Standard--Dissolve 1.6484 g NaCl in deionized water and dilute to one liter.
4. Working Standard Solutions--Usually the following standards are prepared using deionized water for dilutions: 0.5 ug/ml, 1.0 ug/ml, 3.0 ug/ml.
5. Air--Compressed.

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6. Standard Eluent ( $0.003 \text{ M NaHCO}_3$  ,  $0.0024 \text{ M Na}_2\text{CO}_3$ )--Prepare by dissolving 1.0080 g  $\text{NaHCO}_3$  and 1.0176 g  $\text{Na}_2\text{CO}_3$  in deionized water and dilute to 4 liters.
7. Regeneration Solution ( $1\text{N H}_2\text{SO}_4$ )--Dilute 111 ml concentrated sulfuric acid to 4 liters with deionized water.

#### APPARATUS

1. Parr Bomb
2. Stainless steel sample cup
3. Stainless steel firing wire
4. Nylon or white cotton sewing thread
5. Ignition circuit, capable of supplying sufficient current to ignite the nylon or cotton thread without melting the wire.
6. Beakers--600 ml pyrex
7. Speedy-vaps to fit 600 ml beakers
8. Volumetric flasks--100 ml
9. Hot plates
10. Dionex Ion Chromatograph
11. Plastic syringe, 5 ml capacity
12. Millipore cyringe filter unit, 0.22  $\mu\text{m}$
13. Recorder

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#### QUALITY CONTROL REQUIREMENTS

1. Spiked samples and a reagent blank should be analyzed with each group of samples.

#### PROCEDURE

1. Preparation of bomb and sample:

Cut a piece of firing wire approximately 100 mm in length. Coil the middle section ( $\sim 20\text{mm}$ ) and attach the free ends to the terminals.

Arrange the coil so that it will be above and to one side of the sample cup. Insert a nylon or cotton thread into the coil, of such length that one end extends into the sample cup. Place 10 ml  $\text{Na}_2\text{CO}_3$

solution in the bomb and by means of a rubber policeman wet the interior surface of the bomb, including the head, as thoroughly as possible. Weigh 0.8 g 1.0 g sample into the sample cup.

2. Addition of oxygen:

Place the sample cup in position and arrange the thread so that one end dips into the sample. Assemble the bomb and tighten the cover securely. Admit oxygen slowly (to avoid blowing the oil from the cup) until pressure is reached as indicated in the table below:

<u>Bomb Capacity (ml)</u>	<u>Min. Gage Pressure atm.</u>	<u>Max. Gage Pressure atm.</u>
300-350	38	40
350-400	35	37
400-450	30	32
450-500	27	29

3. Combustion:

Immerse the bomb in a cold water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample. Remove the bomb from the bath after immersion of at least 10 min. Release the pressure at a slow uniform rate such that the operation requires not less than one minute. Open the bomb and examine the contents. If traces of unburned oil or sooty deposits are found, discard the determination.

4. Collection of chloride sample:

Using deionized water, rinse the interior of the bomb, the sample cup, and the inner surface of the bomb cover into a 600 ml beaker. Scrub the interior of the bomb and the inner surface of the bomb cover with a rubber policeman. Wash the base of the terminals into the beaker. Cover the beaker with a speedy vap and gently reduce volume to 75 ml or less. Remove from the hot plate and cool to

room temperature. Transfer the contents of the beaker into a 100 ml volumetric flask. Rinse the beaker several times, transferring rinses to the volumetric flask. Dilute to volume.

#### IC ANALYSIS

1. Set up the instrument according to the operator's manual.
2. Inject appropriate standards and record the peak heights.
3. Inject the samples and record the peak heights. Make the necessary dilutions or adjustment of the MHO scale on the instrument to bring the sample in to the linear range.
4. Calculate the linear regression data for the standards, enter the peak height value for each sample and record the calculated ppm value.
5. Calculate % Cl--using the following equation:

$$\text{Wt\% Cl} = \frac{\text{Sample ppm (from curve)} \times \text{Dilution} \times \text{Sample volume (ml)}}{\text{Sample Weight (g)} \times 10,000}$$

#### REFERENCE

1. Standard Test Method for Chlorine in New and Used Petroleum Products (Bomb Method) ANSI/ASTM D 808-63.

#### ORGANIC ANALYSIS PROCEDURES

The procedures used for the determination of volatile and semivolatile organic compounds and PCBs in oil are described in the following pages.

#### PROCEDURE

Volatile organics: tetraglyme extraction with modified Method 624 analysis

Volatile organics (Federal Register Method 624)

Semivolatile organics

Base/neutrals, acids and pesticides (Federal Register Method 625)

PCBs in oil

Pesticides and PCBs (Federal Register Method 608)

#### VOLATILE ORGANICS: TETRAGLYME EXTRACTION WITH MODIFIED METHOD 624 ANALYSIS

##### SCOPE AND APPLICATION

This method is designed to prepare a variety of nonaqueous samples for analysis for volatile organic compounds. It is applicable to oils, sludges, and solid wastes. It allows the detection and quantitation of organic compounds amenable to analysis by the purge and trap method as outlined in Method 624.

##### SUMMARY OF METHOD

A 1 gram portion of sample is extracted with 20 ml of tetraglyme. After phase equilibration, the tetraglyme layer is withdrawn, placed in a screw-cap vial with a minimum of headspace, and stored at 4°C for subsequent analysis. At the time of analysis, an aliquot of the extract is spiked into 25 ml of deionized water and Method 624 protocol are followed for the determination of volatile organics.

##### SAMPLE COLLECTION AND PRESERVATION

Samples collected for volatile organics analysis should be placed in 40 ml glass vials equipped with a septum-lined screw cap (same as in 624).

1. Fill the sample vial as completely as possible. Nonfluid samples should be packed into the vial with a minimum of headspace; fluid samples should be collected so no air bubbles remain in the vial.

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2. Seal the vial.
3. Collect a duplicate sample.
4. Store the sample at 4°C until the time of extraction.

#### INTERFERENCES

1. See Method 624.
2. Daily analysis of method blanks--Analyze a sample of deionized water taken from the Inorganic Wet lab and spiked with 100 ul of tetraglyme from the same batch as that used for sample extraction.
3. After each analysis, rinse the purge tube with deionized water. Bake the trap for a minimum of 20 min. to ensure adequate desorption of high molecular weight purgeables. Nonaqueous samples typically contain more high-molecular weight material than do water samples, and the possibility of system contamination must be recognized.
4. Sample analysis time should be a minimum of 40 minutes to allow elution of higher boiling purgeables. Each chromatogram should be checked for possible chromatographic carryover; should this be suspected, the trap and oven should be baked out for 30 minutes before sample analysis continues.

#### REAGENTS

1. See Method 624.
2. Tetraglyme--cleaned by placing in a rotovap under aspirator suction for a minimum of 30 minutes. Tetraglyme cleaned in this fashion should be analyzed prior to use for extraction. A 100 ul aliquot spiked into 25 ml of deionized water should not interfere with detection of the internal standards used in Method 624 or cause baseline elevation greater than 30 percent the peak height of the highest internal standard used for analysis.

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3. Calibration standards--standards used for Method 624 analysis are spiked with 100 ul tetraglyme prior to calibration curve analyses.

#### PROCEDURE

##### TETRAGLYME EXTRACTION

1. Weigh 1 gram portion of sample into a precleaned 40 ml glass screw cap vial.
2. Cover the sample immediately with 20 ml of tetraglyme measured with a delivery pipette.
3. Replace the vial cap and shake the vial until the layers mix or emulsify (no longer than 1 minute). If the sample is solid or highly viscous, sonication should be performed with the sonicator probe for 30 seconds at a setting of 5 and a 25 percent pulsed duty cycle.
4. Allow the tetraglyme/sample mixture to equilibrate until the layers are clearly separated. The appropriate time will vary with sample type; oil samples should be stored at 4°C overnight in contact with the tetraglyme while extracts from solids will require less time. Some samples may completely mix with the tetraglyme to form a solution.
5. Withdraw the tetraglyme layer and place in a precleaned screw cap glass vial. Allow a minimum of headspace. Store at 4°C.

##### METHOD 624 ANALYSIS

1. The volume of tetraglyme extract appropriate for Method 624 analysis within the established calibration curve will vary with the level of volatile content of the original sample. If this is known to an approximation, the volume of tetraglyme extract to be used for analysis may be calculated by the following equation. This equation

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is based upon the assumption that tetraglyme added to 25 ml water will give most accurate quantitation if the resultant solution is in the middle range of the calibration curve, or about 100 ug/l, for organic volatiles.

Volume of tetraglyme to use (ml) =

$$100 \text{ ug/l} \times 0.025 \text{ liter} \times 20 \text{ ml tetraglyme extract}$$

---

$$\text{Sample wt (g) extracted} \times \text{sample contaminant levels (ug/g)}$$

2. For samples of unknown content, a nominal 10 ul aliquot is measured with a 25 ul syringe and spiked into 25 ml of deionized water.
3. Analysis is conducted by purge and trap protocol as outlined in method 624.
4. The total ion chromatogram is reviewed after analysis to determine whether maximum detection for the sample has been achieved without instrument saturation. This is done by monitoring the abundance of the base peak for major organic volatile components. The mass spectrometer is saturated when the count for the base peak of a compound exceeds 32767 for a single scan. If the maximum counts for the base peaks of the compounds detected as major components are less than 3000, detection limits may be reduced by an order of magnitude or greater. Reanalysis using 100 l or greater portions of the tetraglyme extract may be conducted without instrument saturation occurring.

If instrument saturation occurs during the analysis of the 10 ul tetraglyme aliquot, base peak counts for a single scan will exceed 32767. If this is noted, reanalysis should be conducted on a 1 ul aliquot of the extract.

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Sample Quantitation

1. Determine the level of organic components in the water solution using the established calibration curve.
2. Calculate the level of sample content by the following equation:

Sample concentration (ug/g) =

Water conc. (ug/l) x 0.025 liter x 20 ml tetraglyme

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Vol. of tetraglyme extract spiked into water x wt of sample extracted (g)

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VOLATILE ORGANICS (FEDERAL REGISTER METHOD 624)

1.0 SCOPE AND APPLICATION

This method is designed to detect and quantify volatile organic compounds which are amenable to the purge and trap method; the compounds routinely determined by this procedure are shown in Table B-2. This method has been approved for NPDES monitoring.

2.0 SUMMARY OF METHOD

A 25-ml sample is purged with a stream of helium. The volatile organics present in the sample are transferred to the vapor phase and trapped on a sorbent column. After purging is completed, the sorbent column is heated and backflushed with helium to desorb the components onto a gas chromatographic column. The gas chromatographic column is heated to elute the components which are detected by a mass spectrometer.

3.0 SAMPLE COLLECTION AND PRESERVATION

Grab samples are collected in 40-ml glass vials equipped with a septum and screw cap. Prior to use, the vials, caps and septa must be soap- and water-washed, rinsed with deionized water and dried at 105°C for 1 hour. The following procedure is used for sample collection and storage.

- 3.1 Fill the sample vial completely, do not allow any air bubbles to pass through the sample.
- 3.2 Seal the vial; check that no air bubbles have been trapped in the sample.
- 3.3 Collect a duplicate sample.
- 3.4 Store sample at 4°C until the time of analysis; analysis must be performed within 14 days of sample collection.

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#### 4.0 INTERFERENCES

A variety of interfering substances may be present in more complex samples such as industrial effluents. The most common problem associated with this analysis is contamination; the following steps should be taken to detect contamination introduced in sample handling or analysis.

- 4.1 Analysis of field biased blanks--Deionized water carried through the sampling, storage and analysis procedures.
- 4.2 Daily analysis of method blanks--Analyze a sample of deionized water at the start of each analysis session, this detects contamination introduced by the purge gas or the plumbing ahead to the trap.
- 4.3 After analysis of a high level sample, a blank of deionized water should be analyzed to ensure that contamination of subsequent samples by carry-over does not occur.

#### 5.0 REAGENTS

- 5.1 Organic-free deionized water--Water from the Continental deionized water system should be used. To avoid possible contamination by methylene chloride, obtain the water from the tap in the Inorganic Wet Lab.

#### 5.2 Sorbent Trap Materials

- a. 2,6-Diphenylene oxide polymer - 60/80 mesh Tenax, chromatographic grade.
- b. Methyl silicone packing - 3 percent OV-1 on 60/80 mesh Chromosorb W.
- c. Silica gel, Davison Chemical, 35/60 mesh, grade 15.

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### 5.3 CALIBRATION STANDARDS

5.3.1 Stock solutions - 2 mg/ml: With the exception of the gases, the stock standard solutions may be either purchased from Supelco or prepared from the pure compound. Stock solutions for the gases (bromomethane, chloroethane, chloromethane and vinyl chloride) are always purchased. If stock solutions are prepared from the pure compound the following procedure is used:

- a. Place 9.8 ml of methanol in a 10 ml ground glass stoppered volumetric flask.
  - b. Allow flask to stand unstoppered for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.
  - c. Using a 100  $\mu$ l syringe, add 2 to 3 drops of the standard material to the flask. Be certain that the drops fall directly into the alcohol without contacting the neck of the flask. Reweigh the flask.
4. Store stock standards at 4°C; prepare fresh standards every 2 weeks.

### 5.3.2 WORKING STANDARD SOLUTIONS

- a. Prepare 100 ml of each of the following working standard solutions by dilution of the stock solutions: 5, 10, 50, 100, 150  $\mu$ g/l.

### 5.4 INTERNAL STANDARD SOLUTION

5.4.1 The internal standard spiking solution is prepared by dilution of the Supelco standard which contains the three internal standards, bromochloromethane, 2-bromo-1-chloropropane and 1,4-dichlorobutane, at a concentration of 20 mg/ml. Alternately, this solution may be prepared from the stock standards.

5.4.2 Add 1 ml of the Supelco solution to 90 ml of deionized water in a 100 ml volumetric flask.

5.4.3 Dilute to volume and invert to mix. The concentration of this solution is 0.20 mg/ml. Each sample is spiked with 2.5 ul of this solution.

5.4.4 Prepare a fresh internal standard solution monthly.

5.5 UHP Helium--used as purge gas and GC carrier gas.

#### 6.0 APPARATUS

6.1 Hewlett-Packard 5985 GC/MS.

6.2 Purge and Trap Device - Tekmar LSC 3.

6.3 GC Column - 1 Percent SP-1000 on Carbopack B, 8 ft x 2 mm ID column.

6.4 Syringe - glass, 25-ml hypodermic with Luer-Lok tip.

6.5 Microsyringes - 10, 25, 100 ul.

6.6 Volumetric glassware for preparation of standards.

#### 7.0 QUALITY CONTROL REQUIREMENTS

7.1 A method blank of deionized water must be analyzed daily.

7.2 The volatiles laboratory control sample (LCS) must be analyzed daily. The reported value for each of the parameters should be entered in the instrument logbook. If the reported values for any of the components of the LCS are outside the established control limits ( $\pm 2S$ ), corrective action should be taken prior to running samples.

