



STATE OF WASHINGTON
DEPARTMENT OF ECOLOGY

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May 4, 1990

TO: Kim Anderson
FROM: Marc Heffner
SUBJECT: U.S. Oil and Refining Company June 13-14, 1989 Class II Inspection Report

INTRODUCTION

A Class II inspection was conducted at U.S. Oil and Refining Company (U.S. Oil) on June 13-14, 1989. The inspection was conducted by Pat Hallinan, Carlos Ruiz, and Marc Heffner of the Ecology Compliance Monitoring Section. Representing U.S. Oil were Bill Dabrock, Manager of Administrative Services, and Shauna-May Tecle-Mariam, Laboratory Manager. An April 11, 1989 a tour of the facility was provided by U.S. Oil for inspection planning.

Objectives of the inspection included:

1. Assess plant compliance with NPDES permit effluent limits.
2. Characterize effluent toxicity with priority pollutant scans and bioassays.
3. Review lab procedures to determine conformance with standard techniques. Samples will be split for Ecology and U.S. Oil analysis of permit parameters.
4. Characterize return activated sludge, dewatered digested sludge, and cake from centrifuged effluent with priority pollutant scans.

The refinery, located in Tacoma, processes approximately 30,000 barrels of crude oil daily. Products include gasoline, jet fuel, diesel fuel, bunker oil, and asphalt.

Wastewater is discharged into a shared Port of Tacoma drain pipe flowing into the Lincoln Avenue Ditch. The Ditch flows into the Blair Waterway. The U.S. Oil discharge is limited by NPDES Permit No. WA 000178-3.

Wastewater treatment at U.S. Oil includes both physical and biological units. The physical processes for oil/water separation include a series of three units: an API separator, a corrugated plate separator, and induced air flotation. The biological unit is an Orbal oxidation ditch system with a secondary clarifier. Flow is measured with an in-line meter in the discharge box area. Waste activated sludge is aerobically digested, dewatered, then land applied on site.

Holding ponds and piping options are an important part of the treatment system. Holding tanks/ponds include:

The back pond - a 120,000 gallon concrete pond where asphalt production wastewater is routed and other wastewater can be routed.

The off spec ponds - two 250,000 gallon lined ponds used to hold off spec products or other wastes which must be bled into the treatment system slowly to avoid upsetting the process.

One 1,000,000 gallon lined pond - this pond is used for stormwater retention. Also, empty crude oil or product storage tanks can be used to hold excess stormwater.

The contents of the tanks/ponds can be introduced into the treatment process at the proper point to provide adequate treatment. If treatment provided is inadequate, the piping network allows the effluent to be routed back to one of the ponds/tanks, then re-run through the treatment system to provide satisfactory effluent quality.

PROCEDURES

Ecology sampling included both grab and composite samples. Prior to the inspection, Ecology Isco priority pollutant composite samplers were cleaned for priority pollutant sampling (Table 1). On site, a field transfer blank sample was collected (Table 1). A sampler was set up to collect a composite at the effluent monitoring site, the open concrete box just upstream of the discharge point. Approximately 350 mLs of sample were collected every 30 minutes for 24 hours. The sample collection jug was iced to cool samples as they were collected. Accompanying hand composites, comprised of three grab samples, were collected for bioassay tests. Sampling times and parameters analyzed are included in Table 2.

Composite and grab samples were also collected by U.S. Oil. The U.S. Oil composite sample was collected in a conventional sampler with equal volumes of sample collected approximately every four minutes for 24 hours. The sample was cooled during collection. A sample for a trout bioassay was also collected by U.S. Oil. Approximately three gallons of sample were collected along with the Ecology bioassay samples. The remainder of the water necessary for the test was collected at approximately 1200 hours on June 14. Sampling times and parameters analyzed are included in Table 2.

Three solids samples were collected for analysis; return activated sludge (RAS), dewatered digested sludge (DDS), and cake from centrifuged effluent (CCE). The RAS sample was a grab collected from the return line between the secondary clarifier and Orbal oxidation ditch. The DDS was a grab sample collected after dewatering.

The CCE sample was collected using an Alfa Laval model MAB102 centrifuge. A small centrifugal pump was used to pump effluent from the outlet box to the centrifuge. All contact parts of the setup were teflon or stainless-steel with the exception of some tin-bronze in the centrifuge bowl. Thus, some caution must be used in interpreting the copper data collected using the centrifuge. All contact parts were priority pollutant cleaned prior to the inspection using the procedure noted on Table 1.

Onsite, effluent was pumped through the centrifuge at a rate such that turbidity was not observed in the centrifuge effluent. The centrifuge was stopped at intervals to collect solids from the bowl. The VOA sample was collected from solids accumulated during the first interval. The remainder of the first interval solids and all additional solids were composited, homogenized, and put in the proper containers for analysis (Table 2). Times of unit operation and centrifuge flow rates are noted in Table 3. Centrifuge influent and effluent samples were collected for TSS analysis as a measure of centrifuge efficiency.

Table 2 summarizes sample splits for Ecology and U.S. Oil laboratory analysis. Ecology samples for *Ceriodaphnia dubia*, fathead minnow, and echinoderm bioassays were placed on ice and shipped overnight delivery to ERCE Bioassay Laboratory in San Diego, California. All other samples for Ecology analysis were placed on ice and shipped to the Ecology/EPA Laboratory in Manchester. Ecology analytical methods and laboratories doing the analysis are summarized in Table 4.

RESULTS AND DISCUSSION

The biological treatment system was not operating optimally during the inspection. The secondary clarifier skimmer arm was severely bent causing it to be non-functional (Heffner, 1989). The same problem was noted during the April 11, 1989, reconnaissance survey.

Scum build-up on the clarifier surface was estimated to be two to four inches. The clarifier surface had been manually cleaned the day before the inspection by a crew using a vacuum truck. The rapid accumulation of scum illustrates the need to fix the skimmer. Proper repair and maintenance of treatment system equipment is essential for reliable operation.

Conventional Parameters/NPDES Permit Limits Comparison

Conventional parameter results indicate good effluent quality during the inspection (Table 5). BOD₅, TSS, and NH₃-N concentrations were all relatively low. Inspection effluent loads, calculated using U.S. Oil flow data, were well within all NPDES permit limits (Table 6).

The accuracy of the U.S. Oil flow meter could not be independently confirmed by Ecology. U.S. Oil flow meter calibration records should be reviewed during the next inspection to help assure accuracy.

Priority Pollutants - Water

Few priority pollutants were detected in the effluent sample (Table 7). A summary of parameters analyzed and detection limits is included in Appendix A.

1,1,1-Trichloroethane, the only volatile organic detected, was found in all three grab samples collected (11-15 ug/L). Cyanide was also detected in the three grab samples at 15 ug/L. Two phthalate ester compounds were detected at concentrations below accurate quantification limits in the BNA scan of the composite sample. Several metals were detected, most at fairly low concentrations. Zinc, at approximately 100 ug/L, was found in the highest concentration.

Also included in Table 7 is a comparison of inspection results to toxicity criteria (EPA, 1986b). Freshwater acute criteria were not exceeded while cyanide and mercury freshwater chronic criteria were exceeded. Saltwater acute and chronic criteria were exceeded for cyanide, copper, and zinc; and chronic criteria only were exceeded for mercury and nickel. The mercury concentrations were the same (0.1 ug/L) in the transfer blank and two effluent samples analyzed, suggesting the low values may be the result of analytical imprecision at low concentrations. The phthalate esters chronic criteria (freshwater - 3.0 ug/L; saltwater - 3.4 ug/L) was approached by the sum of the estimated concentrations of the phthalate compounds. The usefulness of this observation is tempered by the uncertainty in the estimates, the finding of one of the phthalates in the transfer blank, and the fairly common occurrence of low concentrations of phthalates.

Quantities of two unknown compounds were estimated as part of the organic scans (Table 8). The estimated concentrations were low (< 12 ug/L), so positive identification and quantification were not attempted.

Bioassays - Water

Little acute toxicity was observed in the bioassays (rainbow trout, Microtox, *Daphnia magna*, *Hyallela azteca*, fathead minnow, and *Ceriodaphnia dubia* - Table 9). All LC₅₀s calculated were greater than 100 percent effluent.

Chronic effects varied from test to test (Table 9). *Daphnia magna* reproduction was not inhibited by the effluent. The *Ceriodaphnia dubia* reproduction test and fathead minnow growth test NOECs were 25 percent effluent indicating inhibition at higher effluent concentrations. The echinoderm fertilization test was most sensitive with an NOEC of 4.8 percent effluent. Chemicals exceeding toxicity criteria are noted in the "Priority Pollutants - Water" portion of the discussion.

Priority Pollutants - Solids

Three solids samples were collected: return activated sludge (RAS), dewatered digested sludge (DDS), and cake from centrifuged effluent (CCE). The quality of the DDS sample was marginal due to U.S. Oil operational problems. The individual that typically runs the dewatering unit was absent from work when the sample was collected. The back-up person was not thoroughly familiar with the dewatering unit and was unable to operate the unit optimally for a time period long enough for a good sample to be collected. Proper training of back-up personnel should be encouraged.

Centrifuge performance, measured as TSS removal by the centrifuge, was good during the inspection (Table 3). All centrifuge effluent TSS measurements were <1 mg/L.

The percent volatile solids and TOC data suggest the DDS and CCE samples were quite similar (Table 5). The RAS sample had a lower percent volatile solids and TOC concentration than the other two samples. The RAS sample also had a low enough percent solids to allow chemical analysis as a liquid. Thus, RAS chemical data are reported on a weight/volume basis while DDS and CCE data are reported on a weight/weight basis.

The VOA fraction detection limits were slightly high, but acceptable. Methylene chloride was detected in the CCE sample at 20,000 ug/L; likely due to residual left in the centrifuge during cleaning (Table 7). Absence of methylene chloride in the RAS and DDS samples supports sample contamination by the centrifuge. 1,1,1-Trichloroethane was identified in all three samples and 1,1-Dichloroethane was found in the CCE sample. 1,1,1-Trichloroethane was also found in the effluent samples. Concentrations were estimated for five other compounds detected at low concentrations in the DDS sample.

BNA detection limits were quite high (example, 4-Methylphenol detection limits were 29 times higher for the RAS sample than for the effluent sample - Table 7). A summary of parameters analyzed and detection limits is included in Appendix A. High detection limits were attributed to an elevated baseline during analysis, presumptive evidence of high boiling point hydrocarbons (Magoon, 1989). The individual compounds elevating the baseline were not chromatographically separable resulting in few peaks of sufficient magnitude to identify (Figure 1).