## 8.0 CALIBRATION AND TUNING

8.1 A five-point calibration curve is used, a new calibration curve is prepared every 2 weeks. Run each of the five working standard solutions as outlined in the procedures section. The information regarding response ratio versus area ratio for each standard is stored in the computer and used to calculate sample concentration.

8.2 Calculate the response factor (RF) for each compound using the following equation:

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where  $A_s$  = Area of the primary characteristic ion for the compound to be measured.

$A_{is}$  = Area of the primary characteristic ion of the internal standard.

$C_{is}$  = Concentration of the internal standard.

$C_s$  = Concentration of the compound to be measured.

8.3 The instrument should be tuned daily according to the following procedures:

8.3.1 Allow column oven and trap to heat to their maximum temperatures (200°C and 180°C, respectively).

8.3.2 Tune the instrument with DFTPA using the following criteria:

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<u>Mass</u>	<u>Ion Abundance Criteria</u>
69	100%
131	28-35% of mass 69
219	22-28% of mass 69

The ion abundance values for mass 131 and mass 219 should differ by 4 to 8 percent.

8.3.3 After allowing the column and trap to cool, analyze the Laboratory Control Sample.

#### 9.0 PROCEDURE

9.1 Remove standards and samples from cold storage at least 1 hour prior to analysis and allow to warm to room temperature.

9.2 Adjust the helium flow rate to 40 ml/minute. Attach the trap inlet to the purging device and set the device to the purge mode. Open the syringe valve located on the purging device sample introduction needle.

9.3 Remove the plunger from a 25-ml syringe; open the sample or standard bottle and pour the sample or standard into the syringe barrel until it overflows. Replace the syringe plunger and adjust the sample volume to 25-ml. Replace the cap on the sample bottle and return to cold storage.

9.4 Remove the Luer tip of the 25-ml syringe and add 2.5 ul of the internal standard solution; replace the Luer-Lok.

9.5 Inject the sample into the purging chamber.

9.6 Purge with helium to transfer the volatile components to the trap. Adjust helium flow rate to 40 ml/min.

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purge and purge the sample for  $10.0 \pm 0.1$  minutes at ambient temperature.

- 9.7 After 10 minutes of purging adjust the device to the desorb mode and begin GC/MS analysis and data acquisition using the following conditions:

GC Conditions

GC Column	1% SP-1000 on Carbopack B, 8 ft x 2 mm ID column.
Temperature Program	Isothermal at 60°C for 4 min, then 10°/min to 220°C and held at that temperature for 15 min.
Injector Temperature	225°C
Carrier Flow	UHP helium, 30 ml/min.

Purge and Trap Conditions

Desorption Temperature	180°C
Desorption Time	4 min
Oven Temperature	200°C

MS Conditions

Emission	300 ua
Electron Energy	70 eV
Scan Rate	133.3 amu/sec
Mass Interval	41-350 amu

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10.0 QUALITATIVE AND QUANTITATIVE DETERMINATION

- 10.1 To qualitatively identify a compound, obtain an Extracted Ion Current Profile (EICP) for the primary ion and at least two other characteristic

These calculations are normally performed by the HP 5985 computer.

11.0 REFERENCE

- 11.1 Method 624, Federal Register, Monday, December 3, 1979, 40 CFR Part 136.

TABLE B-2. REPORTED COMPOUNDS AND THEIR CHARACTERISTIC IONS

Compound	Primary Ion
chloromethane	50
dichlorodifluoromethane	85
bromomethane	94
vinyl chloride	62
chloroethane	64
methylene chloride	84
trichlorofluoromethane	101
1,1-dichloroethylene	96
1,1-dichloroethane	63
trans-1,2-dichloroethylene	96
chloroform	83
1,2-dichloroethane	98
1,1,1-trichloroethane	97
carbon tetrachloride	117
bromodichloromethane	83
bis-chloromethyl ether	79
1,2-dichloropropane	63
trans-1,3-dichloropropene	75
trichloroethylene	130
dibromochloromethane	129

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ions. The following criteria must be met for a qualitative identification:

- 10.1.1 The characteristic ions of each compound of interest must maximize in the same scan or within one scan of each other.
- 10.1.2 The retention time must fall within  $\pm 60$  seconds of the retention time of the authentic compound.
- 10.1.3 The relative peak heights of the characteristic ions in the EICPs must fall within  $\pm 20$  percent of the relative intensities of these ions in a reference mass spectrum. The reference mass spectrum may be that of a standard or from a reference library.
- 10.1.4 Table B-2 shows that compounds routinely reported and the primary ion used to quantify each.
- 10.1.5 Quantitative determination of each compound is done using the internal standard method. Calculate the concentration of each compound according to the following equation:

$$\text{Concentration: ug/l} = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

where  $A_s$  = Area of the primary characteristic ion of the compound to be measured.

$C_{is}$  = Concentration of the internal standard in ug/l in the samples.

$A_{is}$  = Area of the primary characteristic ion of the internal standard.

RF = Response factor of the compound being quantified

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cis-1,3-dichloropropene	75
1,1,2-trichloroethane	83
benzene	78
2-chloroethylvinyl ether	106
bromoform	173
1,1,2,2-tetrachloroethene	166
1,1,2,2-tetrachloroethane	83
toluene	91
chlorobenzene	112
ethylbenzene	91
acrolein	56
acrylonitrile	53

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## SEMIVOLATILE ORGANICS

The following procedures relate to the determination of polynuclear aromatic hydrocarbons, nitrosoamines and related semivolatile compounds in used engine oils. Polynuclear aromatic hydrocarbons (PAH) and nitrosoamines are potential products of the combustion process in internal combustion engines. The following method used in this program involves a column chromatography procedure to remove the aliphatic components of the oil and allow for the quantitation of PAH and nitrosoamines by GC/MS. The following conditions represent those used in this study, when different from the conditions stipulated in the Method 625 procedures.

### MATERIALS/APPARATUS

1. Column - 200 mm x 10 mm ID glass with Teflon stopcock, water jacketed.
2. Silica gel - Davison Grade 950, 60-200 mesh; cleaned by sequential soxhlet extractions with methanol, methylene chloride, and pentane. Activated at 110°C for at least 2 hours, and cooled in a desiccator.
3. Gas Chromatograph/Mass Spectrometer operated under the following conditions:

#### GC Conditions

Column	SE54, 30-meter fused silica capillary
Temperature program	60°C held for 2 minutes and ramped at 10°C/minute to 260°C and held
Injector temperature	275°C
Injection volume	1 ul
Flow	0.5 ml/min

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### MS Conditions

Ionization voltage	70 eV
Emmission voltage	300 uA
Source temperature	200°C
Scan rate	1.0 sec/scan
Range	45 to 450 m/Z

### REAGENTS

1. Pentane - pesticide residue grade
2. Methylene Chloride - pesticide residue grade
3. Methanol - pesticide residue grade
4. Sodium Sulfate - anhydrous, regeant grade

### PROCEDURE

#### COLUMN PREPARATION

1. Slurry-pack the column with 6.0g ( $\pm 0.2$ g) of Silica gel in pentane.  
When the column is fully prepared, allow the pentane to drain to the top of the Silica bed.
2. Add 3.0g ( $\pm 0.2$ g) of sodium sulfate to the top of the column with several pentane washings. Drain pentane to top of sodium sulfate layer.

#### SAMPLE PREPARATION

1. Weigh approximately 0.5g of a homogeneous waste oil sample into a 50 ml beaker to a precision of 0.1 mg.
2. Mix oil sample with sufficient silica gel to provide a relatively dry mix.

#### FRACTIONATION

The following fractions are collected:

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<u>Fraction</u>	<u>Solvent</u>	<u>Volume</u>
1	pentane	25.0 ml
2	50% methylene chloride/50% pentane	10.0 ml
3	95% methylene chloride/5% methanol	10.0 ml
4	50% methylene chloride/50% methanol	10.0 ml

1. Transfer oil/silica gel mix to head of column with several washings of the first elution solvent, 25.0 ml of pentane.
2. Elute with the remaining pentane and reserve the eluate in a 25.0-ml container. Bring to volume (25.0 ml) with pentane if necessary.
3. Repeat procedure for fractions 2,3, and 4, bringing each separate fraction to the specified volume with the proper eluting solvent. Maintain an elution rate of 1.0 ml/min for reproducibility and adequate resolution.
4. Reduce (by Kuderna-Danish evaporation and nitrogen blow down) 5 ml of fractions 2, 3, and 4 to 0.5 ml. Combine the concentration from fractions 3 and 4 for analysis by Method 625. Hold the remainder of the fractions in reserve.
5. Fraction 1 contains the aliphatic hydrocarbons that are responsible for the background interference in this sample matrix. The fraction is reserved though it is unsuitable for analysis without further preparation.

#### REFERENCE

Estes, E.D., W.F. Gutknecht, D.E. Lentsen, D.E. Wagoner. IERL-RTP Procedure Manual: Level 1 Environmental Assessment, 2nd ed., p. 144-148.

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BASE/NUETRALS, ACIDS AND PESTICIDES (FEDERAL REGISTER METHOD 625)

1.0 SCOPE AND APPLICATION

This method is designed to detect and quantify most neutral, basic and acidic organic compounds which are soluble in methylene chloride and amenable to gas chromatography; the method may be used for water and wastewater samples. Method 625 has been approved for NPDES monitoring.

2.0 SUMMARY OF METHOD

The sample is extracted with methylene chloride using a separatory funnel. The extract is dried over sodium sulfate and concentrated to 1 to 2 mls using a Kuderna-Danish (K-D) evaporator. Quantitative analysis is performed by GC/MS.

3.0 SAMPLE COLLECTION AND PRESERVATION

Samples are collected in 1 gallon glass containers and stored at 4°C until the time of analysis. All samples must be extracted within 14 days and analyzed within 30 days of collection.

4.0 INTERFERENCES

A variety of interfering substances may be present in more complex samples such as industrial effluents. Method interferences may be caused by contaminants introduced by reagents or glassware; a method blank must be analyzed with each set of samples to detect any contamination.

5.0 REAGENTS

5.1 Sodium Hydroxide--6N solution

5.2 Sulfuric Acid--6N solution

5.3 Sodium Sulfate--Granular, anhydrous; rinse with methylene chloride (20 ml/g) and dry in fume hood.

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- 5.4 Methylene Chloride--Burdick & Jackson "Distilled-in-Glass" or Baker "Resi-Analyzed" are acceptable grades of this solvent.
- 5.5 UHP Helium
- 5.6 Stock Standards--Stock standards for the compounds shown in Tables B-3, B-4 and B-5 are prepared at a concentration range of 200 to 400 ppb using assayed reference material (obtain from Chem Service)

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TABLE B-3. CHARACTERISTIC IONS AND RETENTION TIMES OF THE BASE/NEUTRAL COMPOUNDS

Compound	Characteristic ion	Retention time (min)
Bis(2-chloroethyl ether)	93	7.3
1,3-Dichlorobenzene	146	7.7
1,4-Dichlorobenzene	146	7.8
1,2-Dichlorobenzene	146	8.2
Bis(2-chloroisopropyl) ether	45	8.5
N-nitroso-dipropyl amine	70	8.8
Hexachloroethane	117	8.9
Nitrobenzene	77	9.1
Isophorone	82	9.7
Bis(2-chloroethoxy)methane	93	10.3
1,2,4-Trichlorobenzene	180	10.6
Naphthalene	128	10.8
Hexachlorobutadiene	225	11.2
Hexachlorocyclopentadiene	237	13.0
2-Chloronaphthalene	162	13.7
Dimethyl phthalate	163	14.7
Acenaphthylene	152	14.7
2,6-Dinitrotoluene	165	14.9
Acenaphthene	154	15.2
2,4-Dinitrotoluene	165	15.8
Fluorene	166	16.5
Diethyl phthalate	149	16.5
4-Chlorophenyl phenyl ether	204	16.5
N-Nitrosodiphenyl amine	169	16.9
1,2-Diphenyl hydrazine	77	16.9
4-bromophenyl phenyl ether	248	17.8
Hexachlorobenzene	284	18.1
Phenanthrene	178	18.9
Anthracene	178	18.9-19.0
Dibutyl phthalate	149	20.6
Fluoranthene	202	21.9
Pyrene	202	22.5
Butyl benzylphthalate	149	24.5
Benz(a)anthracene	228	26.3
Bis(2-ethylhexyl)phthalate	149	27.0
Dioctyl phthalate	149	30.6
Benzo fluoranthene	252	32.4
Benz(a)pyrene	252	34.8
Dibenzo(a,h) anthracene	278	47.7



TABLE B-4. CHARACTERISTIC IONS AND RETENTION TIMES OF THE ACID COMPOUNDS

Compound	Characteristic ion	Retention time (min)
Phenol	94	7.2
2-chlorophenol	128	7.4
2-nitrophenol	139	9.9
2,4-dimethylphenol	107	10.1
2,4-dichlorophenol	162	10.5
p-chloro-o-cresol	142	12.4
2,4,6 trichlorophenol	198	13.4
2,4-dinitrophenol	184	15.6
4-nitrophenol	139	15.9
4,6-dinitro-o-cresol	198	16.9
Pentachlorophenol	266	18.7

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TABLE B-5. CHARACTERISTIC IONS AND RETENTION TIMES OF PESTICIDES/PCBs

Compound	Characteristic ion	Retention time (min)
Aldrin	66	19.8
a-BHC <sup>a</sup>	183	18.5
B-BHC	181	19.5
§-BHC <sup>a</sup>	183	19.1
4,4'-DDD	235	23.6
4,4'-DDE	246	23.0
4,4'-DDT	235	25.0
Dieldrin	79	23.4
Endosulfan I <sup>a</sup>	201	---
Endosulfan II <sup>a</sup>	201	---
Endosulfan Sulfate	272	27.0
Endrin <sup>a</sup>	81	---
Endrin Aldehyde	250	24.3
Heptachlor	100	20.0
Heptachlor epoxide	353	22.0
PCB 1016 <sup>b</sup>	186	19-22
PCB 1221 <sup>b</sup>	188	20-23
PCB 1232 <sup>b</sup>	188	20-23
PCB 1242 <sup>b</sup>	258	21-24
PCB 1248 <sup>b</sup>	292	21-24
PCB 1254 <sup>b</sup>	326	22-25
PCB 1260 <sup>b</sup>	360	22-25

<sup>a</sup>Subject to decomposition deriving from the alkaline conditions of the Method 625 extraction. Recoveries by this method are known to be low.

<sup>b</sup>Mixtures of isomers eluting over a 2- to 3-minute time period.