4,4'-DDT was detected in all three solids samples. There is a strong possibility the 4,4'-DDT detection was a false positive due to an interfering peak (Magoon, 1989).

Tentatively identified compounds (TICs) were noted in the VOA and BNA scans of both the DDS and CCE samples (Table 8). TICs were noted in the RAS sample BNA scan, but none were identified from the VOA scan. Positive identification of the TICs was not attempted.

Total petroleum hydrocarbon analysis of future sludge and centrifuge cake samples is recommended. This analysis may provide useful information, particularly when interferences hamper attempts to identify individual compounds with BNA and Pesticide/PCB scans.

DDS and CCE metals results look quite similar (Table 10). RAS metals concentrations were somewhat lower.

Laboratory Review and Results Comparison

Laboratory procedures were reviewed with Shauna-May Tecle-Mariam, Laboratory Manager. Procedures were generally acceptable with only minor suggestions made. Recommendations are circled on the attached Laboratory Procedure Review Sheet (Appendix B). One item that was the subject of some discussion was BOD₅ seeding (Heffner, 1989). The effluent BOD test is currently seeded with supernatant from settled activated sludge. Thus, the seed is virtually identical to the effluent. Seeding the effluent test is considered unnecessary and could be discontinued if desired. Influent tests should be seeded when run and the seed correction calculated using seed control data rather than seeded blank data.

Comparison of Ecology and U.S. Oil laboratory permit parameter results is good for most parameters (Table 6). The exceptions included TSS; Ecology results were 22 and 23 mg/L and U.S. Oil results were 9.2 and 10.4 mg/L. The Ecology laboratory also detected phenols at 34 ug/L in the grab split while the U.S. Oil laboratory reported <10 ug/L. The last two annual DMR-QA studies found the U.S. Oil laboratory TSS analysis result not acceptable in 1988 and the phenols analysis result not acceptable in 1989. A recheck of both parameters with a known sample and a U.S. Oil effluent sample split is recommended.

Better working knowledge by the U.S. Oil staff of the on-site effluent sampling equipment should be encouraged. The U.S. Oil effluent sample was collected on a time basis, but some equipment was also on hand to collect flow-paced composites. No one individual was knowledgeable about the entire sample collection system. There was some confusion as to which components were part of which composite collection system (Heffner, 1989). U.S. Oil and Ecology composite sample composition was very similar, suggesting U.S. Oil effluent sampler location and sample collection was acceptable.

RECOMMENDATIONS AND CONCLUSIONS

Repair of the clarifier skimmer arm was the most apparent need at the plant. The problem was noted during both the pre-inspection tour and the inspection. Good maintenance and prompt repair of the wastewater pollution control system should be encouraged.

Conventional Parameters/Comparison to NPDES Permit Limits

Treatment was good during the inspection; all permit parameters were within limits. Flow meter calibration should be reviewed during the next inspection to help assure accuracy.

Priority Pollutants - Water

1,1,1-Trichloroethane and several metals were detected in the effluent. Cyanide, copper, zinc, mercury, and nickel concentrations exceeded one or more of the saltwater acute, saltwater chronic, or freshwater chronic toxicity criteria.

Bioassays - Water

Little acute toxicity was found; all LC_{50} s calculated were greater than 100 percent effluent. Some chronic toxicity was noted in the fathead minnow (NOEC 25 percent effluent), *Ceriodaphnia dubia* (NOEC 25 percent effluent), and echinoderm fertilization (NOEC 4.8 percent effluent) bioassays.

Priority Pollutants - Solids

1,1,1-Trichloroethane was found in the three samples collected. Presumptive evidence of high boiling point hydrocarbons was found in the DDS and CCE samples. Total petroleum hydrocarbon analysis should be included when analyzing sludge samples in the future.

Collection of the DDS sample was difficult due to U.S. Oil problems operating the dewatering equipment. Back-up personnel should be adequately trained in wastewater treatment plant operation.

Laboratory Review and Results Comparison

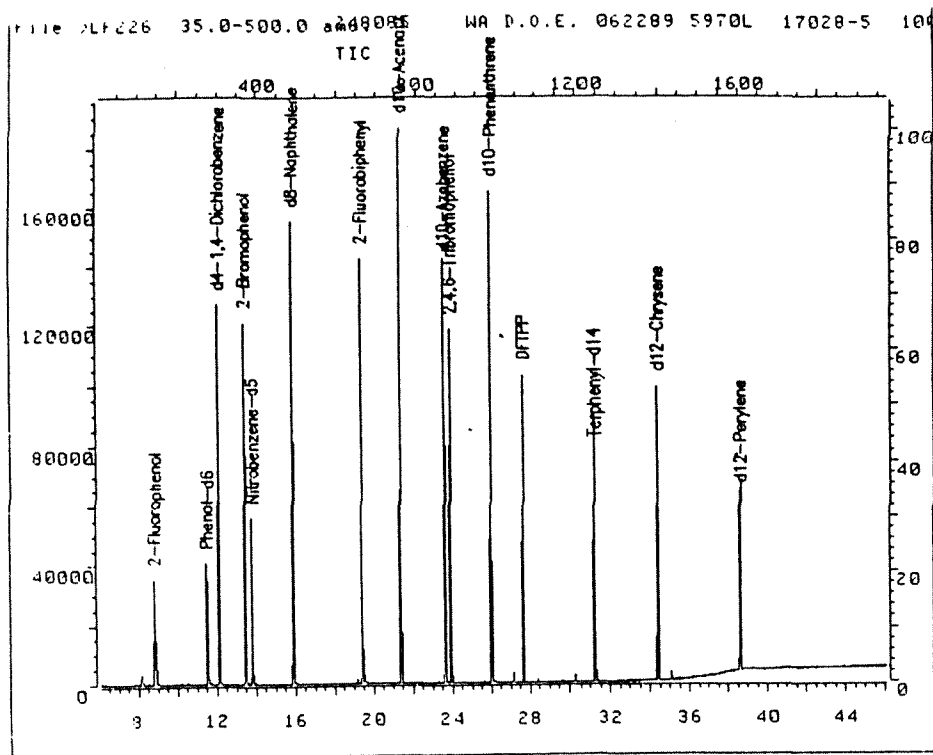
Laboratory and sampling procedures were generally good. Recommendations include:

1. Minor items noted on the "Laboratory Procedure Review Sheet" should be corrected.
2. Better working knowledge by the U.S. Oil staff of the on-site effluent sampling equipment should be encouraged.
3. Sample splits of a known and of the U.S. Oil effluent for TSS and phenols analysis are recommended during a future field visit.
4. Seeding the effluent BOD₅ test could be discontinued if desired.

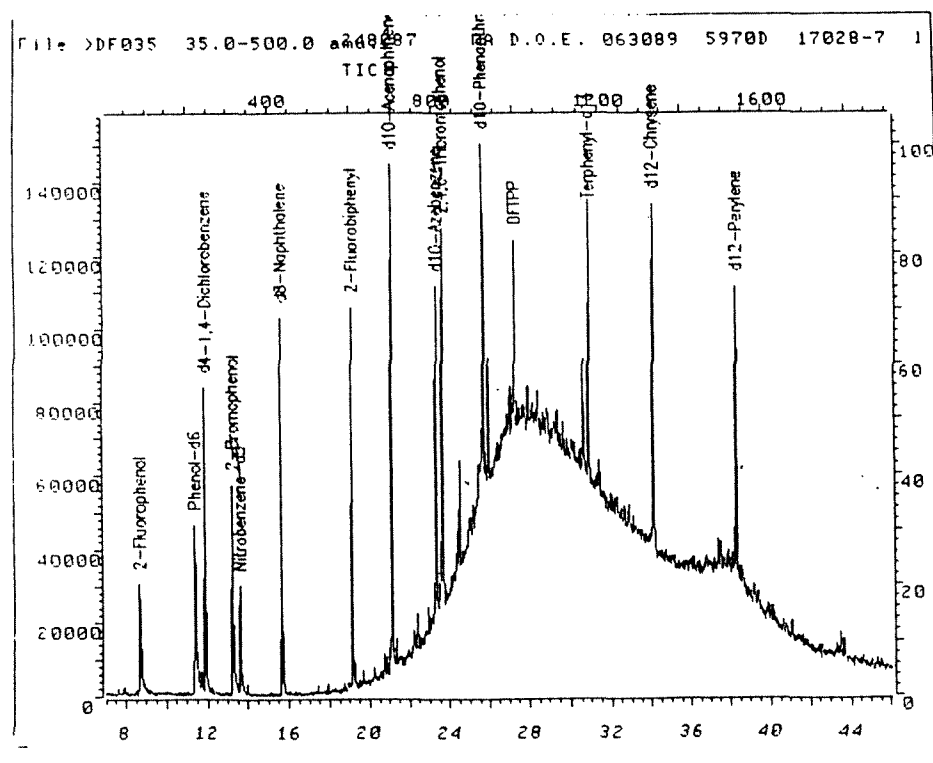
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Transfer Blank - Lab Log #248085



Dewatered Digested Sludge - Lab Log #248087

Figure 1 - BNA Chromatograms - US Oil, June 1989.

Table 1 - Priority Pollutant Cleaning and Field Transfer Blank Procedures -
U.S. Oil, June 1989

PRIORITY POLLUTANT SAMPLING EQUIPMENT CLEANING PROCEDURES

1. Wash with laboratory detergent
2. Rinse several times with tap water
3. Rinse with 10% HNO₃ solution
4. Rinse three (3) times with distilled/deionized water
5. Rinse with high purity methylene chloride
6. Rinse with high purity acetone*
7. Allow to dry and seal with aluminum foil

* when cleaning the centrifuge, a final rinse with organic free water followed the acetone rinse

FIELD TRANSFER BLANK PROCEDURE

1. Pour organic free water directly into appropriate bottles for parameters to be analyzed from grab samples (VOA).
2. Run approximately 1L of organic free water through a compositor and discard.
3. Run approximately 6L of organic free water through the same compositor and put the water into appropriate bottles for parameters to be analyzed from composite samples (BNA, Pesticide/PCB, and metals).