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- 5.6.1 Dissolve the reference material in hexane or other suitable solvent. Dilute to volume in a 100 ml ground glass stoppered volumetric flask using hexane.
- 5.6.2 Transfer the stock solution to 15 ml Teflon lined screw cap vials. Mark with the date and refrigerate. PNA standards must be protected from light.
- 5.6.3 Check standards for signs of degradation or evaporation prior to preparing working standards. Prepare fresh standards as needed.
- 5.7 Working Standards--These are prepared by dilution of the stock solution to result in standards covering the range 20 to 400 ppb. Verify a new set of standards by analyzing the EPA QA samples for base/neutrals and acids.

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- 5.8 Wide Range pH paper.

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6.0 APPARATUS

- 6.1 Hewlett-Packard 5985 GC/MS with SE-54, 30m fused-silica capillary column.
- 6.2 Damon IEC centrifuge.
- 6.3 2000 ml separatory funnel.
- 6.4 Kuderna-Danish apparatus.
- 6.5 Concentrator tube, 15 ml. Ground glass stopper is used to prevent evaporation of extracts.
- 6.6 Drying Column--20 mm ID Pyrex chromatographic column equipped with glass wool plug.

6.7 500 ml Evaporative Flash--Attach to connector tube with springs.

6.8 Snyder Columns--2 ball micro, 3 ball macro.

6.9 Teflon boiling chips, 10/40 mesh, pre-extracted.

6.10 Water Bath.

7.0 QUALITY CONTROL REQUIREMENTS

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7.1 A method blank and a laboratory control sample must be taken through the entire extraction and analysis procedure with each group of 10 to 15 samples processed.

7.2 After calibrating the instrument, analyze the laboratory control sample. Enter the reported values in the instrument QC log. If the results are within acceptance limits ( $\pm 2s$ ), the analysis may proceed. If the results are outside the acceptance limits analysis should be stopped and corrective action taken. After preparation of new standard solutions verify their concentration by analyzing the appropriate EPA QC samples.

8.0 PROCEDURE

8.1 Sample Extraction

8.1.1 Measure 1 liter of water into a 2 liter separatory funnel using a graduated cylinder. If only 1 liter was collected, the entire sample should be used.

8.1.2 Spike the sample with the surrogate compounds, for base neutrals, either Decafluorobiphenyl, 2-fluoroaniline or D<sub>5</sub>-Bromobenzene may be used; for acids, use D<sub>5</sub> phenol. Adjust the sample pH to 11 or greater using 6N NaOH. Multirange pH paper can be used for pH measurement.

- 8.1.3 Rinse the graduated cylinder or sample bottle thoroughly with 60 ml methylene chloride and transfer the solvent rinse to the separatory funnel.
- 8.1.4 Extract the sample by shaking the funnel for 2 minutes, periodically venting to release excess vapor pressure. Allow organic layer to separate from the aqueous phase for a minimum of 10 minutes; if two distinct phases form, proceed to Step 8.1.5. If an emulsion forms which is more than one third the size of the solvent layers, the phase separation is completed by centrifugation according to the following procedure:
- a. Place the methylene chloride layer in a centrifuge bottle.
  - b. Centrifuge for 10 to 15 minutes at approximately 3200 rpm.
  - c. Pour the sample into a 125 ml separatory funnel and collect the methylene chloride layer in a BOD bottle.
  - d. Pour the remaining sample back into the 2-liter separatory funnel, rinse the centrifuge bottle and the 125 ml sep. funnel with the next portion of methylene chloride and continue with Step 8.1.6 below.
- 8.1.5 Collect the methylene chlorida layer in a BOD bottle.
- 8.1.6 Add a second 60 ml volume of methylene chloride to the sample bottle and complete the extraction procedure a second time. Add the second methylene chloride layer to the first in the BOD bottle.
- 8.1.7 Extract with a third 60 ml portion of methylene chloride adding this extract to the first two in the BOD bottle.

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- 8.1.8 Pour the combined extracts through a drying column containing 3 to 4 inches of anhydrous sodium sulfate and collect it in a 500 ml K-D flask equipped with a 15 ml concentrator tube.
- 8.1.9 Rinse the BOD bottle with 20 to 40 ml of methylene chloride and pour this through the drying column. The collected extracts and rinsings are sealed and labeled as the base/neutral fraction.
- 8.1.10 Adjust the pH of the sample, which was previously extracted for base/neutrals, to 2 or below using 6N  $\text{H}_2\text{SO}_4$ .
- 8.1.11 Serially extract with three 60-ml portions of methylene chloride as before.
- 8.1.12 Collect and combine the extracts in a BOD bottle and dry by passing through a column of anhydrous sodium sulfate. Rinse the BOD bottle with 20 to 40 ml of methylene chloride and pour the rinse through the drying column collecting extracts and rinsings as before in a 500 ml K-D flask having a 15 ml concentrator tube. Seal and label as the acid fraction.

8.2 CONCENTRATION

- 8.2.1 To concentrate the base/neutral fraction, use the following procedure:
- Add 1 or 2 clean boiling chips to the K-D flask and attach a 3-ball macro Snyder column. Prewet the Snyder column by adding approximately 1 ml of methylene chloride through the top.
  - Place the K-D apparatus on a warm water bath (60 to 65°C) so that the concentrator tube is partially immersed in the water and the entire lower rounded surface of the flask is bathed with water vapor.

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- c. Adjust the vertical position of the K-D flask and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation, the balls of the column should actively chatter but the chambers should not flood.
- d. When the liquid has reached a volume of less than 1 to 5 ml, remove the K-D apparatus and allow the solvent to drain for at least 10 minutes while cooling.
- e. Rinse the Snyder column, the flask and its lower joint into the concentrator tube with 10 ml of methylene chloride.
- f. Add a clean boiling chip and attach a two ball micro Snyder column to the concentrator tube. Prewet the column by adding about 0.5 ml methylene chloride through the top.
- g. Place the K-D apparatus on a warm water bath (60 to 65°C) so that the concentrator tube is partially immersed in the water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation, the balls of the column actively chatter but the chambers do not flood.
- h. When the liquid reaches an apparent volume of 0.5 ml, remove the K-D from the water bath and allow the solvent to drain and cool for at least 10 minutes.
- i. Remove the micro Snyder column and rinse its lower joint into the concentrator tube with approximately 0.2 ml of methylene chloride. Adjust the volume to 1.0 ml with methylene chloride and place in a 2.0 ml vial with Teflon lined cap. Rinse the concentrator tube with 1.0 ml hexane and combine with the methylene chloride portion. The final volume will be 2.0 ml.

96

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Label as the base neutral fraction. Use the same procedure for the concentration of the acid fraction.

### 8.3 GC/MS ANALYSIS

8.3.1 Tune the instrument daily by bleeding in PFTBA using the following criteria.

<u>Mass</u>	<u>Ion Abundance Criteria</u>
69	100%
131	32 to 37% of Mass 69
219	23 to 29% of Mass 69

The ion abundance values for mass 131 and mass 219 should differ by 6 to 10 percent.

8.3.2 The following instrument operating conditions are used:

#### GC CONDITIONS

Temperature program	50°C held for 2 min, then 10°/min to 260°C and held
Injection volume	Typically 1 ul
Injection temperature	275°C
Column flow	UHP helium, 0.5 ml/min

#### MS CONDITIONS

Emission	300 ua
Electron energy	70 eV
Scan rate	1.0 sec/scan
Mass Interval	45 to 450 amu

8.3.3 Adjust the system sensitivity by injecting phenanthrene. The sensitivity varies as a function of multiplier, column, injector

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and source interface characteristics. The optimum electron multiplier setting is that which will yield approximately 50X the detection limit but less than one-fourth of the value where serious saturation occurs for 50 ng of phenanthrene. For the HP 5985, the total ion count for the 188 peak should be about 20,000.

- 8.3.4 Program the instrument to operate in the Extracted Ion Current Profile (EICP) mode and collect EICP for the characteristic 100 percent ion shown in Tables B-3, B-4 and B-5 for each compound being measured.
- 8.3.5 Operating in the EICP mode, calibrate the system using the internal standard method.
- a. Determine the response factor using the following equation:

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where  $A_s$  = the integrated area or peak height of the characteristic ion for the pollutant standard.

$A_{is}$  = the area of the characteristic ion for the internal standard.

$C_{is}$  = the amount (ug) of the internal standard.

$C_s$  = the amount (ug) of the pollutant standard.

- b. Prepare a calibration curve by plotting the response factor against the standard concentration using a minimum of three standards in the range of interest. The concentration of the upper standard should be in the range of 200 to 300 ppb, the concentration of the lower standard should be one-tenth that of the upper standard of 20 to 30 ppb.

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- c. Verify the calibration curve daily; if significant drift has occurred, a new curve should be prepared.
- d. To quantify, add the internal standard to the concentrated sample extract. The following compounds are used as internal standards:

d<sub>8</sub>-naphthalene

d<sub>10</sub>-anthracene

d<sub>12</sub>-chrysene

- e. Calculate the concentration of each compound present using the following equation:

$$\text{Concentration (ug/l)} = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

where  $A_s$  = area of the primary characteristic ion of the compound to be measured.

$C_{is}$  = concentration of the internal standard in ug/l in the samples.

$A_{is}$  = area of the primary characteristic ion of the internal standard.

RF = response factor of the compound being quantified.

These calculations are normally performed by the HP 5985 computer.

## 9.0

### REFERENCE

1. Method 625, Federal Register, Monday, December 3, 1979, 40 CFR Part 136.

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## PCBs IN OIL

The procedure used in this program for the determination of PCBs in Oil was a modification of EPA Method 608. The modifications used in sample preparation are shown below; the full text of Method 608 is contained in the following pages.

### PREPARATION PROCEDURE

1. Take an aliquot of oil weighing approximately 1.0 g, add 5 ml hexane and 5 ml concentrated sulfuric acid.
2. Shake the sample for approximately 1 minute. If the sample extract is not clear, it must be applied to a Florisil column as described in Section 8.2 of EPA Method 608.
3. Analyze as described in Section 9.0 of Method 608; report results as ug/g.

## PESTICIDES AND PCBs (FEDERAL REGISTER METHOD 608)

### 1.0 SCOPE AND APPLICATION

This method may be used in the determination of certain organochlorine pesticides and polychlorinated byphenyls (PCBs) in water and wastewater. This method has been approved for NPDES monitoring.

### 2.0 SUMMARY OF METHOD

A 1-liter sample is extracted with methylene chloride using separatory funnel techniques. The extract is dried, concentrated and solvent exchanged into hexane. If necessary, sample concentrates are cleaned by Florisil chromatography to remove interferences. Identification and quantitation is performed using a gas chromatograph equipped with an electron capture detector.

### 3.0 SAMPLE COLLECTION AND PRESERVATION

Grab samples are collected in 1 liter glass containers following normal

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sample collection procedures; the bottle should not be prewashed with sample prior to collection. If composite samples are collected the samples should be placed in refrigerated glass containers. Tygon tubing should not be used in sampling equipment.

The samples should be stored at 4°C until extraction. Samples must be extracted within 14 days and analyzed within 30 days of collection.

#### 4.0 INTERFERENCES

Contamination may be introduced by reagents or inadequate glassware cleaning. A variety of interfering substances may be present in more complex samples such as industrial effluents. Contact with plastics must be avoided since phthalate esters can be a major interference. Prior to use, all glassware should be cleaned using the following procedure:

- 4.1 Solvent rinse immediately after use.
- 4.2 Wash thoroughly with soap and hot water.
- 4.3 Rinse with hot water, deionized water, acetone and pesticide quality hexane.
- 4.4 Heavily contaminated glassware may require treatment by heating at 400°C for 15 to 30 minutes. However certain high boiling materials such as PCBs may not be eliminated by this treatment; contaminated Class A volumetric glassware (e.g. pipets, volumetric flasks) must not be cleaned by this method.
- 4.5 Clean glassware should be capped with aluminum foil prior to storage.
- 4.6 Immediately before use; rinse the glassware with methylene chloride or hexane.

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5.0 REAGENTS

5.1 Preservatives:

5.1.1 Sodium hydroxide, J.T. Baker, 10 N in distilled water.

5.1.2 Sulfuric acid, J.T. Baker reagent grade 1+1 with distilled water.

5.2 Methylene Chloride--Baker "Resi-Analyzed" or Burdick and Jackson "Distilled-in-Glass."

5.3 Hexane--Baker "Resi-Analyzed" or Burdick and Jackson "Distilled-in-Glass."

5.4 Sodium Sulfate--Granular anhydrous, soxhlet extract with a mixture of 85 percent hexane, 15 percent methylene chloride and dry in a fume hood.

5.5 Stock Standards--Prepare stock standards at concentrations of 1.0 ug/ul by dissolving 0.010g of assayed reference material in pesticide quality hexane and diluting to volume in a 10 ml ground glass stoppered volumetric flask. Transfer the stock solution to screw-capped reagent bottles and store in a refrigerator. Prior to preparing working standards, check the stock solution for signs of degradation or evaporation.

5.6 Working Standards--Prepare mixed working standards at concentrations that will bracket the working range of the chromatographic system, usually 1 ng/ml to 100 ng/ml.

5.7 Diethyl ether--Burdick and Jackson Distilled-in-Glass, must be free of peroxides as indicated by EM Quant Test Strips (EM Laboratories, Inc., 500 Executive Blvd., Elmsford, NY 10523)

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- 5.8 Florisil--PR grade (60/100 mesh), purchase activated at 1250°F. Soxhlet extract in hexane and dry in a fume hood. Store in glass containers with glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 hours at 130°C in a foil covered glass container.
- 5.9 Multi-range pH paper.
- 6.0 APPARATUS
- 6.1 Hewlett Packard 5840 Gas Chromatograph with Ni<sup>63</sup> electron capture detector and HP 7671A automatic sampler.
- 6.2 Columns: Primary Column--1.5 percent OV-17/1-95 percent, on 100/120 Chromosorb WHP, 6 ft x 2 mm
- Confirmatory Column--5 percent SP 2401 on 100/120 Supel-coport, 6 ft x 2 mm.
- 6.3 Büchi-Brinkmann Rotary Evaporator.
- 6.4 Beroza Trap, 24/40 fitting.
- 6.5 Damon IEC Centrifuge
- 6.6 Separatory Funnel--2000 ml with Teflon stopcock.
- 6.7 Drying Column--20 mm I.D. Pyrex chromatographic column with coarse frit.
- 6.8 Boiling Flask--250-ml with 24/40 ground glass fittings.
- 6.9 Teflon Boiling Chips--10/40 mesh, pre-extracted.
- 6.10 Volumetric Glassware--flasks, pipets.

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6.11 Water bath.

## 7.0 QUALITY CONTROL REQUIREMENTS

7.1 A method blank and a laboratory control sample must be taken through the entire extraction and analysis procedure with each group of 10 to 15 samples processed. EPA-EMSL concentrates containing pesticides and PCBs are available.

7.2 After calibrating the instrument, analyze a laboratory control sample. Enter the reported values in the instrument QC log. If the results are within acceptable limits ( $\pm 2S$ ), the analysis may proceed. If the results are outside the acceptable limits, analysis should be stopped and corrective action taken.