Table 2 - Sampling Schedule and Parameters Analyzed - US Oil, June 1989.

| | Sample: | Effluent | Effluent | Effluent | Effluent | Effluent | Transfer Blank | RAS | Digested Sludge | Centrifuge Solids | Centrifuge Efficiency |
|----------------------------|-------------------------------------|----------|----------|----------|-----------|-----------|-------------------|---------|--------------------|----------------------|--------------------------|
| | Date: | 6/13 | 6/13 | 6/14 | 6/13-14 | 6/13-14 | 6/13 | 6/14 | 6/14 | 6/13-14 | 6/13-14 |
| | Time: | 1040 | 1455 | 0905 | 1030-1030 | 1030-1030 | 1000 | 1355 | 1345 | * | * |
| | Sampler: | Ecology | Ecology | *** | Ecology | US Oil | Ecology | Ecology | Ecology | Ecology | Ecology |
| | Type: | Grab | Grab | Grab | Composite | Composite | | Grab | Grab | * | Grabs |
| | Lab Log #: | 248080 | 248081 | 248082 | 248083 | 248084 | 248085 | 248086 | 248087 | 248088 | |
| <u>Field Analysis</u> | | | | | | | | | | | |
| | Sulfide | E | E | E US | | | | | | | |
| | pH | E | E | E | | | | | | | |
| | Temperature | E | E | E | | | | | | | |
| | Conductivity | E | E | E | | | | | | | |
| <u>Laboratory Analysis</u> | | | | | | | | | | | |
| | Conductivity | E | E | E | E | E | | | | | |
| | Alkalinity | | | | E | E | | | | | |
| | Hardness | | | | E | E | | | | | |
| | NH ₃ -N | E | E | E | E US | E US | | | | | |
| | NO ₃ +NO ₂ -N | | | | E | E | | | | | |
| | Total-P | | | | E | E | | | | | |
| | TS | | | | E | E | | | | | |
| | TNVS | | | | E | E | | | | | |
| | TSS | E | E | E | E US | E US | | | | | E |
| | TNVSS | | | | E | E | | | | | |
| | COD | E | E | E | E US | E US | | | | | |
| | BOD ₅ | | | | E | E US | | | | | |
| | Oil and Grease | E | E | E US | | | | | | | |
| | TOC | | | | | | | E | E | E | |
| | % Solids | | | | | | | E | E | E | |
| | % Volatile Solids | | | | | | | E | E | E | |
| | Cyanide | E | E | E | | | | | | | |
| | Phenols | E | E | E US | | | | | | | |
| | VOA | E | E | E | | | E | E | E | E | |
| | BNA | | | | E | | E | E | E | E | |
| | Pest/PCB | | | | E | | E | E | E | E | |
| | pp metals | | | | E | E | E | E | E | E | |

Table 2 - Continued - US Oil, June 1989.

| Sample: | Effluent | Effluent | Effluent | Effluent | Effluent | Transfer Blank | RAS | Digested Sludge | Centrifuge Solids | Centrifuge Efficiency |
|---------------------|----------|----------|----------|-----------|-----------|-------------------|---------|--------------------|----------------------|--------------------------|
| Date: | 6/13 | 6/13 | 6/14 | 6/13-14 | 6/13-14 | 6/13 | 6/14 | 6/14 | 6/13-14 | 6/13-14 |
| Time: | 1040 | 1455 | 0905 | 1030-1030 | 1030-1030 | 1000 | 1355 | 1345 | * | * |
| Sampler: | Ecology | Ecology | *** | Ecology | US Oil | Ecology | Ecology | Ecology | Ecology | Ecology |
| Type: | Grab | Grab | Grab | Composite | Composite | | Grab | Grab | * | Grabs |
| Lab Log #: | 248080 | 248081 | 248082 | 248083 | 248084 | 248085 | 248086 | 248087 | 248088 | |
| Hexavalent Chromium | E | E | E | E | US | | | | | |
| Total Chromium | | | | E | E US | | | | | |
| Trout | | | | E ** | | | | | | |
| Daphnia Magna | | | | E ** | | | | | | |
| Microtox | | | | E ** | | | | | | |
| Fathead Minnow | | | | E ** | | | | | | |
| Ceriodaphnia | | | | E ** | | | | | | |
| Echinoderm | | | | E ** | | | | | | |
| Hyallolella | | | | E ** | | | | | | |

E - Ecology Laboratory analysis

US - US Oil Laboratory analysis

* - see Table 3 for centrifuge sampling times

** - Samples for bioassay analysis were grab composites. Equal volumes were collected on 6/13 at 1050, on 6/13 at 1510, and on 6/14 at 0940.

*** - Samples for Ecology analysis were collected by Ecology. Samples for US Oil analysis were collected by US Oil.

Table 3 - Centrifuge Records - US Oil, June 1989.

| Date | Action * | Time | <u>Centrifuge Efficiency</u> | | TSS (mg/L) | Comments |
|------|----------|------|------------------------------|---------------------------|---------------|--|
| | | | Intake Flow Rate (gpm) | Effluent TSS (mg/L) | | |
| 6/13 | Start | 1105 | | | | |
| | | 1120 | 1.9 | 9 | 1U | |
| | Stop | 1330 | | 11 | 1U | VOA sample collected Bowl O-ring replaced |
| | Start | 1440 | | | | |
| | | 1600 | 1.9 | | | Flow Rate Adjusted |
| | | 1605 | 1.5 | | | |
| 6/14 | Stop | 2130 | 1.5 | 1 | 1U | Composite sample spilled |
| | Start | 2200 | 1.5 | | | |
| | Stop | 0840 | 1.4 | 2 | 1U | First grab of new composite |
| | Start | 0900 | | | | |
| | | 1025 | 1.5 | | | |
| | Stop | 1400 | 1.4 | 19 | 1U | |
| | Start | 1425 | | | | |
| | | 1445 | 1.4 | | | |
| | Stop | 2025 | 1.5 | | | |

* solids removed when centrifuge stopped

Table 4 - Ecology Analytical Methods - US Oil, June 1989.

| | Method Used for Ecology Analysis (Ecology, 1988&89) | Laboratory Performing Analysis |
|--------------------------------------|---|--------------------------------------|
| -----Laboratory Analyses----- | | |
| Conductivity | EPA #120.1 | Ecology |
| Alkalinity | EPA #310.1 | Ecology |
| Hardness | EPA #130.2 | Ecology |
| NH ₃ -N | EPA #350.1 | Ecology |
| NO ₃ + NO ₂ -N | EPA #353.1 | Ecology |
| Total-P | EPA #365.1 | Ecology |
| TS | EPA #160.3 | Ecology |
| TNVS | EPA #160 | Ecology |
| TSS | EPA #160.2 | Ecology |
| TNVSS | EPA #160 | Ecology |
| COD | EPA #410.1 | Ecology |
| BOD ₅ | EPA #405.1 | Ecology |
| Oil and Grease | EPA #413.1 | Ecology |
| TOC | Tetra Tech, 1986 | ARI |
| % Solids | EPA #160.3 | ARI |
| % Volatile Solids | EPA #160.4 | ARI |
| Cyanide | EPA #335.3 | Ecology |
| Phenols | EPA #420.1 | Ecology |
| VOA (water) | EPA #624 | Laucks |
| VOA (solids) | EPA #8240 | Laucks |
| BNA (water) | EPA #625 | Laucks |
| BNA (solids) | EPA #8270 | Laucks |
| Pest/PCB (water) | EPA #608 | Laucks |
| Pest/PCB (solids) | EPA #8080 | Laucks |
| pp metals | EPA #200 | ARI |
| Hexavalent Chromium | EPA #200 | Ecology |
| Trout | Ecology, 1981 | Ecology |
| Daphnia Magna | EPA, 1987 | Ecology |
| Microtox | Beckman, 1982 | ECOVA |
| Fathead Minnow | EPA, 1985a | ERCE |
| Ceriodaphnia | EPA, 1985b | ERCE |
| Echinoderm | Dinnel, 1987 | ERCE |
| Hyallolela | Nebeker, 1984* | Ecology |
| -----Field Analyses----- | | |
| Sulfide | EPA #376.2** | |
| pH | APHA, 1985: #423 | |
| Conductivity | APHA, 1985: #205 | |
| Temperature | APHA, 1985: #212 | |

ARI - Analytical Resources, Inc.
ERCE - ERCE Bioassay Laboratory
* - modified for water samples

ECOVA - ECOVA
Laucks - Laucks Testing Laboratories, Inc.
** - Chemetrics test kit

Table 5 - Ecology Conventional Parameter Results - US Oil, June 1989.

| Sample: | Effluent | Effluent | Effluent | Effluent | Effluent | RAS | Digested Sludge | Centrifuge Solids |
|----------------------------|----------|----------|----------|-----------|-----------|---------|--------------------|----------------------|
| Date: | 6/13 | 6/13 | 6/14 | 6/13-14 | 6/13-14 | 6/14 | 6/14 | 6/13-14 |
| Time: | 1040 | 1455 | 0905 | 1030-1030 | 1030-1030 | 1355 | 1345 | * |
| Sampler: | Ecology | Ecology | Ecology | Ecology | US Oil | Ecology | Ecology | Ecology |
| Type: | Grab | Grab | Grab | Composite | Composite | Grab | Grab | * |
| Lab Log #: | 248080 | 248081 | 248082 | 248083 | 248084 | 248086 | 248087 | 248088 |
| <u>Field Analysis</u> | | | | | | | | |
| Sulfide (mg/L) | <0.1 | <0.1 | 0.1 | | | | | |
| pH (S.U.) | 8.3 | 7.7 | 8.0 | | | | | |
| Temperature (C) | 24.6 | 25.1 | 24.8 | | | | | |
| Conductivity (umhos/cm) | 2550 | 2670 | 2690 | | | | | |
| <u>Laboratory Analysis</u> | | | | | | | | |
| Conductivity (umhos/cm) | 2660 | 2620 | 2660 | 2530 | 2600 | | | |
| Alkalinity (mg/L as CaCO3) | | | | 242 | 239 | | | |
| Hardness (mg/L as CaCO3) | | | | 127 | 130 | | | |
| NH3-N (mg/L) | 0.05 | 0.03 | 0.09 | 0.07 | 0.09 | | | |
| NO3+NO2-N (mg/L) | | | | 5.5 | 5.6 | | | |
| Total-P (mg/L) | | | | 2.5 | 2.5 | | | |
| TS (mg/L) | | | | 1750 | 1750 | | | |
| TNVS (mg/L) | | | | 1550 | 1560 | | | |
| TSS (mg/L) | 11 | 20 | 3 | 22 | 23 | | | |
| TNVSS (mg/L) | | | | 8 | 6 | | | |
| COD (mg/L) | 80 | 94 | 69 | 82 | 81 | | | |
| BOD5 (mg/L) | | | | 7 | 7 | | | |
| Oil & Grease (mg/L) | 1 | 6 | 2 | | | | | |
| TOC (g/Kg) | | | | | | 210 | 390 | 330 |
| % Solids | | | | | | 0.63 | 9.7 | 16.8 |
| % Volatile Solids | | | | | | 55.6 | 72.4 | 77.3 |
| Cyanide (ug/L) | 15 | 15 | 15 | | | | | |
| Phenols (ug/L) | 10 | 10 | 34 | | | | | |
| Hexavalent Chromium (ug/L) | 3.5 | 3.1 | 3.3 | 3.8 | | | | |

* see Table 3 for sample times and centrifuge efficiency data

Table 6 - NPDES Permit Limits and Laboratory Results Comparison - US Oil, June 1989.

| | | NPDES Permit Limits | | | | | | Ecology Laboratory Results | | | | | US Oil Laboratory Results | | |
|---------------------|----------------|---------------------------|---------|-----------|-----------|-----------|-----------|----------------------------|-----------|-----------|----------|----------|---------------------------|----------|----------|
| | | | | | | | | Sample: | Effluent | Effluent | Effluent | Effluent | Effluent | Effluent | Effluent |
| | | Date: | 6/13 | 6/13 | 6/14 | 6/13-14 | 6/13-14 | 6/14 | 6/13-14 | 6/13-14 | | | | | |
| | | Time: | 1040 | 1455 | 0905 | 1030-1030 | 1030-1030 | 0905 | 1030-1030 | 1030-1030 | | | | | |
| | | Sampler: | Ecology | Ecology | Ecology | Ecology | US Oil | US Oil | Ecology | US Oil | | | | | |
| Type: | Grab | Grab | Grab | Composite | Composite | Grab | Composite | Composite | | | | | | | |
| Lab Log #: | 248080 | 248081 | 248082 | 248083 | 248084 | | | | | | | | | | |
| BOD ₅ | (mg/L) | | | | | | | 7 | 7 | | | | 4.9 | | |
| | (lbs/D) | 120 | 220 | 24 | 44 | 144 | 264 | 21 | 21 | | | | 15 | | |
| | (lbs/1000 gal) | | | 0.22 | 0.40 | | | | | | | | | | |
| COD | (mg/L) | | | | | | | 82 | 81 | | | 77.9 | 72.7 | | |
| | (lbs/D) | 580 | 1120 | 165 | 330 | 745 | 1450 | 251 | 246 | | | 237 | 221 | | |
| | (lbs/1000 gal) | | | 1.5 | 3.0 | | | | | | | | | | |
| TSS | (mg/L) | | | | | | | 22 | 23 | | | 9.2 | 10.4 | | |
| | (lbs/D) | 100 | 150 | 20 | 31 | 120 | 181 | 67 | 70 | | | 28 | 32 | | |
| | (lbs/1000 gal) | | | 0.18 | 0.28 | | | | | | | | | | |
| Oil & Grease | (mg/L) | | 15 | | | | | 1 | 6 | 2 | | 2.0 | | | |
| | (lbs/D) | 35 | 68 | 7 | 14 | 42 | 82 | 3 | 18 | 6 | | 6 | | | |
| | (lbs/1000 gal) | | | 0.067 | 0.13 | | | | | | | | | | |
| Phenols | (ug/L) | | | | | | | 10 | 10 | 34 | | <10 | | | |
| | (lbs/D) | 0.31 | 1.31 | | | 0.31 | 1.31 | 0.03 | 0.03 | 0.10 | | <0.03 | | | |
| NH ₃ -N | (mg/L) | | | | | | | | | 0.07 | 0.09 | | <1 | | |
| | (lbs/D) | 12 | 27 | | | 12 | 27 | | | 0.2 | 0.3 | | <3 | | |
| Sulfide | (mg/L) | | | | | | | <0.1 | <0.1 | 0.1 | | <0.1 | | | |
| | (lbs/D) | 0.6 | 1.4 | | | 0.6 | 1.4 | <0.3 | <0.3 | 0.3 | | <0.3 | | | |
| Total Chromium | (ug/L) | | | | | | | | | 8 | 7 | | 7 | | |
| | (lbs/D) | 0.39 | 1.09 | | | 0.39 | 1.09 | | | 0.02 | 0.02 | | 0.02 | | |
| Hexavalent Chromium | (ug/L) | | | | | | | | | 3.8 | | | ND | | |
| | (lbs/D) | 0.03 | 0.07 | | | 0.03 | 0.07 | | | 0.01 | | | 0.00 | | |
| pH | (S.U.) | within range of 6.0 - 9.0 | | | | | | 8.3 | 7.7 | 8.0 | | | | | |
| Flow | (gpm) (MGD) | | | | | | | | | | | | 253.2 0.36 | | |

* Based on a process flow of 175 gpm: runoff flow is; 253 - 175 = 78 gpm = 0.11 MGD

Table 7 - Priority Pollutants Detected - US Oil, June 1989.

| | Sample: | Effluent | Effluent | Effluent | Transfer | | Digested | Centrifuge | <u>Toxicity Criteria (ug/L - EPA, 1986b)</u> | | | |
|----------------------------------|------------|-------------|----------|----------|----------|------------------|----------------------|------------|--|---------|------------------|---------|
| | Lab Log #: | 248080 | 248081 | 248082 | Blank | RAS | Sludge | Solids | | | | |
| | Type: | Grab | Grab | Grab | Grab | Grab | Grab | ECO-Comp | | | | |
| | Date: | 6/13 | 6/13 | 6/14 | 6/13 | 6/14 | 6/14 | 6/13-14 | | | | |
| | Time: | 1040 | 1455 | 0905 | 1000 | 1355 | 1345 | | <u>Freshwater</u> | | <u>Saltwater</u> | |
| | | | | | | | | | Acute | Chronic | Acute | Chronic |
| --- VOA Compounds --- | | ug/L | ug/L | ug/L | ug/L | ug/L | ----ug/Kg dry wt---- | | | | | |
| Methylene Chloride | | 5 U | 5 U | 5 U | 5 U | 5 U | 53 J | 20000 D | | | | |
| 1,1-Dichloroethane | | 5 U | 5 U | 5 U | 5 U | 5 U | 50 J | 100 | | | | |
| 1,1,1-Trichloroethane | | 11 | 12 | 15 | 5 U | 13 | 170 | 110 | | | 31200 * | |
| Tetrachloroethene | | 5 U | 5 U | 5 U | 5 U | 5 U | 87 J | 78 U | | | | |
| Toluene | | 5 U | 5 U | 5 U | 5 U | 5 U | 88 J | 78 U | | | | |
| Total Xylenes | | 5 U | 5 U | 5 U | 5 U | 5 U | 79 J | 78 U | | | | |
| ----- | | | | | | | | | | | | |
| Cyanide (ug/L) | | 15 | 15 | 15 | | | | | 22 | 5.2 | 1 | 1 |
| | | | | | | | | | | | | |
| | Sample: | Effluent | Effluent | Transfer | | Digested | Centrifuge | | | | | |
| | Lab Log #: | 248084 | 248083 | Blank | RAS | Sludge | Solids | | | | | |
| | Type: | US Oil-Comp | ECO-Comp | Grab | Grab | Grab | ECO-Comp | | | | | |
| | Date: | 6/13-14 | 6/13-14 | 6/13 | 6/14 | 6/14 | 6/13-14 | | | | | |
| --BNA Compounds-- | | | ug/L | ug/L | ug/L | --ug/Kg dry wt-- | | | | | | |
| 4-Methylphenol | | | 10 U | 10 U | 290 U | 210000 U | 15000 J | | | | | |
| Di-n-Butyl Phthalate | | | 1 J | 10 U | 290 U | 210000 U | 120000 U | 940 *+ | 3 *+ | 2944 *+ | 3.4 *+ | |
| Bis(2-Ethylhexyl)phthalate | | | 2 J | 1 J | 39 JB | 11000 JB | 10000 JB | 940 *+ | 3 *+ | 2944 *+ | 3.4 *+ | |
| --Pest/PCB Compounds-- | | | | | | | | | | | | |
| 4,4'-DDT | | | 0.10 U | 0.10 U | 0.78 | 140 J | 130 | | | | | |
| --Priority pollutant metals **-- | | ug/L | ug/L | ug/L | ug/L | --mg/Kg dry wt-- | | | | | | |
| Antimony | | 2 | 2 U | 1 U | 18 | 1.00 U | 1.80 | 9000 * | 1600 * | | | |
| Arsenic | | 5 U | 5 U | 1 U | 70 | 11.1 | 15.8 | | | | | |
| Beryllium | | 1 U | 1 U | 1 U | 2 | 1.03 U | 0.56 U | | | | | |

Table 7 - Continued - US Oil, June 1989.

| | Sample: | Effluent | Effluent | Transfer | RAS | Digested | Centrifuge | <u>Toxicity Criteria (ug/L - EPA, 1986b)</u> | | | | | | | | |
|---------------|------------|-------------|----------|----------|--------|----------|------------|--|-----------|-------|---------|---|-------|---|-------|-------|
| | Lab Log #: | 248084 | 248083 | Blank | 248086 | Sludge | Solids | | | | | | | | | |
| | Type: | US Oil-Comp | ECO-Comp | Grab | Grab | Grab | ECO-Comp | Freshwater | Saltwater | | | | | | | |
| | Date: | 6/13-14 | 6/13-14 | 6/13 | 6/14 | 6/14 | 6/13-14 | Acute | Chronic | Acute | Chronic | | | | | |
| Cadmium | 2 | U | 2 | U | 2 | U | 12 | 2.05 | U | 2.63 | | | | | | |
| Chromium | 7 | | 8 | | 5 | U | 584 | 125 | | 119 | 2153 | + | 257 | + | 10300 | * |
| Chromium (VI) | | | 3.8 | | | | | | | | 16 | | 11 | | 1100 | 50 |
| Copper | 7 | | 4 | | 2 | U | 1230 | 256 | | 275 | 23 | + | 15 | + | 2.9 | 2.9 |
| Lead | 2 | U | 2 | U | 1 | U | 320 | 68.3 | | 67.8 | | | | | | |
| Mercury | 0.1 | | 0.1 | | 0.1 | | 37 | 10.0 | | 9.38 | 2.4 | | 0.012 | | 2.1 | 0.025 |
| Nickel | 20 | | 20 | | 10 | U | 470 | 106 | | 95.1 | 1771 | + | 197 | + | 75 | 8.3 |
| Selenium | 5 | U | 5 | U | 1 | U | 30 | 11.8 | | 13.2 | | | | | | |
| Zinc | 123 | | 91 | | 4 | U | 2260 | 502 | | 480 | 146 | + | 132 | + | 95 | 86 |

U - indicates compound was analyzed for but not detected at the given detection limit

J - indicates an estimated value when result is less than specified detection limit

B - This flag is used when the analyte is found in the blank as well as the sample. Indicates possible/probable blank contamination

D - value from analysis of a diluted sample

UJ - indicates compound was analyzed for but not detected at the given detection limit, and the internal standard on which detection limit quantification was based was outside acceptance limits

JB - comment J plus comment B

M - indicates an estimated value of analyte found and confirmed by analyst but with low spectral match parameters

** - metals digestion for the different samples was:

Lab Log #'s 248083, 248084, and 248085; total recoverable for all metals except Hg, which was total. Lab Log # 248086; As, Sb, and Se are total recoverable, all others are total.