## 8.0 PROCEDURE

### 8.1 SAMPLE EXTRACTION

8.1.1 Measure 1 liter of water into a 2 liter separatory funnel using a graduated cylinder. If only 1 liter of sample was collected, the entire amount should be used.

8.1.2 Check the pH of the sample using wide-range pH paper. If necessary, adjust the pH to within the range of 5 to 9 with sodium hydroxide or sulfuric acid.

8.1.3 Rinse the graduated cylinder and/or sample bottle thoroughly with 60 ml methylene chloride and transfer the solvent rinse to the separatory funnel.

8.1.4 Extract the sample by shaking the funnel for 2 minutes, periodically venting to release excess vapor pressure. Allow the organic layer to separate from the aqueous phase for a minimum of 10 minutes; if

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two distinct phases form, proceed to Step 8.1.5. If an emulsion forms which is more than one third the size of the solvent layers, phase separation is completed by centrifugation according to the following procedure:

- a. Place the methylene chloride layer in a centrifuge bottle.
- b. Centrifuge for 10 to 15 minutes at approximately 3200 rpm.
- c. Pour the sample into a 125 ml separatory funnel and collect the methylene chloride layer in a BOD bottle.
- d. Pour the remaining sample back into the 2-liter separatory funnel. Rinse the centrifuge bottle and the 125 ml separatory funnel with the next portion of methylene chloride and continue the extraction as in Step 8.1.6, below.

8.1.5 Collect the methylene chloride layer in a BOD bottle.

8.1.6 Add a second 60 ml volume of methylene chloride to the sample bottle and complete the extraction procedure a second time. Add the second methylene chloride layer to the first in the BOD bottle.

8.1.7 Extract with a third 60 ml portion of methylene chloride adding this extract to the first two in the BOD bottle.

8.1.8 Pour the combined extracts through a drying column containing 3 to 4 inches of anhydrous sodium sulfate and collect it in a 250 ml boiling flask.

8.1.9 Rinse the BOD bottle with methylene chloride and pour the rinse through the drying column collecting the rinse in the boiling flask.

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- 8.1.10 Place the 250-ml boiling flask on the rotary evaporator with a 30 to 36°C water bath. Evaporate to a volume of 1 to 5 ml.
- 8.1.11 Solvent exchange into hexane; add 50 ml of hexane to the boiling flask and raise the temperature of the water bath to 40 to 45°C. Evaporate to a volume of less than 10 ml.
- 8.1.12 Wash the extract from the boiling flask into a 10 ml volumetric with hexane; dilute to volume with hexane and label as the pesticide/PCB fraction.

8.2 CLEANUP AND SEPARATION

- 8.2.1 Add a weight of Florisil (nominally 21 grams) predetermined by calibration to a chromatographic column. Settle the Florisil by tapping the column; add sodium sulfate to the top of the Florisil to form a layer 1 to 2 cm deep. Add 60 ml of hexane to wet and rinse the sodium sulfate and Florisil. Just prior to the exposure of the sodium sulfate to air, stop the elution of the hexane by closing the stopcock on the chromatography column. Discard the eluate.
- 8.2.2 Adjust the sample extract to an appropriate volume and transfer it from the volumetric flask to the Florisil column. Rinse the flask with 1 to 2 ml hexane and add each rinse to the column.
- 8.2.3 Place a 250-ml round bottom flask under the chromatography column. Drain the column into the flask until the sodium sulfate layer is nearly exposed. Elute the column with 200 ml of 6 percent ethyl ether in hexane (Fraction 1) using a drop rate of about 5 ml/min. Remove the flask and set aside.
- 8.2.4 Elute the column again using 200 mls of 15 percent ethyl ether in hexane (Fraction 2); collect in a second 250 ml round bottom flask.

- 8.2.5 Perform the third elution using 200 ml of 50 percent ethyl ether in hexane (Fraction 3). The elution patterns for the pesticides and PCBs are shown in Table B-6.
- 8.2.6 Concentrate the eluates by rotary evaporation using a water bath at about 85°C. Adjust the final volume to equal the volume initially applied to the Florisil column.

8.3 ANALYSIS

- 8.3.1 Calibrate the instrument using a minimum of three calibration standards.
- 8.3.2 The following instrument conditions are used:

Temperature	175°C
Injector temperature	270°C
Injection volume	4.0 ul
Detector temperature	270°C
Run time	45 min
Carrier flow	UHP argon/methane, 50 ml/min

- 8.3.3 Record the volume injected to the nearest 0.05 ul and the resulting peak size in area units.

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TABLE B-6. DISTRIBUTION AND RECOVERY OF CHLORINATED PESTICIDES  
AND PCBs USING FLORISIL COLUMN CHROMATOGRAPHY

Parameter	Recovery (percent) by fraction		
	1 (6%)	2 (15%)	3 (50%)
Aldrin	100		
α-BHC	100		
β-BHC	97		
γ-BHC	98		
γ-BHC	100		
Chlordane	100		
4,4'-DDD	99		
4,4'-DDE	98		
4,4'-DDT	100		
Dieldrin	0	100	
Endosulfan I	37	64	
Endosulfan II	0	7	91
Endosulfan Sulfate	0	0	106
Endrin	4	96	
Endrin Aldehyde	0	68	26
Heptachlor	100		
Heptachlor Epoxide	100		
Toxaphene	96		
PCB 1016	97		
PCB 1221	97		
PCB 1232	95	4	
PCB 1242	97		
PCB 1248	103		
PCB 1254	90		
PCB 1260	95		

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8.3.4 If the peak area exceeds the linear range of the system, dilute the extract and reanalyze.

8.3.5 If measurement of the peak area is prevented by the presence of interferences, further sample cleanup is required.

8.3.6 The compounds determined by this procedure, their retention times and detection limits are shown in Table B-7.

#### 9.0 QUALITATIVE AND QUANTITATIVE DETERMINATION

9.1 Identification and quantitation is performed by comparison of the retention times and area counts noted for the sample with those of the standard.

9.2 Linear regression data are obtained by entering the standard concentrations and area counts into the Texas Instruments Programmable 58C Calculator; the slope, intercept and correlation coefficient are recorded.

9.3 The sample concentration is determined using the equation for a line,  $y = mx + b$  and solving for  $x$ :

$$x = \frac{y - b}{m}$$

where  $x$  = sample concentration, ng/ml

$y$  = area counts of the compound of interest in the sample

$b$  =  $y$  intercept

$m$  = slope

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- 9.4 The final sample concentration is determined by the following calculation:

Final concentration (ng/ml) =

$$\frac{\text{ng/ml in extract} \times \text{extract vol (ml)} \times \text{dilution factor}}{\text{initial sample volume (ml)}}$$

10.0 REFERENCE

- 10.1 Method 608, Federal Register, Monday, December 3, 1979, 40 CFR Part 136.

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TABLE B-7. GAS CHROMATOGRAPHY OF PESTICIDES AND PCBs

Parameter	Retention time (min)		Detection limit (ug/l)
	-----		
	Column 1	Column 2	
Aldrin	4.08	2.73	0.01
a-BHC	2.07	2.13	0.01
B-BHC	3.29	2.41	0.01
§-BHC	3.81	2.73	0.01
Y-BHC	2.73	2.13	0.01
Chlordane	a	a	0.04
4,4'-DDD	13.24	9.21	0.01
4,4'-DDE	8.96	5.45	0.01
4,4'-DDT	15.64	10.19	0.01
Dieldrin	9.15	7.40	0.01
Endosulfan I	7.62	6.18	0.01
Endosulfan II	13.21	10.91	0.01
Endosulfan Sulfate	13.21	10.91	0.01
Endrin	10.83	8.65	0.01
Endrin Aldehyde	17.08	17.04	0.01
Heptachlor	3.35	2.41	0.01
Heptachlor Epoxide	6.24	4.85	0.01
Toxaphene	a	a	0.40
PCB 1016	a	a	0.10
PCB 1221	a	a	0.10
PCB 1232	a	a	0.10
PCB 1242	a	a	0.10
PCB 1248	a	a	0.10
PCB 1254	a	a	0.10
PCB 1260	a	a	0.10

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<sup>a</sup> Multiply peak response, refer to Federal Register Method 608, pages 69505-69509.

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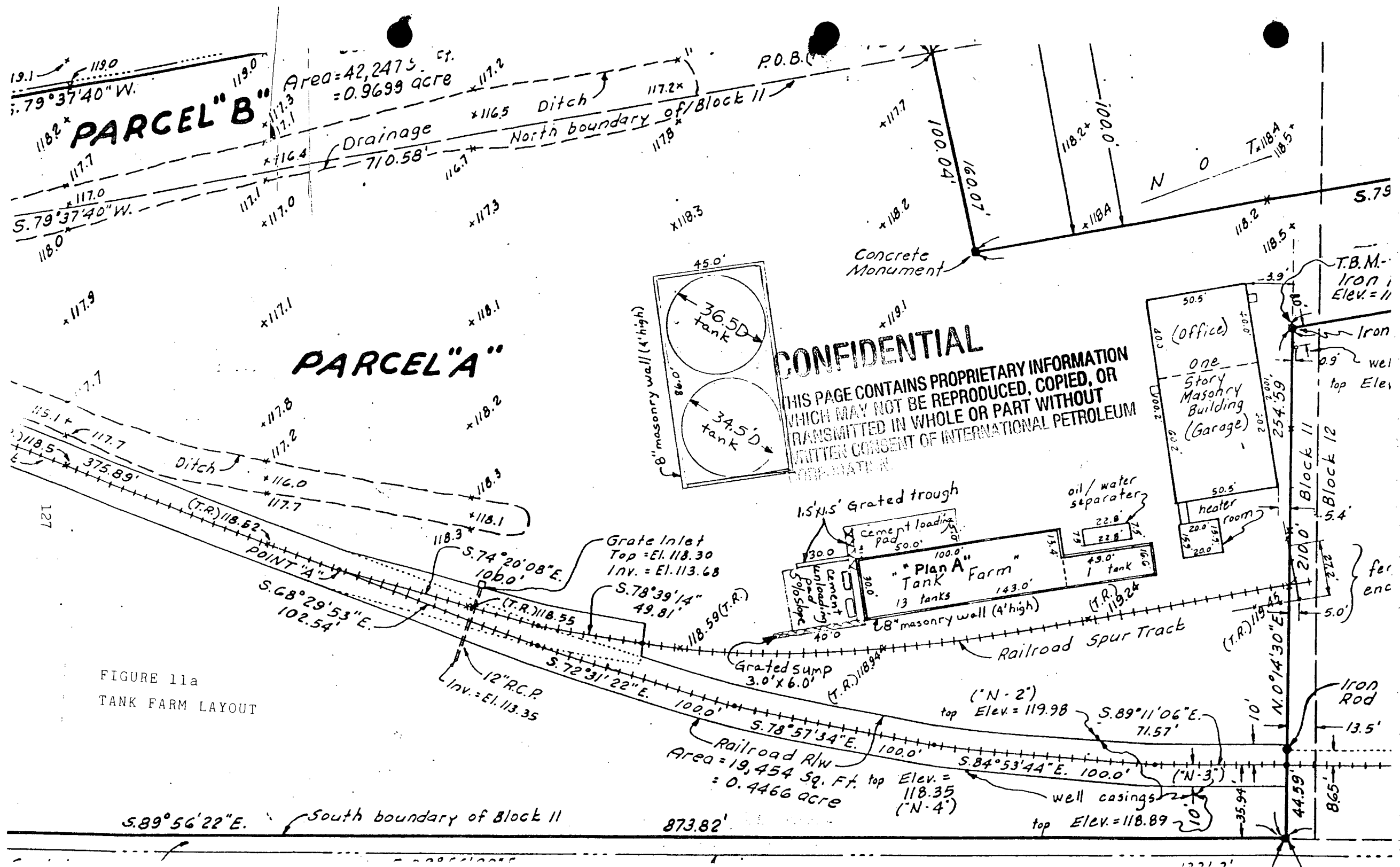
## Aerial Photograph Legend

- 1) Bulk and Finished Oil Storage
- 2) Parts and Equipment Storage
- 3) Tanker Parking
- 4) Bobtail Parking
- 5) Refueling Island
- 6) Truck Unloading
- 7) Rail Unloading
- 8) Preliminary and Analysis Storage
- 9) Secondary Storage
- 10) Demulsification and Demetalization
- 11) Percolation and Evaporation Ponds
- 12) Boiler house
- 13) Garage/Lab
- 14) Office
- 15) Blending Tanks
- 16) Processing Tanks
- 17) Desludging Operations
- 18) Oil/ Water Separation

The load bearing capacity is greater than 2500 psi, as defined in attached sheet by Central Florida Testing Labs, Inc. The surface of the roads is limerock.

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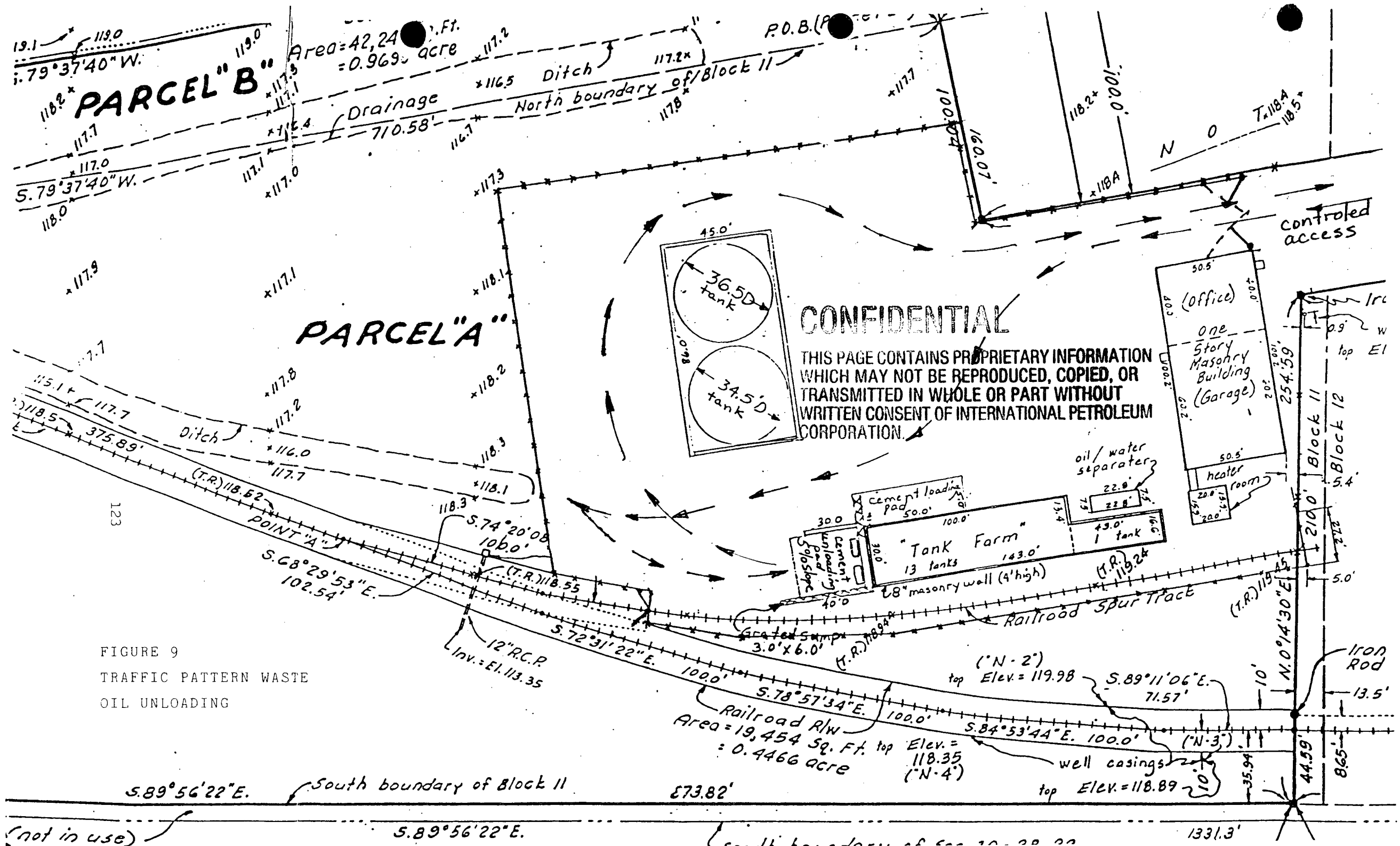


FIGURE 9  
TRAFFIC PATTERN WASTE  
OIL UNLOADING

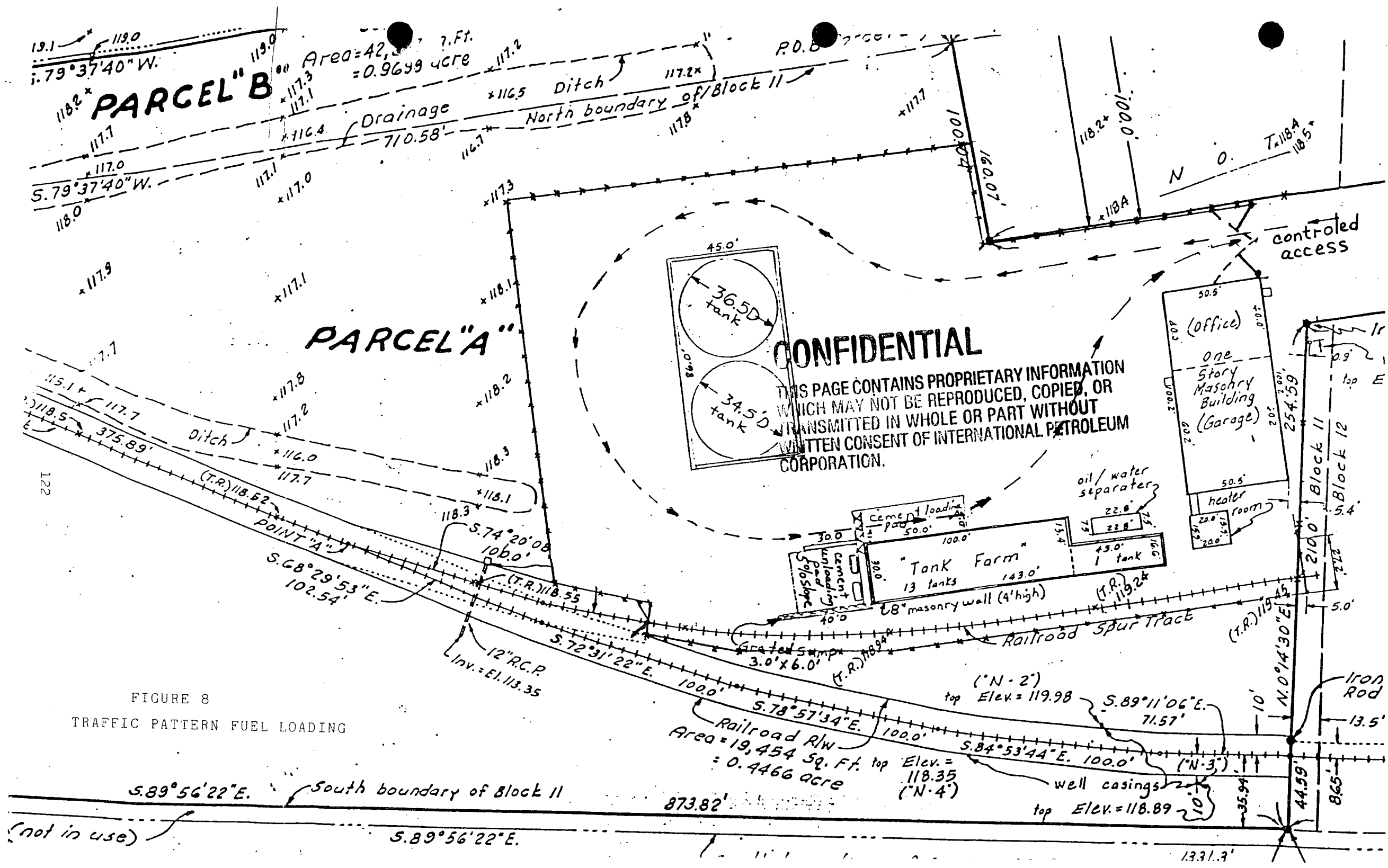


FIGURE 8

TRAFFIC PATTERN FUEL LOADING

Central Florida Testing Laboratories, Inc.

*Testing Development and Research*

NOV 30 REC'D

STARKEY ROAD

LARGO, FLORIDA 33540

PH. (813) 585-3006 - 581-7019

November 27, 1979

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Ferris Constructors, Inc.  
Post Office Box 270269  
Tampa, FL 33688

Re: National Oil Service - Plant City, FL  
Our Lab N<sup>o</sup> 14027

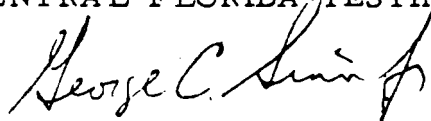
Gentlemen:

This letter is to serve as certification that our office has inspected the over-excavation of the footer line on the north end of the office building and found that satisfactory removal of deleterious materials was accomplished within the limits of excavation.

The attached soil density report indicates adequate compaction was applied to backfill soils and the construction should now safely support foundation pressures of 2500 pounds per square foot.

Sincerely,

CENTRAL FLORIDA TESTING LABORATORIES, INC.



George C. Sinn, Jr. P.E.

GCS/sbm

Attachment



Figure 11B  
Tank Farm Layout

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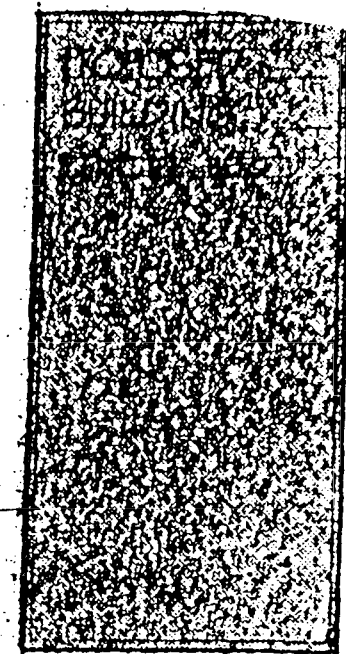
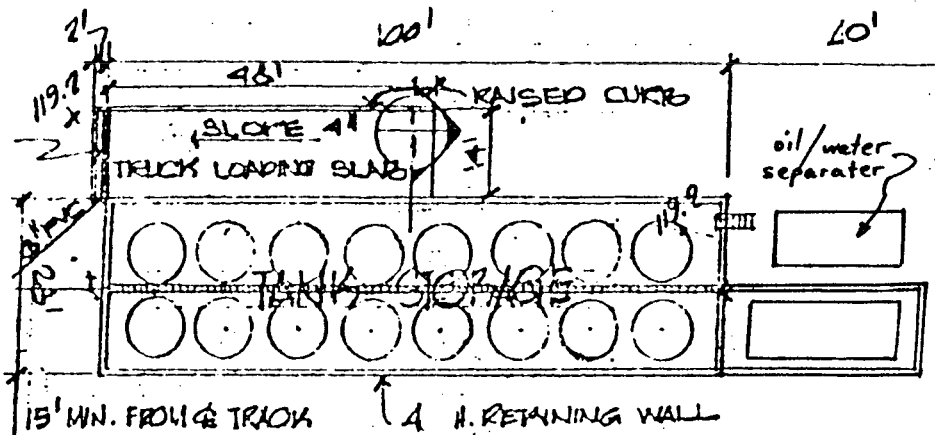
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x119.5

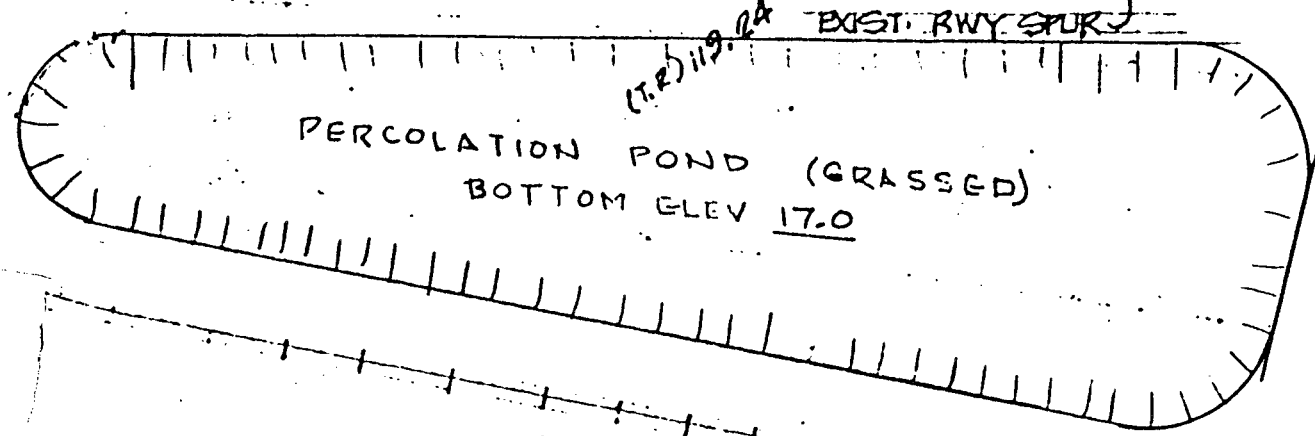
EXIST. PAVING

x119.2

TRENCH DRAIN  
W/ GRATING



F.F. EL. 119.6



N. 0° 14' 30" E.

128



# CONFIDENTIAL

## SECURITY PLAN

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### INTRODUCTION

The security measures for the plant site provide security and control of access to deny unauthorized ingress or egress, except by willful entry, and prevent entry by domestic livestock.

The coordinator/dispatcher is responsible for administering all security measures.

The entire plant site is surrounded by a 6 foot hurricane chain link fence which is topped with three strands of barbed wire. Entry is controlled at all times through a 20 foot gate to the active portion of the facility. Access is limited to the active portion of the yard by a locked gate (see Figure 10 and 14). An attendant stays with the drivers, who are not allowed to leave their vehicle, at all times. A driver's lounge is furnished as a comfort station.

At night all gates are locked. An alarm system is activated when the gates are closed. It is in effect whenever the site is unoccupied. The alarm system is inter-connected with the telephone company. In the event of unauthorized entry into the plant site via the gate/fence or into the plant buildings themselves, the police and Plant Manager are automatically alerted. The Plant Manager is on a 24 hour beeper.

Warning signs visible for more than 25 feet are posted at the gate and other locations to keep unauthorized persons out.

1. No trespassing signs are posted around the fence line at approximately 200 feet intervals.
2. Warning signs: Danger: Do Not Enter, are posted on each side of the only entrance gate.
3. Signs advising the drivers to stay in their vehicles are posted at the gate.
4. Visitor signs are posted directing them to the front office.

FIGURE 14

Secured Entry To Facility

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5. Company employees are advised to keep their vehicle out by a sign:  
No employee vehicles beyond this point (See Figure 7).

All tanks are hard piped meeting API and ASTM standards. All valves which permit direct outward flow of a tank are locked closed in non-operating or standby status. The loading and unloading connections (Kamlock fittings) of oil pipelines are padlocked with dustcaps when not in service or on a standby status for extended periods. Starter controls on all oil pumps in non-operating or non-standby modes are locked in off position or electrically isolated in the off position. At the end of each working day the main circuit breakers are cut off.

The facility is lighted with incandescent lights. The tank farm and buildings are lighted with mercury vapor or sodium vapor high intensity lights controlled by photo cells. The whole area is lit on weekends.

#### EXTERNAL SECURITY

The following measures are employed to restrict entrances and exits at the site:

1. The integrity of the chain link fence surrounding the site is maintained; it is inspected each shift.
2. Entry control at the gate consists of the following measures:
  - a) All vehicles must stop for permission to enter from the dispatcher.
  - b) All visitors must register with the coordinator and must be accompanied by company personnel while on the premises.
  - c) During shutdown at the plant, the gate is securely locked.
3. Warning signs forbidding unauthorized entry in large, block letters on a highly contrasting background are posted at the entry gate and at 200-foot intervals along the perimeter fence. Photographs of entrance with signs and security are shown in Figure 14.
4. Lighting is utilized for security purposes.

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## INTERNAL SECURITY

The following measures are employed to protect the health of employees and visitors, and to prevent the contact of these persons with hazardous materials:

1. Plant management personnel prescribe the necessary personnel protective equipment for all IPC employees and authorized visitors and identify to site employees and authorized visitors the safety procedures to be utilized at all points on site. Failure by a site employee to use the required protective clothing and equipment, or to observe necessary safety precautions in any site activity constitutes basis for dismissal.
2. Access to facilities at the plant is restricted to employees actively engaged in execution of their job duties or to visitors accompanied by an authorized representative of IPC.
3. Appropriate safety equipment is stationed at strategic locations.
4. All areas of the site in which operations occur during periods of darkness are well lighted.
5. All site employees receive job training in the handling of hazardous waste materials and the operation of site emergency equipment.
6. Signs warning of areas of possible human contact with hazardous wastes or materials are posted near all waste handling facilities. Signs are also posted showing the location of personnel decontamination equipment and emergency response equipment.
7. To prevent on-site collisions, the speed limit within the site is 5 MPH.
8. A "No Smoking" rule is enforced at designated areas.

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## GENERAL INSPECTION PLAN

### INTRODUCTION

The purpose of this General Inspection Plan is to provide a mechanism and schedule for site inspections to identify malfunctions and deteriorations, operator errors, or discharges which may lead to a release of hazardous waste constituents to the environment or a threat to human health. The objective of the inspection program is to identify problems in time for corrective measures to be undertaken and before they result in a threat to human health or the environment.