Lab Log #'s 248087 and 248088; total with sediment/soil digestion.

* - insufficient data to develop criteria - Lowest Observed Effect Level (LOEL) presented

+ - criteria based hardness (130 mg/L for US Oil)

+* - criteria for Chromium (III) - based on hardness

*+ - LOEL for phthalate esters

Table 8 - Tentatively Identified Organics - US Oil, June 1989.

| | | Sample: Lab Log #: | Effluent 248080 | Effluent 248081 | Effluent 248082 | Transfer Blank 248085 | RAS 248086 | Digested Sludge 248087 | Centrifuge Solids 248088 |
|------------------------------|--|-----------------------|--------------------|--------------------|--------------------|--------------------------|---------------|---------------------------|-----------------------------|
| | | Type: | Grab | Grab | Grab | Grab | Grab | Grab | ECO-Comp |
| | | Date: | 6/13 | 6/13 | 6/14 | 6/13 | 6/14 | 6/14 | 6/13-14 |
| | | Time: | 1040 | 1455 | 0905 | 1000 | 1355 | 1345 | |
| | | Retention Time | ug/L | ug/L | ug/L | ug/L | ug/L | ug/Kg | ug/Kg |
| --- VOA Compounds --- | | | | | | | | | |
| Methanethiol | | 2.75 | | | | | | | 7000 J |
| Methanamine, N,N-dimethyl- | | 2.90 | | | | | | 660 J | |
| Methane, thiobis- | | 5.30-5.32 | | | | | | 5600 J | 1400 J |
| 1-Propanethiol | | 8.35 | | | | | | | 110 J |
| Ethane, (methylthio)- | | 8.52 | | | | | | | 140 J |
| 1-Propanethiol, 2-methyl- | | 11.97 | | | | | | | 170 J |
| Disulfide, dimethyl | | 13.17-13.27 | | | | | | 190 J | 710 J |
| Butane, 2-(methylthio)- | | 13.97 | | | | | | 150 J | |
| Thiophene, 2-methyl- | | 14.05 | | | | | | | 91 J |
| Cyclohexane, (1-methylethyl) | | 18.27 | | | | | | 190 J | |
| 1-Hexane, 3,5,5-trimethyl- | | 19.10 | | | | | | 240 J | |
| Benzene, 1,3,5-trimethyl- | | 19.85 | | | | | | 240 J | |
| Decane | | 20.10 | | | | | | 530 J | |
| Methane, sulfonylbis- | | 20.35 | | | | | | | 160 J |
| Benzene, 1,2,3-trimethyl- | | 20.62 | | | | | | 440 J | |

Table 8 - Continued - US Oil, June 1989.

| | | Sample: Lab Log #: Type: Date: | Effluent 248083 ECO-Comp 6/13-14 | Transfer Blank 248085 Grab 6/13 | RAS 248086 Grab 6/14 | Digested Sludge 248087 Grab 6/14 | Centrifuge Solids 248088 ECO-Comp 6/13-14 |
|-----------------------|------------------------------|---|---|--|-------------------------------|---|--|
| | | Retention Time | | | | | |
| --- BNA Compounds --- | | | ug/L | ug/L | ug/L | ug/Kg dry wt | ug/Kg dry wt |
| 2 | Unknown | 22.42 | | | | | 48000 J |
| | Unknown | 24.45 | | | | | 39000 J |
| | Dedecane, 2,7,10-trimethyl- | 24.55 | | | | 260000 J | |
| | Unknown | 24.55 | | | | | 64000 J |
| | Unknown | 24.84 | | | 350 J | | |
| | Heptadecane, 2,6,10,15-tetra | 26.00 | | | | 300000 J | |
| | Unknown | 26.01 | | | | | 120000 J |
| | Unknown | 26.32 | | | 280 J | | |
| | Unknown | 27.84 | | | | | 190000 J |
| | Unknown | 28.09 | | | | | 270000 J |
| | Unknown | 30.32 | 11 J | | | | |
| | Unknown | 30.68 | | | | | 98000 J |
| | Unknown | 30.69 | | | | 240000 J | |
| | Unknown | 31.00 | | | 390 J | | |
| | Unknown | 33.14 | 8 J | | | | |
| | Unknown | 37.41 | | | | 75000 J | |
| | Unknown | 37.42 | | | | | 100000 J |
| | Unknown | 37.94 | | | 230 J | | |

J - indicates an estimated value when result is less than specified detection limit

Table 9 - Ecology Bioassay Results - US Oil, June 1989.

| Rainbow Trout (<i>Oncorhynchus mykiss</i>) * - 96 hour survival test | | | | | | |
|--|----------|------------|----------|------------|-------------------|------------------|
| Lab Log # | Sample | % Effluent | # Tested | # Survived | Percent Mortality | Percent Survival |
| | Control | 0 | 30 | 30 | 0 | 100 |
| 248083 | Effluent | 65 | 30 | 30 | 0 | 100 |

* - US Oil reported 100% survival in the trout bioassay they conducted in 65% effluent

| Microtox | | | |
|-----------|----------|-----------------------------|----------|
| Lab Log # | Sample | 15 minute EC50 (% effluent) | Ranking* |
| 248083 | Effluent | >100% | low |

* - priority ranking for further toxicity evaluation based on the EC50 (EPA, 1980)

Daphnia magna - 7 day survival and reproduction test

| Lab Log # | Sample | # Tested | # Survived | Percent Mortality | Percent Survival | Mean # Young per Original Female |
|-----------|-----------------|----------|------------|-------------------|------------------|----------------------------------|
| | Control | 10 | 9 | 10 | 90 | 9.0 |
| 248083 | 6.25 % Effluent | 10 | 10 | 0 | 100 | 17.1 |
| 248083 | 12.5 % Effluent | 10 | 10 | 0 | 100 | 18.4 |
| 248083 | 25.0 % Effluent | 10 | 10 | 0 | 100 | 16.5 |
| 248083 | 50.0 % Effluent | 10 | 10 | 0 | 100 | 20.0 |
| 248083 | 100 % Effluent | 10 | 10 | 0 | 100 | 20.1 |

Acute
LC50 > 100% effluent
NOEC = 100% effluent

Chronic
NOEC = 100% effluent

Table 9 - Continued - US Oil, June 1989.

Hyaella azteca - 72 and 96 hour survival tests

| Lab Log # | Sample | Test Duration (hrs) | % Effluent | # Tested * | # Survived | Percent Mortality | Percent Survival |
|-----------|----------|---------------------|------------|------------|------------|-------------------|------------------|
| | Control | 72 | 100 | 50 | 49 | 2 | 98 |
| | | 96 | 100 | 50 | 45 | 10 | 90 |
| 248083 | Effluent | 72 | 100 | 60 | 58 | 3 | 97 |
| | | 96 | 100 | 50 | 50 | 0 | 100 |

* tests were conducted with 5 replicates of 10 organisms each, with the exception of one of the 72-hour effluent replicates in which 20 organisms were tested.

Fathead Minnow (Pimephales promelas) - 96 hour survival and 7 day growth test

| Lab Log # | Sample | # Tested | after 96 hours | | | after 7 days | | | |
|-----------|-----------------|----------|----------------------------|-------------------|------------------|---------------------|-------------------|------------------|---------------------------|
| | | | # Survived | Percent Mortality | Percent Survival | # Survived | Percent Mortality | Percent Survival | Mean Weight per Fish (mg) |
| | Control | 30 | 29 | 3 | 97 | 26 | 13 | 87 | 0.68 |
| 248083 | 6.25 % Effluent | 30 | 29 | 3 | 97 | 28 | 7 | 93 | 0.51 |
| 248083 | 12.5 % Effluent | 30 | 27 | 10 | 90 | 27 | 10 | 90 | 0.54 |
| 248083 | 25.0 % Effluent | 30 | 26 | 13 | 87 | 25 | 17 | 83 | 0.53 |
| 248083 | 50.0 % Effluent | 30 | 25 | 17 | 83 | 16 | 47 | 53 | N/A |
| 248083 | 100 % Effluent | 30 | 21 | 30 | 70 | 4 | 87 | 13 | N/A |
| | | | Acute | | | Chronic | | | |
| | | | 96 hr LC50 > 100% effluent | | | NOEC = 25% effluent | | | |
| | | | | | | LOEC = 50% effluent | | | |

Table 9 - Continued - US Oil, June 1989.