Prepared in connection with this plan are inspection forms and records which are to be filed at the site for at least three years.

### ITEMS TO BE INSPECTED

An inspection schedule of all safety and emergency equipment, security devices, communications systems, and operating and structural equipment that are important to preventing, detecting, or responding to environmental or health hazards is included with this plan. All inspections are to be performed at least once each operating shift and are to be performed under the direction of Plant Manager.

All observations made during site inspections are to be recorded on the site inspection form and signed in blue or black ink by the person conducting the inspection. All instances of improper operation, spills or other discharges, and all situations requiring remedial action will be reported immediately to the Plant Manager. Site inspection forms are to be filed in chronological order at the facility office.

### EQUIPMENT

The plant is equipped with communications and fire control equipment as described in the Contingency Plan and Emergency Procedures.

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#### TESTING AND MAINTENANCE OF EQUIPMENT

All the plant communications or alarm systems, fire protection equipment, spill control, and decontamination equipment are tested, checked, and maintained as necessary to assure their proper operation in time of emergency.

#### ACCESS TO COMMUNICATIONS SYSTEM

When hazardous materials are being handled, all persons involved in the operation must have immediate access to the communications system or be in voice contact with other employees.

#### AISLE SPACE

Sufficient aisle space to allow the unobstructed movement of personnel, fire protection equipment, and decontamination equipment to any area at the plant is to be maintained at all times.

#### DRAINAGE DISCHARGES

Contaminated water will not be allowed to drain or be pumped into drainage ditches. The perimeter drainage ditches and percolation ponds are monitored on a daily basis to assure no discharge of contaminated water has occurred.

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## 1.0 DESCRIPTION

### 1.1 DESCRIPTION OF FACILITY OPERATIONS

The primary operation of the facility consists of the dispatching of vehicles and equipment for waste oil pickup services to the petroleum industries in general. The facility buys used or waste oil directly from facilities that generate it and facilities that do not generate it such as collectors, processors, brokers and distributors (See Table 3 for Company Buying Standard). The land is presently used as a tank farm for the handling, treating and storage of raw, in process and finished oils. The raw or unprocessed oils include and are limited to: automotive crankcase oil, diesel crankcase oil, hydraulic fluid, industrial lubricants, PCB free transformer oils, and virgin fuel No.'s 2, 4, 5 and 6 oils. It specifically excludes solvents, metal working oils, rolling oils, sludges and tank bottoms from tank cleaning. These oils are provided both as supply to other facilities and sold as fuel. None is burned at this facility. Some are further processed to demetalize and dewater and then blended at the facility for burner fuel. None is disposed of as a waste.\* None is used, sold or provided to another facility for use as a non-fuel. Certain oils are blended as number 4 and 5 oil for flot for the phosphate industry.

\* Hazardous wastes have been disposed of by Rollins, Waste Control Systems and Chemical Waste Management, Inc.

#### 1.1.1 SUMMARY OF OPERATIONS

International Petroleum Corporation collects, transports, treats and stores waste oils according to State and Federal regulations as a hazardous waste management facility. It then renders the waste non-hazardous by demetalizing and blending it to ASTM, NBS, and API specifications on site. It thereby recycles the waste oil as burner fuel and flot products equivalent to or superior to virgin fuels and sells them off-site.

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PROCESS FLOW OUTLINE

Oil is collected from approximately 6000 locations like service stations, trucking terminals, auto dealerships, airports etc. It is unloaded into receiving day tanks. It is processed after certification by detailed analytical analyses. It is then stored in master batches until it is sold or transferred to other facilities. The process is detailed below. The oil collected during each day by our trucks is delivered to the plant usually after 3:00 P.M. daily. The oil collected is pumped from each truck into receiving tanks. The daily deliverable capacity of the receiving tanks seldom exceeds 25,000 gallons. The combined volume of these tanks is 60,000 gallons. This is more than adequate for day to day operation. This projects to annual sales of approximately 5 - 6 million gallons per year. The collected oil is allowed to settle overnight in the receiving tanks. The following morning the water that has settled out of the oil is pumped from the receiving tanks to the water holding tank. The remaining oil is transferred to the treat tank via the hot oil heat exchanger. The oil is heated to a temperature of 150° - 160° F. during passage through the heat exchanger and held at 140° - 150° F. in the treat tank. A proprietary set of regents are added to the hot oil. These chemicals drastically reduce the solubility of the metals in the oil and break emulsions. These metals include the wear metals like cadmium, chromium, and iron; the additive metals like barium, calcium, zinc, magnesium and sodium; and the lead from oils used in gasoline engines. As the water decreases in the oil the metallic salts grow into larger crystals which are then removed through a clarifier by polymeric addition. The heat imparted causes any residual water to drop out. As new oil is added to the treat tank, the older dry demetalized oil is pumped into various tanks in the farm for batch blending according to its specifications.

As it accumulates, the finished product is pumped into storage tanks and then it is transferred to rail tank cars or transports and shipped. The water holding tank also serves as a reservoir for oil-water separation. It also serves as a spill receiver in the

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unlikely event. Under normal operations it never exceeds the half-filled condition. After the residual oil is separated from the water by an API separator, the water is directed to waste treatment. The separated oil is returned to the process. See Figure D2B for a schematic of the aqueous treatment system.

### 1.3 PROCESSING & BULK STORAGE TANKS

All tanks are either of riveted and welded or all welded steel construction. All are ultrasonically inspected at least annually and visually inspected daily and repaired as needed to prevent rupture or other failures. All tanks are more than 20 meters (65 feet) from the facility's property line.

All tanks and tank to tank connections are hard piped and comply with ASTM & API standards. Adequate valving provides isolation of tanks and/or hard piping in case of repairs, maintenance or leaking. All facilities are padlocked closed except when in service for oil transfer.

The areas around the tanks are diked to contain any releases that could occur as a result of overflow or rupture. Figure 5 lists potential release points, accident type, control and tank size. The volumetric capacity of each contained area is sufficiently large to contain the entire contents of the largest tank located in the respective area as well as to provide at least 10 - 12 inches of freeboard. The 10 - 12 inch freeboard allows for an abnormally heavy rainfall that could conceivably occur coincidentally. The freeboard is adequate to also protect releases from any wind or wave action. The dike height is 4 feet above grade. The diked area surrounds all tanks.

The base underlying all the tanks is concrete free from cracks with a central sump and a 4' high cement block wall surrounding the pad and is sufficient to contain all discharges and accumulated precipitation until the collected material is collected, processed and

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removed. The dike surrounding the treatment tank rises to a height  $4\frac{1}{2}$  feet above grade. It will accommodate the 20,000 gallon capacity of the tank again with enough freeboard (10% excess) to allow for an unusually heavy coincidental rainfall and eliminate release to the atmosphere by wind or wave action.

#### 1.4 PROCESS AND TRANSFER OPERATIONS

The operating design of the facility provides a closed hardline tank to tank and overflow system. Thus, discharges are virtually limited to tank rupture, pumping operations, piping or hose ruptures on pump suction side.

#### 1.5 TRUCK UNLOADING AND TANK CAR LOADING

Truck unloading into receiving day tanks is conducted via hose to the suction of Pump P-1 or P-2 and thence by hardline into the respective day tanks. Each day tank has a capacity of slightly more than 10,000 gallons. They are provided with an interconnecting overflow line. Maximum daily collections are not expected to ever exceed 25,000 gallons. Further, each previous day's collection is processed before subsequent collections are transferred into these receiving tanks. Consequently, overflow spills are not ever expected to occur.

Accidental releases during making or breaking of the hose connections to the trucks are controlled by drip troughs and/or containers positioned at the truck discharge site. Additionally, a special discharge hose line fitting is provided to permit air displacement of liquid in the hose line following emptying of the truck and closure of the truck discharge valve.

Prior to loading any tank car, a careful check is made to be sure that the tank car discharge valve is closed and the cap on the car's discharge line is securely in place and sealed.

Truck unloading and tank car loading are also continuously monitored by the plant operator to forestall any unexpected event that could result in an accidental release.

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The base of the truck unloading area is concrete free of cracks or gaps. It is sloped to drain into a sump and operated to drain and remove liquids resulting from leaks, spills or precipitation. The sump has sufficient capacity to contain at least 10% of the volume of the largest containers unloaded. All spilled or leaked material and accumulated precipitation is removed from the sump in a timely fashion as necessary to prevent overflow. It is pumped into a tank and managed as a hazardous waste.

#### 1.6 OPERATING MAINTENANCE PROCEDURE

All valves permitting flow to or from the internal plant system are padlocked in the closed position when not specifically used for waste oil transfer operations. Furthermore, all the final outlets and hose connections are covered by dust caps which remain padlocked in place. It is mandatory for discharge free operation that all of the above valves and lines are unlocked immediately before use and immediately closed and locked when no longer in service.

All material transfers or product movement operations are continuously monitored by the plant operators or other appropriately trained personnel until such transfers or operations are completed. When unloading, all tank trucks are positioned so that the tank discharge valve is always over a drip-trough. This procedure catches any drippage when the dust covers are removed prior to connecting the pump suction hose to the tank truck outlet.

The drip-trough accumulations are never allowed to remain overnight. Unless required earlier, the pump of the last truck each day is used to empty the drip-trough accumulations during the time the truck is being transferred.

Upon completing the removal of oil from each truck, the truck discharge valve is closed and the air bleed valve on the suction line opened. The pump is then allowed to continue running for 1 or 2 minutes to empty the hose and pipe lines of any liquid transferred.

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Then the appropriate pump discharge valve is closed. Complete inspection of all fluid transfer lines and valves are made daily by the plant operator and the manager individually and at separate times. Any indication of leakage at joints valve bonnets, packing glands, etc. is corrected immediately.

Upon completion of transfers into tank cars, the liquid transfer valve on the suction side of the pump is closed. At the same time, the air bleed valve on the suction line is opened. The pump again is allowed to run for 1 or 2 minutes to clear the lines of any liquid. Then the appropriate pump discharge valve is closed and locked.

All valves not involved in any active transfer circuit must remain closed and locked. Careful check is made whenever a transfer circuit is changed, shut down or a new transfer circuit is started to be certain that only those valves involved in the circuit are opened and that all others are closed. This careful check is made each time before any pump is started. When pumping is finished all valves are locked closed after line clearance is completed.

Oil accumulated in the oil sump adjacent to the separator is pumped out daily or more often as required.

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FIGURE 5 Potential Spills--Prediction and Control

<u>Source</u>	<u>Major Type of Failure</u>	<u>Maximum Spill Gallons</u>	<u>Secondary Containment</u>
Above ground tank 1 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 1 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 2 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 2 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 3 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 3 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 4 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 4 Vertical	Tank Rupture	15,000	Concrete Retaining Wall

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FIGURE 5 CONTINUED  
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<u>Source</u>	<u>Major Type of Failure</u>	<u>Maximum Spill Gallons</u>	<u>Secondary Containment</u>
Above ground tank 5 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 5 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 6 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 6 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 7 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 7 Vertical	Tank Rupture	15,000	Concrete Retaining Wall
Above ground tank 8 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 8 Vertical	Tank Rupture	15,000	Concrete Retaining Wall

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FIGURE 5 CONTINUED

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Source	Major Type of Failure	Major Type of Spill	Gallons	Secondary Containment
Above ground tank 9 Vertical	Overflow		1,000	Concrete Retaining Wall
Above ground tank 9 Vertical	Tank Rupture		15,000	Concrete Retaining Wall
Above ground tank 10 Vertical	Overflow		1,000	Concrete Retaining Wall
Above ground tank 10 Vertical	Tank rupture		15,000	Concrete Retaining Wall
Above ground tank 11 Vertical	Overflow		1,000	Concrete Retaining Wall
Above ground tank 11 Vertical	Tank Rupture		20,000	Concrete Retaining Wall
Above ground tank 12 Vertical	Overflow		1,000	Concrete Retaining Wall
Above ground tank 12 Vertical	Tank Rupture		20,000	Concrete Retaining Wall

FIGURE 5 CONTINUED

<u>Source</u>	<u>Major Type of Failure</u>	<u>Maximum Spill Gallons</u>	<u>Secondary Containment</u>
Above ground tank 13 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 13 Vertical	Tank Rupture	24,000	Concrete Retaining Wall
Above ground tank 14 Vertical	Overflow	1,000	Concrete Retaining Wall
Above ground tank 14 Vertical	Tank Rupture	14,000	Concrete Retaining Wall
Above ground tank T-150 Vertical	Tank Rupture	210,000	Concrete Retaining Wall
Above ground tank T-83 Vertical	Tank Rupture	210,000	Concrete Retaining Wall

Total Capacity

648,000

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2.0

EMERGENCY COORDINATOR

For the purposes of this Contingency Plan, the Plant Manager has designated the chief-on-site Emergency Coordinator. In his absence, and to assure 24-hour coordination, other personnel are designated as Deputy Emergency Coordinators (Exhibit 1). Whenever practical, the Deputy Emergency Coordinator on duty at the time of any emergency shall attempt to contact the Chief Emergency Conductor. In any event, the responsibility for coordinating all emergency response measures will be with the Chief or Deputy Emergency Coordinator on duty at the time of the emergency. The Emergency Coordinator on duty at the time of the emergency is authorized to direct all plant personnel in responding to an emergency situation and making the necessary decisions for appropriate response and action. A list of emergency phone numbers is in the front of this document. A list is also posted in the window by the front door outside the gate to shorten response time for outsiders in case of an emergency.

The coordinators shall be thoroughly familiar with all aspects of this contingency plan, all operations at the plant site, the location and characteristics of all wastes which are handled and stored, the location and contents of product storage areas, the locations of manifests and other records, and the locations and use of all fire control equipment, decontamination equipment, first aid and emergency equipment, vehicles and motorized equipment.

EXHIBIT 1.

COORDINATORS ON-SITE		PHONE #	OFF-SITE	PHONE #
Chief	Garry Allen	754-1264	Jerry Blair	305-989-4000
Deputy	Frank Shibetti	546-9230	Richard Lane	504-288-1077
Deputy	Murphy Shepherd	754-1264	Charlie Hunt	404-794-0325
Deputy	Fran Chewning	644-4956		

3.0

EMERGENCY RESPONSE TEAM

All operations personnel for each shift will comprise the Emergency Response Team and will be under the authority of the Emergency Coordinator.

The Emergency Coordinator will determine whether additional assistance is needed from local fire or police units or other emergency response agencies.

#### 4.0 EMERGENCY EQUIPMENT

Portable UL approved ABC fire extinguishers are strategically located throughout the site as designated and approved by the Fire Insurance Underwriters Assoc. The Plant Manager is responsible for ensuring that all extinguishers are inspected and maintained in accordance with standard safety guidelines and that the locations and maintenance are in accordance with requirements of Fire Control Codes. A water fire protection system capable of pumping approximately 400 gpm with enough hose to service all plant facilities is functional at all times. A backup system supported by shallow wells is also available in the event the city water which normally services the fire protection system, is non-operable.