Ceriodaphnia dubia - 48 hour survival and 7 day reproduction test

| Lab Log # | Sample | # Tested | after 48 hours | | | after 7 days | | | |
|-----------|-----------------|-------------|----------------------------|----------------------|---------------------|---------------------|----------------------|---------------------|-------------------------------------|
| | | | # Survived | Percent Mortality | Percent Survival | # Survived | Percent Mortality | Percent Survival | Mean # Young per Original Female |
| | Control | 10 | 10 | 0 | 100 | 9 | 10 | 90 | 24.4 |
| 248083 | 6.25 % Effluent | 10 | 10 | 0 | 100 | 10 | 0 | 100 | 28.2 |
| 248083 | 12.5 % Effluent | 10 | 10 | 0 | 100 | 9 | 10 | 90 | 29.0 |
| 248083 | 25.0 % Effluent | 10 | 10 | 0 | 100 | 8 | 20 | 80 | 24.3 |
| 248083 | 50.0 % Effluent | 10 | 6 | 40 | 60 | 6 | 40 | 60 | 15.0 |
| 248083 | 100 % Effluent | 10 | 10 | 0 | 100 | 9 | 10 | 90 | 9.8 |
| | | | Acute | | | Chronic | | | |
| | | | 48 hr LC50 > 100% effluent | | | NOEC = 25% effluent | | | |
| | | | | | | LOEC = 50% effluent | | | |

Lytechinus pictus (sea urchin) - Echinoderm fertilization test *

| Lab Log # | Sample | Average % Unfertilized Eggs | II=====II | |
|-----------|-----------------|--------------------------------|-----------|---|
| | Control | 18.8 | II | II |
| 248083 | 4.8 % Effluent | 26.0 | II | LOEC - lowest observable effects concentration II |
| 248083 | 9.6 % Effluent | 32.8 | II | LC50 - lethal concentration for 50% of the organisms II |
| 248083 | 19.2 % Effluent | 44.0 | II | EC50 - effect concentration for 50% of the organisms II |
| 248083 | 38.5 % Effluent | 40.3 | II | II |
| 248083 | 76.9 % Effluent | 89.5 | II=====II | II |

NOEC = 4.8% effluent

* salinity adjusted using a brine solution concentrated
from seawater by the test laboratory

Table 10 - Solids Samples Metals Summary - US Oil, June 1989.

| | | | | |
|------------|--------|--------|-----------------|-------------------|
| Sample: | RAS | RAS* | Digested Sludge | Centrifuge Solids |
| Lab Log #: | 248086 | 248086 | 248087 | 248088 |
| Type: | Grab | Grab | Grab | ECO-Comp |
| Date: | 6/14 | 6/14 | 6/14 | 6/13-14 |

| | | | | |
|------------------------------------|------|------------------------|--------|--------|
| -- Priority pollutant metals ** -- | ug/L | -----mg/Kg dry wt----- | | |
| Antimony | 18 | 2.9 | 1.00 U | 1.80 |
| Arsenic | 70 | 11.1 | 11.1 | 15.8 |
| Beryllium | 2 | 0.3 | 1.03 U | 0.56 U |
| Cadmium | 12 | 1.9 | 2.05 U | 2.63 |
| Chromium | 584 | 92.7 | 125 | 119 |
| Copper | 1230 | 195.2 | 256 | 275 |
| Lead | 320 | 50.8 | 68.3 | 67.8 |
| Mercury | 37 | 5.9 | 10.0 | 9.38 |
| Nickel | 470 | 74.6 | 106 | 95.1 |
| Selenium | 30 | 4.8 | 11.8 | 13.2 |
| Zinc | 2260 | 358.7 | 502 | 480 |

U - indicates compound was analyzed for but not detected at the given detection limit

* - calculation based on 0.63% solids

** - metals digestion for the different samples was:

Lab Log # 248086; As, Sb, and Se are total recoverable, all others are total.

Lab Log #'s 248087 and 248088; total with sediment/soil digestion.

APPENDIX A

Appendix A - Results of VOA, BNA, Pest/PCB and metal priority pollutant scans -
US Oil, June 1989.

| Sample: Lab Log #: | Effluent 248080 | Effluent 248081 | Effluent 248082 | Transfer Blank 248085 | RAS 248086 | Digested Sludge 248087 | Centrifuge Solids 248088 |
|----------------------------|--------------------|--------------------|--------------------|--------------------------|---------------|---------------------------|-----------------------------|
| Type: | Grab | Grab | Grab | Grab | Grab | Grab | ECO-Comp |
| Date: | 6/13 | 6/13 | 6/14 | 6/13 | 6/14 | 6/14 | 6/13-14 |
| Time: | 1040 | 1455 | 0905 | 1000 | 1355 | 1345 | |
| --- VOA Compounds --- | ug/L | ug/L | ug/L | ug/L | ug/L | ug/Kg dry wt | ug/Kg dry wt |
| Chloromethane | 10 U | 10 U | 10 U | 10 U | 10 U | 250 U | 160 U |
| Bromomethane | 10 U | 10 U | 10 U | 10 U | 10 U | 250 U | 160 U |
| Vinyl Chloride | 10 U | 10 U | 10 U | 10 U | 10 U | 250 U | 160 U |
| Chloroethane | 10 U | 10 U | 10 U | 10 U | 10 U | 250 U | 160 U |
| Methylene Chloride | 5 U | 5 U | 5 U | 5 U | 5 U | 53 J | 20000 D |
| Acetone | 10 U | 10 U | 10 U | 10 U | 10 U | 250 U | 160 U |
| Carbon Disulfide | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| 1,1-Dichloroethene | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| 1,1-Dichloroethane | 5 U | 5 U | 5 U | 5 U | 5 U | 50 J | 100 |
| 1,2-Dichloroethene (total) | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| Chloroform | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| 2-Butanone | 10 U | 10 U | 10 U | 10 U | 10 U | 250 U | 160 U |
| 1,2-Dichloroethane | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| 1,1,1-Trichloroethane | 11 | 12 | 15 | 5 U | 13 | 170 | 110 |
| Carbon Tetrachloride | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| Vinyl Acetate | 10 U | 10 U | 10 U | 10 U | 10 U | 250 U | 160 U |
| Bromodichloromethane | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| 1,2-Dichloropropane | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| Trichloroethene | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| Benzene | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| Dibromochloromethane | 5 U | 5 U | 5 U | 5 U | 5 U | 120 UJ | 78 U |
| 1,1,2-Trichloroethane | 5 U | 5 U | 5 U | 5 U | 5 U | 120 UJ | 78 U |
| Bromoform | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| 4-Methyl-2-Pentanone | 10 U | 10 U | 10 U | 10 U | 10 U | 250 UJ | 160 U |
| 2-Hexanone | 10 U | 10 U | 10 U | 10 U | 10 U | 250 UJ | 160 U |
| 1,1,2,2-Tetrachloroethane | 5 U | 5 U | 5 U | 5 U | 5 U | 120 UJ | 78 U |
| Tetrachloroethene | 5 U | 5 U | 5 U | 5 U | 5 U | 87 J | 78 U |
| Toluene | 5 U | 5 U | 5 U | 5 U | 5 U | 88 J | 78 U |
| Chlorobenzene | 5 U | 5 U | 5 U | 5 U | 5 U | 120 UJ | 78 U |
| trans-1,3-Dichloropropene | 5 U | 5 U | 5 U | 5 U | 5 U | 120 UJ | 78 U |
| Ethylbenzene | 5 U | 5 U | 5 U | 5 U | 5 U | 120 UJ | 78 U |
| cis-1,3-Dichloropropene | 5 U | 5 U | 5 U | 5 U | 5 U | 120 U | 78 U |
| Styrene | 5 U | 5 U | 5 U | 5 U | 5 U | 120 UJ | 78 U |
| Total Xylenes | 5 U | 5 U | 5 U | 5 U | 5 U | 79 J | 78 U |
| 2-Chloroethylvinylether | | | | | | | |
| ----- | | | | | | | |
| Cyanide (ug/L) | 15 | 15 | 15 | | | | |

Appendix A (Continued) - US Oil, June 1989.

| Sample: Lab Log #: Type: Date: | Effluent 248083 ECO-Comp 6/13-14 | Transfer Blank 248085 Grab 6/13 | RAS 248086 Grab 6/14 | Digested Sludge 248087 Grab 6/14 | Centrifuge Solids 248088 ECO-Comp 6/13-14 |
|---|---|--|-------------------------------|---|--|
| --- | ug/L | ug/L | ug/L | ug/Kg dry wt | ug/Kg dry wt |
| Phenol | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Aniline | | | | | |
| Bis(2-Chloroethyl)Ether | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2-Chlorophenol | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 1,3-Dichlorobenzene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 1,4-Dichlorobenzene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Benzyl Alcohol | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 1,2-Dichlorobenzene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2-Methylphenol | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Bis(2-chloroisopropyl)ether | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 4-Methylphenol | 10 U | 10 U | 290 U | 210000 U | 15000 J |
| N-Nitroso-Di-n-Propylamine | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Hexachloroethane | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Nitrobenzene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Isophorone | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2-Nitrophenol | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2,4-Dimethylphenol | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Benzoic Acid | 50 U | 50 U | 1400 U | 1000000 U | 600000 U |
| Bis(2-Chloroethoxy)Methane | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2,4-Dichlorophenol | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 1,2,4-Trichlorobenzene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Naphthalene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 4-Chloroaniline | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Hexachlorobutadiene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 4-Chloro-3-Methylphenol | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2-Methylnaphthalene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Hexachlorocyclopentadiene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2,4,6-Trichlorophenol | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2,4,5-Trichlorophenol | 50 U | 50 U | 1400 U | 1000000 U | 600000 U |
| 2-Chloronaphthalene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2-Nitroaniline | 50 U | 50 U | 1400 U | 1000000 U | 600000 U |
| Dimethyl Phthalate | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Acenaphthylene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 3-Nitroaniline | 50 U | 50 U | 1400 U | 1000000 U | 600000 U |
| Acenaphthene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2,4-Dinitrophenol | 50 U | 50 U | 1400 U | 1000000 U | 600000 U |
| 4-Nitrophenol | 50 U | 50 U | 1400 U | 1000000 U | 600000 U |
| Dibenzofuran | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2,4-Dinitrotoluene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 2,6-Dinitrotoluene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Diethyl Phthalate | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 4-Chlorophenyl-Phenylether | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Fluorene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 4-Nitroaniline | 50 U | 50 U | 1400 U | 1000000 U | 600000 U |
| 4,6-Dinitro-2-Methylphenol | 50 U | 50 U | 1400 U | 1000000 U | 600000 U |
| N-Nitrosodiphenylamine | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 1,2-Diphenylhydrazine | | | | | |
| 4-Bromophenyl-Phenylether | 10 U | 10 U | 290 U | 210000 U | 120000 U |

Appendix A (Continued) - US Oil, June 1989.