The plant intercom system and telephones are to be used for an alarm and communications system.

Clean-up equipment and decontamination equipment including vehicle-mounted pumps and tanks, vacuum trucks, loading and excavation equipment, oyster shell and other items are available on short notice. Oil sorbents are always kept in the ditches that drain from the site.

#### 4.1 SAFETY SHOWERS, EYE BATHS AND FIRST AID

A permanent safety shower and eye bath station is located on the site in accordance with an OSHA survey and recommendations. A first aid station is maintained at the Administration Building. A change room and showers are also located in the Administration Building. The Plant Manager serves as Safety Director. He is responsible for training all plant personnel in the use of safety devices and first aid. All personnel attend a safety and first-aid training session at least once a month.

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The Safety Director maintains an inventory of personnel protective equipment including, but not limited to the following:

- A. Respirators, both cartridge and air
- B. Safety glasses and safety shields
- C. Hard hats
- D. Acid suits
- E. Rubber boots and gloves

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#### 4.1.2 EQUIPMENT AND MATERIALS

- 1) One 3" gasoline powered pump - approximately 320 gpm capacity.
- 2) One 2" gasoline powered pump - approximately 150 gpm capacity.
- 3) Five 2000 gallon capacity pump trucks, capable of pumping rates of approximately 100 gpm each.
- 4) Two 3000 gallon capacity pump trucks, capable of pumping rates of approximately 100 gpm each.
- 5) One hundred fifty feet of 3" suction hose.
- 6) Three hundred feet of 2" suction hose.
- 7) Twenty-five 40 lb. bags of oil absorbent for emergency diking.
- 8) Three shovels.
- 9) One wheelbarrow.
- 10) Seven cubic yards of sand and seven cubic yards of shell stock-piled adjacent to the major operating area.

#### 4.1.3 EXTERNALLY AVAILABLE EQUIPMENT AND MATERIALS

- 1) Lee Kirk & Sons --- various 2000 and 3000 vacuum trucks.
- 2) B.F.I. Inc. --- similar equipment to above.
- 3) Numerous Septic Tank Cleaning Services --- various skid mounted tanks, as well as inflatable rubber tanks.
- 4) Barron's Excavating and Hauling Services --- bulldozers, dump trucks and other earth moving equipment.
- 5) Several equipment rental firms with available pumping equipment and hand tools.
- 6) Available borrowed labor from neighboring plants (approximately 5 to 10 men).
- 7) Need-A-Driver, Inc., Tampa, FL available on two (2) hours notice.

## 5.0 RESPONCE AND RECOVERY

Whenever there is an imminent or actual emergency situation such as a fire, explosion of discharge or release of hazardous materials which has the potential for damaging human health or the environment, the alarm system shall immediately be activated and the Emergency Coordinator notified.

Where applicable, the Emergency Coordinator shall activate the internal communications system to notify all personnel of the imminent or actual emergency situation.

The Emergency Coordinator will immediately assess the nature of the emergency and direct the appropriate response of the Emergency Response Team. He will dispatch personnel and equipment to the scene of the emergency and direct all emergency response activities.

At his discretion, the Emergency Coordinator shall determine what additional assistance may be required and shall notify appropriate State, Federal, and local agencies should an emergency at the site present a potential threat to surrounding areas.

In the event of a discharge or release of hazardous materials, the Emergency Coordinator will determine the character, amount, and the extent of any released materials. He will do this by observation, on the basis of his personal knowledge of the materials and their source or on the basis of his review of records and manifests. If necessary he will communicate with IPC technical personnel in Atlanta, GA, New Orleans, LA and Hallandale, FL for suggested precautions.

Concurrently, the Emergency Coordinator will assess the possible hazards to human health or the environment that may result from the fire, explosion or release. He must consider both direct and indirect effects of the emergency and, in cooperation with appropriate authorities, he shall assess the possible imminent hazard to local

communities associated with a discharge. This assessment shall include consideration of indirect effects, such as toxic, irritating, or asphyziating gases, hazardous surface runoff due to water or chemical agents used to control fire, and heat-induced explosions.

The Emergency Coordinator will determine whether evacuation of local areas is advisable and, if so, he will immediately notify local authorities and, in cooperation with them, assist in the evacuation.

The Emergency Coordinator must take all reasonable measures to ensure that the emergency situation does not recur or spread to other hazardous materials. These measures shall include the cessation of all plant operations during the emergency, removal or isolation of containers, vehicles, tanks, tank trailers, etc.

The Emergency Coordinator will provide for the proper handling and disposal of recovered material resulting from the emergency. This includes contaminated soil, waste, contaminated water, or other material. The recovered material, contaminated soil or contaminated water will be considered a hazardous waste to be disposed at a permitted off-site disposal facility.

The Emergency Coordinator is responsible for the proper handling and segregation of incompatible materials and ingitable materials. All equipment clean-up and decontamination activities will be supervised by the Emergency Coordinator. All equipment items will be restored to pre-accident condition, inspected, and returned to their respective locations.

Following the emergency situation, the Emergency Coordinator or his designee will inspect tanks, valves, pipelines, couplings and/or other equipment to monitor for potential ruptures, leaks, pressure buildups, or gas generation, wherever it is appropriate.

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A preliminary written report of the emergency shall be prepared by the Emergency Coordinator and sent to the Regional Administrator, Region IV, Environmental Protection Agency. The report is to be written with the assistance of Company management, on Company stationery, and is to include the following:

1. The name, address and telephone number of the operator of the plant site and the location of the plant site where the incident occurred;
2. Date, time and type of incident (fire, explosion or release or discharge);
3. Name and quantity of material or materials involved;
4. The extent of injuries, if any;
5. An assessment of actual or potential hazards to human health or the environment, where this is applicable;
6. The estimated quantity and disposition of recovered material that resulted from the incident.

Plant management and company representatives will review the circumstances of the emergency, recommend, and implement the necessary adjustments in plant operations to minimize the potential for additional emergencies.

#### 6.0 CONTROL PROCEDURE FOR FIRE, EXPLOSION OR RELEASE OF HAZARDOUS WASTE

In the unlikely event that a fire and/or explosion should occur thereby releasing oil, the tanks are physically located far enough apart to prevent spreading of fire from one tank to another by movement of the released oil or explosion of the oil. If oil has to be removed from tanks in near proximity during an emergency it will be either pumped to tankage on the other side of the property or to rail tankers available to IPC.

Additionally, within one week IPC can move its total volume at the Tampa facility to its other locations in New Orleans, LA and Atlanta, GA.

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#### 6.1 CONTROL PROCEDURE IN CASE OF FLOOD

Even though the property is not in the 100 year flood plain, flood potential has been considered. In the extremely unlikely event of a flood, IPC can pump all of its tanks full of water in approximately 3 hours. Since the oil is lighter than water, it will float and the water at the bottom of the tanks will keep the tanks from tearing loose from their supports and floating off or releasing hazardous waste. Once the flood is over and activity on the site can resume, the water can be separated and treated with normal processing technology.

#### 7.0 MANAGEMENT

It is the Plant Manager's responsibility, in coordination with other plant and company personnel, to identify needs and potential problem areas, plan, schedule, and organize all elements of the contingency plan and other emergency response procedures. Records are to be maintained to provide immediate assessments of personnel training, condition of plant operations and emergency equipment, and maintenance requirements. The Plant Manager must also maintain inspection reports, safety meeting minutes, and lists of applicable numbers.

All safety and emergency equipment is to be inspected once each week. The Emergency Coordinator will file a weekly report stating the condition of the equipment and whether any repairs or replacements are needed.

All fences and other security devices pumps, curbed areas and tanks associated with the product storage area, the wastewater facilities, and the active facilities area are to be inspected daily to determine the presence of leaks, pump failures, volume of waste, or other circumstances which necessitate immediate attention. A report is to be filed for each inspection.

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EMPLOYEE TRAINING

The primary intent of this contingency plan is to minimize hazards to health or environment by familiarizing all facility personnel with a planned course of action should an emergency develop. The contingency plan is not intended to replace the IPC regularly scheduled Safety Training Program, but rather is intended to supplement the current program of continuous safety preparedness and management.

In the interest of personnel familiarization, the Emergency Coordinator shall be responsible for a thorough review of this plan with all facility employees on a quarterly basis in the scheduled monthly safety meetings. Furthermore, the Emergency Coordinator shall be responsible for staging periodic unscheduled drills to test the preparedness of facility personnel and to file a report in the facility records.

The company shows a series of training films on spill prevention control and cleanup in their monthly safety meetings. It has personnel certified in hazardous waste cleanup and spill prevention by Texas A & M.

All operations and maintenance employees are to receive adequate training in plant operations, safety and occupational health procedures, first aid practices, and fire control and emergency procedures. Office personnel will receive instruction for evacuation plans or support services that might be provided in the event of an emergency.

Training will be initiated upon employment and will be a continuous process. All new employees will receive orientation training from the Safety Director. The training will consist of an introduction to plant operations, the use and location of equipment, the use and location of showers, eye baths, respirators, fire control equipment, first aid devices, etc.

Films, demonstrations and practice sessions will be important methods of instruction.

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Specific information conveyed to employees during the training program will include, but not be limited to, the following:

- 1) A comprehensive review of the location and proper operation of all emergency equipment including fire extinguishers, water connections, communications system, alarm system, and safety showers.
- 2) A review of evacuation routes leaving the facility and leaving the area.
- 3) A review of the procedure for notification of the Emergency Coordinator on duty.
- 4) A review of the proper voice alarm which will be sounded immediately upon discovery of an emergency.
- 5) General group discussions (during quarterly meetings) concerning potential emergency situations, necessary immediate actions, and hypothetical emergency situations and responses for employees.

#### 9.0 EVACUATION

Should an emergency occur, it may be necessary to evacuate employees or contractor personnel from the facility. The Emergency Coordinator on duty shall decide upon and direct the evacuation of appropriate areas of the facility. Should the emergency be severe enough to necessitate an evacuation, the Emergency Coordinator will coordinate with the Emergency Response Agencies to determine the necessity of evacuating nearby residents and proceed under their direction.

While the methods specified for handling hazardous materials at this facility minimize the possibility for any severe emergency to occur, it is the responsibility of the Emergency Coordinator to provide a copy of this plan, including the evacuation route to the appropriate Emergency Response Agencies. It is important to emphasize that the decision to evacuate any local residents should be made only by the Emergency Response Agency in charge after consulting with the facility Emergency Coordinator to determine the full extent of the emergency. It is not the intent of this plan to cause unwarranted alarm by providing an evacuation plan, but rather to be prepared for any and all emergencies.

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#### 10.0 ARRANGEMENTS WITH LOCAL AUTHORITIES

Arrangements have been made to inform representatives of local fire and police departments of plant layout, location of possible hazards, emergency equipment location and operation, the evacuation plan and route, and other critical information.

Arrangements have also been made to familiarize hospitals with the types of injuries or illnesses which could result from fires, explosions or releases at the plant. Inasmuch as the IPC area is surrounded by industrial plants, hospitals and resident physicians are knowledgeable of treatment methods resulting from accidents at such industries. Nevertheless, the Plant Manager should inform company physicians of the necessity to make arrangements with nearby hospitals.

Where State or Local authorities decline to cooperate or enter into such arrangements, the Plant Manager is to document their refusal.

#### 11.0 WASTE ANALYSIS PLAN

Each lot of material considered by I.P.C. is analyzed whether it is utilized or not. Further each lot disposed of by the facility is analyzed to characterize it. All lots are analyzed according to Mil-F-859-E. Special analyses are done by commercial laboratories as needed. A summary of test methods occurs in Tables 1 and 2.

#### 11.1 WASTE GENERATION

Wastes that are generated at the facility are the result of washing the outside surface of truck-tractor units, empty tank trailer and other equipment; precipitation and residual water dropped from oil lots. Occasionally the interior of a tank trailer may be washed, but it is rarely done on site. Normally it is done by an outside cleaning contractor. The resulting water is collected at a sump which is contained within a concrete pad. It is then pumped to a steel tank for storage and processes through an API oil/water separator and disposed of after analyses to assure compliance with all existing industrial

waste water criteria. In all cases, the identity of material that may be contained is known. Characteristics of the waste water are known on the basis of waste, source sampling and on the basis of manifests which accompany the transportation of the materials.

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Table 1. Test Method

<u>TEST</u>	<u>ASTM</u>
Flash point	D93
Pour Point	D97
Water and Sediments (1-4 grade fuels)	D1796
Water (5 & 6 grade fuels)	D95
Viscosity	D86 or D445
Gravity, API	D287
Sulfur	D1552, D129, D1551 or D2662

Table 2. Summary of oil analyses

<u>Property of Test</u>	<u>Oil</u>
Viscosity (SUS @ 40° C)	90 - 850
API Gravity (60° F)	10 - 34
Water % by Volume	0 - 10
BS & W % by Volume	0 - 11
Flashpoint (°F)	140 - 700
Heating Value (BTU/lb)	13000 - 19600
Chloride (% by wt)	< 0.1
Sulfur (% by wt)	0.1 - 2.1
Zinc ppm	10 - 1089
Chromium ppm	0 - 20
Calcium ppm	2 - 2200
Barium ppm	tr - 3650
Phosphorous ppm	0 - 50
Silica ppm	20 - 90
Iron ppm	tr - 1200
Aluminum ppm	tr - 150
Lead ppm	0 - 1000
Vanadium ppm	4 - 150

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# INTERNATIONAL PETROLEUM CORPORATION

## COMPANY BUYING STANDARD

LOTS SHALL NOT EXCEED:

### PRIMARY METAL STANDARD

	<u>ppm</u>
arsenic	5.
barium	3000.
cadmium	5.
chloride (organic plus inorganic)	1000.
chromium	5.
cyanide	5.
calcium	2500.
iron	2500.
lead	1000.
mercury	0.2
selenium	1.
sulfur	-----
zinc	1500.
PCB	50.

### PRIMARY HERBICIDES AND PESTICIDES

endrine	0.02
lindane	0.4
methoxychlor	10.
toxaphene	0.5
2,4 D	10.
2,4,5 TP Silvex	1.

### SECONDARY HERBICIDES AND PESTICIDES

aldrin and dieldren	0.003
chlorodane	0.01
DDT	0.001
Demeton	0.1
Endosulfan	0.003
Guthion	0.01
Heptachlor	0.001
Malathion	0.1
Mirex	0.001
Parathion	0.04
phthalate esters	3.

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105 South Alexander Street, Plant City, Florida 33566  
Area Code (813) 229-1739 Fla WATS 800-282-9585

#### 11.2 SAMPLING METHOD

Prior to disposal, representative samples of waste water are removed from the tank at various levels. These are then combined as a composite. Compability of the waste water is then determined. Its reaction or evolution of gas resulting from the addition of acid to a portion of the sample to pH less than 1 and the addition of soda ash to another portion of the sample until the pH of the mixture is approximately 13 is determined. On the basis of these observations; already knowing the chemical characteristics of the water, sufficient information regarding the waste is obtained for proper treatment and disposal.

#### 11.3 FREQUENCY OF ANALYSIS

The contents of each storage tank are subjected to the analytical tests and observations prior to disposal. Bench scale tests are performed each time a lot of waste water is disposed of.

#### 11.4 PARAMETERS ANALYZED

Prior to disposal, the following observations and analyses are made on the waste water:

1. Water Solubility
2. Flashpoint
3. Odor
4. Color
5. Number of Phases
6. TSS and TDS
7. Viscosity
8. Volume
9. pH, Temp., Conductivity
10. Reactivity with Air and Water
11. Oil and Grease
12. Heavy Metals included Lead and Cadmium
13. Pesticides/Herbecides included Lindone, Methoxyclor, Toxaphene, 2, 4, D, 245TP Silvex and Endrine

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Since the waste water is mostly water, it is normally within acceptable discharge limits. In the unlikely event it is not, it is treated, solar evaporated or shipped to a hazardous waste disposal site properly equipped to handle the waste.

11.5 RECORD KEEPING AND ADMINISTRATION

Careful records including bills of lading, manifest and sources and identities of wastes are maintained. Sample and analysis results and a list of contaminants and their source are included with each shipment.

12.0 POST EMERGENCY EQUIPMENT MAINTENANCE

All equipment used and/or involved either superficially or directly with the emergency must be cleaned and gone through to ensure its fitness for its intended use before normal facility operations are resumed. All solvents, protective clothing and equipment used or damaged as the result of the emergency must be replaced or fixed within two weeks in order to assure adequate response to future mishaps.

13.0 SECURITY PLAN

13.1 INTRODUCTION

The security measures for the plant site must provide security and control of access to deny unauthorized ingress or egress, except by willful entry, and prevent entry by domestic livestock.

The coordinator/dispatcher is responsible for administering all security measures.

13.2 EXTERNAL SECURITY

The following measures are employed to restrict entrances and exits at the site:

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- 13.2.1 The integrity of the chain link fence surrounding the site will be maintained; it is to be inspected each shift.
- 13.2.2 Entry control at the gate will consist of the following measures.
- a) All vehicles must stop for permission to enter from the dispatcher.
  - b) All visitors must register with the coordinator and must be accompanied by company personnel while on the premises.
  - c) During shutdown at the plant, the gate is securely locked.
- 13.2.3 Warning signs forbidding unauthorized entry in large, blocked letters on a highly contrasting background are posted at the entry gate and at 200-foot intervals along the perimeter fence.
- 13.2.4 Lighting is utilized for security purposes.

13.3 INTERNAL SECURITY

The following measures will be employed to protect the health of employees and visitors and to prevent the contact of these persons with hazardous materials:

1. Plant management personnel will prescribe the necessary personnel protective equipment for all BFI employees and authorized visitors and will identify to site employees and authorized visitors the safety procedures to be utilized at all points on site. Failure by a site employee to use the required protective clothing and equipment, or to observe necessary safety precautions in any site activity constitutes basis for dismissal.
2. Access to facilities at the plant is restricted to employees actively engaged in execution of their job duties or to visitors accompanied by an authorized representative of BFI.
3. Appropriate safety equipment is to be stationed at strategic locations.
4. All areas of the site in which operations may occur during periods of darkness are to be well lighted.

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5. All site employees are to receive job training in the handling of hazardous waste materials and the operation of site emergency equipment.
6. Signs warning of areas of possible human contact with hazardous wastes or materials are to be posted near all waste handling facilities. Signs are also to be posted showing the location of personnel decontamination equipment and emergency response equipment.
7. To prevent on-site collisions, the speed limit within the site is 5 MPH.
8. A "No Smoking" rule is to be enforced at designated areas.

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D. E. R.

MAY 29 1984

SOUTH WEST DISTRICT  
TAMPA



# NOTE:

1. Elevations shown are in feet and refer to N.O.S. datum (0.00 = mean sea level).  
T.B.M. No. 1 is top railroad spike at southerly corner of tract on the West R/W line of Alexander Street, Elevation = 119.38.

2. (T.R.) with elevation indicates elevation refers to top of north railroad rail.

3. Some scattered patches of asphalt pavement and limerock base on property not measured.

4. Existing fences not shown.

## LEGEND

F.R.R.S. = Found Railroad Spike  
F.P.K.N. = Found P.K. Nail  
F.I.P. = Found Iron Pipe  
U.P.L. = Utility Pole w/Light  
U.P. = Utility Pole  
P.P. = Power Pole  
-P/- = Power & Telephone Lines  
-P- = Power Line  
-T- = Telephone Line  
G.W. = Guy Wire & Anchor

### Parcel "A":

Parts each of Blocks 11 and 12 of EAST FOREST PARK Subdivision, according to map or plat thereof recorded in Plat Book 7, Page 14, public records of Hillsborough County, Florida, described as follows: From the Southeast corner of said Block 12, run North 89°56'22" West along the South boundaries of said Blocks 12 and 11 a distance of 435.0 feet to a point-of-beginning; From said point-of-beginning, run North 0°14'30" East, parallel to and 435.0 feet West of the East boundary of said Block 12, a distance of 254.59 feet; run thence North 79°36'30" East a distance of 394.0 feet to a point on the Westerly right-of-way line of Alexander Street (State Road No. 39-A) (Note: This course, if extended an additional 48.6 feet, intersects the East boundary of said Block 12 at a point 334.7 feet North of the Southeast corner of said Block 12); run thence North 5°14'00" West along said Westerly right-of-way line of Alexander Street a distance of 65.02 feet to a point which is 200.0 feet (measured at right angles) Southeasterly from the centerline of the Seaboard Coast Line Railroad right-of-way; run thence South 79°37'40" West, parallel to and 200.0 feet Southerly of said centerline of Seaboard Coast Line Railroad right-of-way, a distance of 545.03 feet to a point which is 600.0 feet on a bearing of South 79°37'40" West from the East boundary of said Block 12; run thence North 12°10'30" West a distance of 100.04 feet to a point on the Northerly boundary of said Block 11, which point is 621.88 feet on a bearing of South 79°37'40" West from the Northeast corner of said Block 12; run thence South 79°37'40" West along the Northerly boundary of said Block 11, parallel to and 100.0 feet Southerly of said centerline of Seaboard Coast Line Railroad right-of-way, a distance of 710.58 feet to the Northwest corner of said Block 11, which point is 7.5 feet East of the West boundary of said Block 12; run thence North 0°03'40" West along the West boundary of said Block 11, parallel to and 7.5 feet East of the West boundary of said Southeast 1/4 of the Southeast 1/4 of Section 30, Township 28 South, Range 22 East, Hillsborough County, Florida; run thence South 0°03'40" West along the West boundary of said Block 11, parallel to and 7.5 feet East of the West boundary of said Southeast 1/4 of the Southeast 1/4 of Section 30, a distance of 261.12 feet (measured) (255.0 feet by plat) to the Southwest corner of said Block 11; run thence South 89°56'22" East along the South boundary of said Block 11, parallel to and 7.5 feet North of the South boundary of said Southeast 1/4 of the Southeast 1/4 of Section 30, a distance of 873.82 feet to the point-of-beginning; Easement: That part thereof lying within 10.0 feet either side of a centerline, said centerline being described as follows: From the Northwest corner of said Block 11, run North 0°03'40" East along the Northerly extension of the West boundary of said Block 11 a distance of 24.97 feet to the point-of-beginning of said centerline being described hereby: From said point-of-beginning, run South 71°33'58" East a distance of 65.67 feet; run thence South 69°09'55" East a distance of 375.89 feet to a point hereby designated as Point "A"; run thence South 68°29'53" East a distance of 102.54 feet; run thence Southeasterly along a curve to the left, which curve is along the centerline of the existing railroad tracks, and which curve is sub-tended by the following described chords: South 72°31'22" East 100.0 feet; South 78°57'34" East 100.0 feet; South 84°53'44" East 100.0 feet; South 89°11'06" East 71.57 feet to a point which is 35.94 feet North of the South boundary and 13.5 feet West of the East boundary of said Block 11, and which point is the termination of said centerline; Also: That part thereof lying within 10.0 feet either side of the following described centerline: Beginning at aforementioned Point "A", run South 74°20'08" East 100.0 feet; run thence South 78°39'14" East a distance of 49.81 feet to the termination of said centerline.

The above described parcel contains 307,318 square feet, or 7.0550 acres.

### Parcel "B":

A tract in the Southeast 1/4 of the Southeast 1/4 of Section 30, Township 28 South, Range 22 East, Hillsborough County, Florida, described as follows: From the Northeast corner of Block 12 of EAST FOREST PARK Subdivision, according to map or plat thereof recorded in Plat Book 7, Page 14, public records of Hillsborough County, Florida, run South 79°37'40" West along the Northerly boundaries of Blocks 12 and 11 of said EAST FOREST PARK Subdivision, parallel to and 100.0 feet South of the centerline of the Seaboard Coast Line Railroad right-of-way, a distance of 621.88 feet to a point-of-beginning; From said point-of-beginning, run North 12°10'30" West a distance of 80.03 feet to a point which is 40.0 feet (measured at right angles) Southeasterly from the centerline of the Seaboard Coast Line Railroad right-of-way and 635.0 feet on a bearing of South 79°37'40" West from the Northerly extension of the East boundary of said Block 12; run thence South 79°37'40" West, parallel to and 40.0 feet Southerly of the centerline of said Seaboard Coast Line Railroad right-of-way, a distance of 697.64 feet to a point on the Northerly extension of the West boundary of said Block 11; run thence South 0°03'40" West along said Northerly extension of the West boundary of Block 11 a distance of 61.01 feet to the Northwest corner of said Block 11; run thence North 79°37'40" East along the North boundary of said Block 11, parallel to and 100.0 feet South of said centerline of Seaboard Coast Line Railroad right-of-way, a distance of 710.58 feet to the point-of-beginning; Easement: That part thereof lying within 10.0 feet either side of a centerline, said centerline being described as follows: From the Northwest corner of said Block 11, run North 0°03'40" East along the Northerly extension of the West boundary of said Block 11 a distance of 24.97 feet to the point-of-beginning of said centerline being described hereby: From said point-of-beginning, run South 71°33'58" East a distance of 65.67 feet, more or less, to a point on the North boundary of said Block 11 and the termination of said centerline (the termination of said centerline and the boundaries of said excepted tract being on the North boundary of said Block 11).

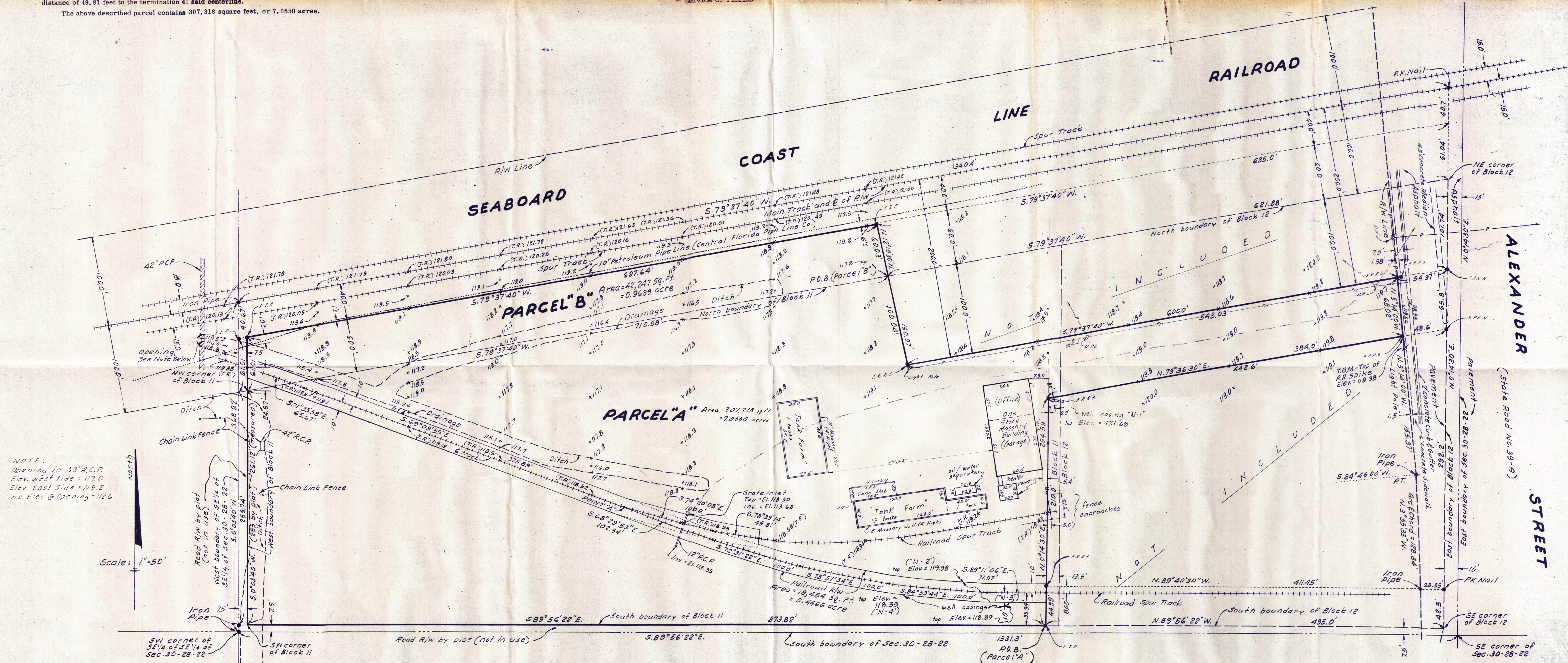
The above described parcel contains 42,247 square feet, or 0.9699 acre.

NOTE: This sketch is a copy of the sketch prepared for the survey of this property dated June 29, 1979 (revised August 23, 1979 and August 30, 1979) modified to show specific, limited data as requested (location and elevation of four wells; location and size of office/garage building; outline of "Tank Farm"). This is not a complete resurvey, and changes from previous survey, other than indicated, are not shown.

March 16, 1983  
April 18, 1983 - corrected location of well casing "N-2"  
January 14, 1984 - Update Survey  
for: National Oil  
Service of Florida

PIMM-WOODS ENGINEERING CO.

Maurice C. Pimm  
Maurice C. Pimm  
Reg. Surveyor No. 1001



NOTE:  
Opening in 42" R.C.P.  
Elev. West Side = 117.0  
Elev. East Side = 115.2  
Inv. Elev. @ Opening = 116.6

Scale: 1"=50'

PIMM - WOODS ENGINEERING CO.

CIVIL ENGINEERS - LAND SURVEYORS  
2906 N. FLORIDA AVE., TAMPA, FLORIDA 33602 (813) 223-1747