| Sample: Lab Log #: Type: Date: | Effluent 248083 ECO-Comp 6/13-14 | Transfer Blank 248085 Grab 6/13 | RAS 248086 Grab 6/14 | Digested Sludge 248087 Grab 6/14 | Centrifuge Solids 248088 ECO-Comp 6/13-14 |
|---|---|--|-------------------------------|---|--|
| | ug/L | ug/L | ug/L | ug/Kg dry wt | ug/Kg dry wt |
| Hexachlorobenzene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Pentachlorophenol | 50 U | 50 U | 1400 U | 2100000 U* | 600000 U |
| Phenanthrene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Anthracene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Di-n-Butyl Phthalate | 1 J | 10 U | 290 U | 210000 U | 120000 U |
| Fluoranthene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Pyrene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Benzidine | | | | | |
| Butylbenzylphthalate | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| 3,3'-Dichlorobenzidine | 20 U | 20 U | 570 U | 420000 U | 250000 U |
| Benzo(a)Anthracene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Chrysene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Bis(2-Ethylhexyl)phthalate | 2 J | 1 J | 39 JB | 11000 JB | 10000 JB |
| Di-n-Octyl Phthalate | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Benzo(b)Fluoranthene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Benzo(k)Fluoranthene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Benzo(a)Pyrene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Indeno(1,2,3-cd)Pyrene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Dibenzo(a,h)Anthracene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| Benzo(g,h,i)Perylene | 10 U | 10 U | 290 U | 210000 U | 120000 U |
| --- Pest/PCB Compounds --- | | | | | |
| alpha-BHC | 0.050 U | 0.050 U | 0.17 U | 85 U | 50 U |
| beta-BHC | 0.050 U | 0.050 U | 0.17 U | 85 U | 50 U |
| delta-BHC | 0.050 U | 0.050 U | 0.17 U | 85 U | 50 U |
| gamma-BHC (Lindane) | 0.050 U | 0.050 U | 0.17 U | 85 U | 50 U |
| Heptachlor | 0.050 U | 0.050 U | 0.17 U | 85 U | 50 U |
| Aldrin | 0.050 U | 0.050 U | 0.17 U | 85 U | 50 U |
| Heptachlor Epoxide | 0.050 U | 0.050 U | 0.17 U | 85 U | 50 U |
| Endosulfan I | 0.050 U | 0.050 U | 0.17 U | 85 U | 50 U |
| Dieldrin | 0.10 U | 0.10 U | 0.33 U | 170 U | 99 U |
| 4,4'-DDE | 0.10 U | 0.10 U | 0.33 U | 170 U | 99 U |
| Endrin | 0.10 U | 0.10 U | 0.33 U | 170 U | 99 U |
| Endosulfan II | 0.10 U | 0.10 U | 0.33 U | 170 U | 99 U |
| 4,4'-DDD | 0.10 U | 0.10 U | 0.33 U | 170 U | 99 U |
| Endosulfan Sulfate | 0.10 U | 0.10 U | 0.33 U | 170 U | 99 U |
| 4,4'-DDT | 0.10 U | 0.10 U | 0.78 | 140 J | 130 |
| Methoxychlor | 0.50 U | 0.50 U | 1.7 U | 850 U | 500 U |
| Endrin Ketone | 0.10 U | 0.10 U | 0.33 U | 170 U | 99 U |
| alpha-Chlordane | 0.50 U | 0.50 U | 1.7 U | 850 U | 500 U |
| gamma-Chlordane | 0.50 U | 0.50 U | 1.7 U | 850 U | 500 U |
| Toxaphene | 1.0 U | 1.0 U | 3.3 U | 1700 U | 990 U |
| Aroclor-1016 | 0.50 U | 0.50 U | 1.7 U | 850 U | 500 U |
| Aroclor-1221 | 0.50 U | 0.50 U | 1.7 U | 850 U | 500 U |
| Aroclor-1232 | 0.50 U | 0.50 U | 1.7 U | 850 U | 500 U |
| Aroclor-1242 | 0.50 U | 0.50 U | 1.7 U | 850 U | 500 U |
| Aroclor-1248 | 0.50 U | 0.50 U | 1.7 U | 850 U | 500 U |
| Aroclor-1254 | 1.0 U | 1.0 U | 3.3 U | 1700 U | 990 U |
| Aroclor-1260 | 1.0 U | 1.0 U | 3.3 U | 1700 U | 990 U |
| Endrin Aldehyde | | | | | |

Appendix A (Continued) - US Oil, June 1989.

| Sample: Lab Log #: Type: Date: | Effluent 248084 US Oil-Comp 6/13-14 | Effluent 248083 ECO-Comp 6/13-14 | Transfer Blank 248085 Grab 6/13 | RAS 248086 Grab 6/14 | Digested Sludge 248087 Grab 6/14 | Centrifuge Solids 248088 ECO-Comp 6/13-14 |
|---|--|---|--|-------------------------------|---|--|
| --- Priority pollutant metals ** --- | ug/L | ug/L | ug/L | ug/L | mg/Kg dry wt | mg/Kg dry wt |
| Antimony | 2 | 2 U | 1 U | 18 | 1.00 U | 1.80 |
| Arsenic | 5 U | 5 U | 1 U | 70 | 11.1 | 15.8 |
| Beryllium | 1 U | 1 U | 1 U | 2 | 1.03 U | 0.56 U |
| Cadmium | 2 U | 2 U | 2 U | 12 | 2.05 U | 2.63 |
| Chromium | 7 | 8 | 5 U | 584 | 125 | 119 |
| Copper | 7 | 4 | 2 U | 1230 | 256 | 275 |
| Lead | 2 U | 2 U | 1 U | 320 | 68.3 | 67.8 |
| Mercury | 0.1 | 0.1 | 0.1 | 37 | 10.0 | 9.38 |
| Nickel | 20 | 20 | 10 U | 470 | 106 | 95.1 |
| Selenium | 5 U | 5 U | 1 U | 30 | 11.8 | 13.2 |
| Silver | 3 U | 3 U | 3 U | 3 U | 3.08 U | 1.68 U |
| Thallium | 2 U | 2 U | 1 U | 2 U | 1.03 U | 0.56 U |
| Zinc | 123 | 91 | 4 U | 2260 | 502 | 480 |

U - indicates compound was analyzed for but not detected at the given detection limit

J - indicates an estimated value when result is less than specified detection limit

B - This flag is used when the analyte is found in the blank as well as the sample. Indicates possible/probable blank contamination

M - indicates an estimated value of analyte found and confirmed by analyst but with low spectral match parameters

D - value from analysis of a diluted sample

UJ - indicates compound was analyzed for but not detected at the given detection limit, and the internal standard on which detection limit quantification was based was outside acceptance limits

JB - comment J plus comment B

* - lab contamination suspected in original analysis. Listed result was a re-run at a greater dilution factor.

** - metals digestion for the different samples was:
Lab Log #'s 248083, 248084, and 248085; total recoverable for all metals except Hg, which was total.
Lab Log # 248086; As, Sb, and Se are total recoverable, all others are total.
Lab Log #'s 248087 and 248088; total with sediment/soil digestion.

APPENDIX B

Laboratory Procedure Review Sheet

Discharger: *US Oil*

Date: *6/13/89*

Discharger representative: *Shauna-May Tecle-Mariam*

Ecology reviewer: *Heffner*

Instructions

Questionnaire for use reviewing laboratory procedures. Circled numbers indicate work is needed in that area to bring procedures into compliance with approved techniques. References are cited to help give guidance for making improvements. References cited include:

Ecology = Department of Ecology Laboratory User's Manual, December 8, 1986.

SM = APHA-AWWA-WPCF, Standard Methods for the Examination of Water and Wastewater, 16th ed., 1985.

SSM = WPCF, Simplified Laboratory Procedures for Wastewater Examination, 3rd ed., 1985.

Sample Collection Review

1. Are grab, hand composite, or automatic composite samples collected for influent and effluent BOD and TSS analysis?
2. If automatic compositor, what type of compositor is used?
The compositor should have pre and post purge cycles unless it is a flow through type. Check if you are unfamiliar with the type being used.
3. Are composite samples collected based on time or flow?
*~30 mLs every 4 minutes was our measurement **
4. What is the usual day(s) of sample collection? *7/wk*
5. What time does sample collection usually begin? *8AM-8AM*
6. How long does sample collection last? *24 hours*
7. How often are subsamples that make up the composite collected? *~ 4 min*
8. What volume is each subsample? *~30 mLs ** ← *Ecology measurement*
9. What is the final volume of sample collected? *~ 1.5-2 gallons*
10. Is the composite cooled during collection? *yes*

* sampler volume doubled for inspection - usual volume ~ 15 mLs

11. To what temperature? $\approx 4^{\circ}\text{C}$
The sample should be maintained at approximately 4 degrees C (SM p41, #5b: SSM p2).
12. How is the sample cooled?
Mechanical refrigeration or ice are acceptable. Blue ice or similar products are often inadequate.
13. How often is the temperature measured? *monthly*
The temperature should be checked at least monthly to assure adequate cooling.
14. Are the sampling locations representative? *OK*
15. Are any return lines located upstream of the influent sampling location? *N/A*
This should be avoided whenever possible.
16. How is the sample mixed prior to withdrawal of a subsample for analysis? *OK*
The sample should be thoroughly mixed.
17. How is the subsample stored prior to analysis? *OK*
The sample should be refrigerated (4 degrees C) until about 1 hour before analysis, at which time it is allowed to warm to room temperature.
18. What is the cleaning frequency of the collection jugs? *daily*
The jugs should be thoroughly rinsed after each sample is complete and occasionally be washed with a non-phosphate detergent.
19. How often are the sampler lines cleaned? *replaced monthly*
Rinsing lines with a chlorine solution every three months or more often where necessary is suggested.

pH Test Review

1. How is the pH measured? *continuous meter - monthly comparison to lab meter*
A meter should be used. Use of paper or a colorimetric test is inadequate and those procedures are not listed in Standard Methods (SM p429).
2. How often is the meter calibrated? *continuous meter - monthly*
The meter should be calibrated every day it is used.
3. What buffers are used for calibration? —
Two buffers bracketing the pH of the sample being tested should be used.

If the meter can only be calibrated with one buffer, the buffer closest in pH to the sample should be used. A second buffer, which brackets the pH of the sample should be used as a check. If the meter cannot accurately determine the pH of the second buffer, the meter should be repaired.

BOD Test Review

1. What reference is used for the BOD test? *Std Mthds*
Standard Methods or the Ecology handout should be used.
2. How often are BODs run? *weekly*
The minimum frequency is specified in the permit.
- ③ How long after sample collection is the test begun? *within 4 hours*
The test should begin within 24 hours of composite sample completion (Ecology Lab Users Manual p42). Starting the test as soon after samples are complete is desirable.
keep sample refrigerated until 1 hour before analysis
4. Is distilled or deionized water used for preparing dilution water?
+
5. Is the distilled water made with a copper free still? *glass*
Copper stills can leave a copper residual in the water which can be toxic to the test (SSM p36).
6. Are any nitrification inhibitors used in the test? *no* What?
2-chloro-6(trichloro methyl) pyridine or Hach Nitrification Inhibitor 2533 may be used only if carbonaceous BODs are being determined (SM p 527, #4g: SSM p 37).
7. Are the 4 nutrient buffers of powder pillows used to make dilution water?
If the nutrients are used, how much buffer per liter of dilution water are added?
1 mL per liter should be added (SM p527, #5a: SSM p37).
8. How often is the dilution water prepared? *weekly*
Dilution water should be made for each set of BODs run.
- ⑨ Is the dilution water aged prior to use? *24 hours*
Dilution water with nitrification inhibitor can be aged for a week before use (SM p528, #5b).
Dilution water without inhibitor should not be aged.
10. Have any of the samples been frozen? *no*
If yes, are they seeded?
Samples that have been frozen should be seeded (SSM p38).
11. Is the pH of all samples between 6.5 and 7.5? *yes*
If no, is the sample pH adjusted?
The sample pH should be adjusted to between 6.5 and 7.5 with 1N NaOH or 1N H2SO4 if 6.5 > pH > 7.5 if caustic alkalinity or acidity is present (SM p529, #5e1: SSM p37).
High pH from lagoons is usually not caustic. Place the sample in the dark to warm up, then check the pH to see if adjustment is necessary.

If the sample pH is adjusted, is the sample seeded?
The sample should be seeded to assure adequate microbial activity if the pH is adjusted (SM p528, #5d).

12. Have any of the samples been chlorinated or ozonated? *no*
If chlorinated are they checked for chlorine residual and dechlorinated as necessary?

How are they dechlorinated?

Samples should be dechlorinated with sodium sulfite (SM p529, #5e2: SSM p38), but dechlorination with sodium thiosulfate is common practice. Sodium thiosulfate dechlorination is probably acceptable if the chlorine residual is < 1-2 mg/L.

If chlorinated or ozonated, is the sample seeded?

The sample should be seeded if it was disinfected (SM p528, #5d&5e2: SSM p38).

13. Do any samples have a toxic effect on the BOD test? *no*
Specific modifications are probably necessary (SM p528, #5d: SSM p37).

14. How are DO concentrations measured? *Y5/ meter*

If with a meter, how is the meter calibrated? *air* *suggest use one*

Air calibration is adequate. Use of a barometer to determine saturation is desirable, although not mandatory. Checks using the Winkler method of samples found to have a low DO are desirable to assure that the meter is accurate over the range of measurements being made.

How frequently is the meter calibrated? *before use*

The meter should be calibrated before use.

15. Is a dilution water blank run? *yes*

A dilution water blank should always be run for quality assurance (SM p527, #5b: SSM p40, #3).

What is the usual initial DO of the blank? *≈ 8.3*

The DO should be near saturation; 7.8 mg/L @ 4000 ft, 9.0 mg/L @ sea level (SM p528, #5b). The distilled or deionized water used to make the dilution water may be aged in the dark at ~20 degrees C for a week with a cotton plug in the opening prior to use if low DO or excess blank depletion is a problem.

What is the usual 5 day blank depletion? *< 0.2*

The depletion should be 0.2 mg/L or less. If the depletion is greater the cause should be found (SM p527-8, #5b: SSM p41, #6).

16. How many dilutions are made for each sample? *3*

At least two dilutions are recommended. The dilutions should be far enough apart to provide a good extended range (SM p530, #5f: SSM p41).

17. Are dilutions made by the liter method or in the bottle?

Either method is acceptable (SM p530, #5f).

18. How many bottles are made at each dilution? *3*

How many bottles are incubated at each dilution? *2*

When determining the DO using a meter only one bottle is necessary. The DO is measured, then the bottle is sealed and incubated (SM p530, #5f2)

When determining the DO using the Winkler method two bottles are necessary. The initial DO is found of one bottle and the other bottle is sealed and incubated (Ibid.).

19. Is the initial DO of each dilution measured? *yes*

What is the typical initial DO?

The initial DO of each dilution should be measured. It should approximate saturation (see #14).

20. What is considered the minimum acceptable DO depletion after 5 days?

What is the minimum DO that should be remaining after 5 days?

The depletion should be at least 2.0 mg/L and at least 1.0 mg/L should be left after 5 days (SM p531, #6: SSM p41). *low BOD in sample prevents this*

(21) Are any samples seeded? *yes*

Which? *all*

What is the seed source? *settle activated sludge*

Primary effluent or settled raw wastewater is the preferred seed.

Secondary treated sources can be used for inhibited tests (SM p528, #5d: SSM p41).

How much seed is added to each sample? *20 mLs/L*

Adequate seed should be used to cause a BOD uptake of 0.6 to 1.0 mg/L due to seed in the sample (SM p529, #5d).

→ How is the BOD of the seed determined? *with seeded blank*

Dilutions should be set up to allow the BOD of the seed to be determined just as the BOD of a sample is determined. This is called the seed control (SM p529, #5d: SSM p41).

(22) What is the incubator temperature? *OK*

The incubator should be kept at 20 +/- 1 degree C (SM p531, #5i: SSM p40, #3).

→ How is incubator temperature monitored? *suggest*

A thermometer in a water bath should be kept in the incubator on the same shelf as the BODs are incubated.

How frequently is the temperature checked? *daily*

The temperature should be checked daily during the test. A temperature log on the incubator door is recommended.

How often must the incubator temperature be adjusted? *seldom*

Adjustment should be infrequent. If frequent adjustments (every 2 weeks or more often) are required the incubator should be repaired.

Is the incubator dark during the test period? *yes*

Assure the switch that turns off the interior light is functioning.

23. Are water seals maintained on the bottles during incubation? *yes*

Water seals should be maintained to prevent leakage of air during the incubation period (SM p531, #5i: SSM p40, #4).

- ②4 Is the method of calculation correct? *should properly correct for seed*
 Check to assure that no correction is made for any DO depletion in the blank and that the seed correction is made using seed control data.

Standard Method calculations are (SM p531, #6):

for unseeded samples;

$$\text{BOD (mg/L)} = \frac{D1 - D2}{P}$$

for seeded samples;

$$\text{BOD (mg/L)} = \frac{(D1 - D2) - (B1 - B2)f}{P}$$

Where: D1 = DO of the diluted sample before incubation (mg/L)
 D2 = DO of diluted sample after incubation period (mg/L)
 P = decimal volumetric fraction of sample used
 B1 = DO of seed control before incubation (mg/L)
 B2 = DO of seed control after incubation (mg/L)

$$f = \frac{\text{amount of seed in bottle D1 (mL)}}{\text{amount of seed in bottle B1 (mL)}}$$

Total Suspended Solids Test Review

Preparation

1. What reference is used for the TSS test? *Std Mthds*
2. What type of filter paper is used?
Std. Mthds. approved papers are: Whatman 934AH (Reeve Angel), Gelman A/E and Millipore AP-40 (SM p95, footnote: SSM p23)
3. What is the drying oven temperature? *OK*
The temperature should be 103-105 degrees C (SM p96, #3a: SSM p23).
4. Are any volatile suspended solids tests run? *Not on ditch*
If yes--What is the muffle furnace temperature?
The temperature should be 550+/- 50 degrees C (SM p98, #3: SSM p23).
5. What type of filtering apparatus is used?
Gooch crucibles or a membrane filter apparatus should be used (SM p95, #2b: SSM p23).
6. How are the filters pre-washed prior to use? *OK*
The filters should be rinsed 3 times with distilled water (SM p23, #2: SSM p23, #2).

Are the rough or smooth sides of the filters up? *OK*
The rough side should be up (SM p96, #3a: SSM p23, #1)

How long are the filters dried? *60 minutes*
The filters should be dried for at least one hour in the oven. An additional 20 minutes of drying in the furnace is required if volatile solids are to be tested (Ibid).
- How are the filters stored prior to use? *OK*
The filters should be stored in a dessicator (Ibid).
7. How is the effectiveness of the dessicant checked? *OK*
All or a portion of the dessicant should have an indicator to assure effectiveness.

Test Procedure

8. In what is the test volume of sample measured? *~250 mls*
The sample should be measured with a wide tipped pipette or a graduated cylinder.
9. Is the filter seated with distilled water? *OK*
The filter should be seated with distilled water prior to the test to avoid leakage along the filter sides (SM p97, #3c).

10. Is the entire measured volume always filtered? *OK*

The entire volume should always be filtered to allow the measuring vessel to be properly rinsed (SM p97, #3c: SSM p24, #4).

11. What are the average and minimum volumes filtered?

| | Minimum | Average |
|----------|---------|---------|
| Influent | | |
| Effluent | | |

12. How long does it take to filter the samples? *< 5 minutes*

| | Time |
|----------|------|
| Influent | |
| Effluent | |

13. How long is filtering attempted before deciding that a filter is clogged? *25 minutes maximum*

Prolonged filtering can cause high results due to dissolved solids being caught in the filter (SM p96, #1b). We usually advise a five minute filtering maximum.

14. What do you do when a filter becomes clogged? *pitch*

The filter should be discarded and a smaller volume of sample should be used with a new filter.

15. How are the filter funnel and measuring device rinsed onto the filter following sample addition? *OK*

Rinse 3x's with approximately 10 mLs of distilled water each time (?).

16. How long is the sample dried? *60 minutes*

The sample should be dried at least one hour for the TSS test and 20 minutes for the volatile test (SM p97, #3c; p98, #3: SSM p24, #4). Excessive drying times (such as overnight) should be avoided.

17. Is the filter thoroughly cooled in a dessicator prior to weighing? *OK*

The filter must be cooled to avoid drafts due to thermal differences when weighing (SM p97, #3c: SSM p97 #3c).

18. How frequently is the drying cycle repeated to assure constant filter weight has been reached (weight loss < 0.5 mg or 4%, whichever is less: SM p97, #3c)? *suggest*

We recommend that this be done at least once every 2 months.

19. Do calculations appear reasonable?

Standard Methods calculation (SM p97, #3c).

$$\text{mg/L TSS} = \frac{(A - B) \times 1000}{\text{sample volume (mL)}}$$

where: A = weight of filter + dried residue (mg)
B = weight of filter (mg)