

Wastewater Treatment System Operation and Maintenance Manual

for

Inland Empire Paper Company
Spokane, WA

April, 2019

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Acronyms and Abbreviations

BOD ₅	-	Biochemical oxygen demand
DAF	-	Dissolve air flotation
DCS	-	Distributed control system
DO	-	Dissolved oxygen
gpm	-	Gallons per minute
hp	-	Horsepower
HRT	-	Hydraulic residence time
IEP	-	Inland Empire Paper Company
kV	-	Kilovolt
MGD	-	Million gallons per day
MLSS	-	Mixed liquor suspended solids
NCCW	-	Non-contact cooling water
NPDES	-	National pollution discharge elimination system
MDT	-	Metric dry tons
O&M	-	Operation and maintenance manual
O ₂	-	Oxygen
ONP	-	Old newspaper
OUR	-	Oxygen utilization rate
PAX	-	Poly-aluminum chloride
RAS	-	Return activated sludge
RPM	-	Rotations per minute
RTD	-	Resistance temperature detector
SCFM	-	Standard cubic foot per minute
TMDL	-	Total maximum daily load
TMP	-	Thermo-mechanical pulping
TSS	-	Total suspended solids
VFD	-	Variable frequency drive
VOC	-	Volatile organic compound
WAS	-	Waste activated sludge
WQBEL	-	Water quality based effluent limits
WWTS	-	Wastewater treatment system

1. TREATMENT SYSTEM OPERATING PROGRAM

1.1 Introduction

Inland Empire Paper Company (IEP) produces pulp and newsprint from a thermo-mechanical pulping system (TMP), and the recycling of old newsprint processes. Wastewater from these manufacturing processes is treated in a facility designed to comply with limits on Biological Oxygen Demand (BOD₅), Total Suspended Solids (TSS), Total Zinc, Total Lead, Total Cadmium, Total Phosphorus and pH range as specified in NPDES permit no. WA-000082-5. The following sections outline the treatment system, the unit processes, control targets and/or set point ranges, operational procedures including upsets, and maintenance procedures and schedules.

Inland Empire Paper Company (IEP) produces newsprint and specialty paper products (Standard Industrial Classification (SIC) Code 2621) at its Millwood, Washington pulp and paper mill. Present production ranges from 500 to 580 metric dry tons (MDT) per day of finished product. The paper machine has a design capacity of 675 tons per day. The paper machine was installed in 2001.

The pulp mill (SIC Code 2611) can produce up to 775 tons of pulp per day from its Thermo-Mechanical Pulping (TMP) Systems and 350 tons of recycled deinked pulp per day from recycled old newsprint (ONP), magazines and office paper. Wastewater from the pulp mill and paper machine is treated by the mill's wastewater treatment plant prior to discharge to the Spokane River. IEP has implemented numerous water conservation, reclamation and reuse projects over the past several years, resulting in average process wastewater flows of approximately 3.0 million gallons per day (MGD). Approximately 3.6 MGD of non-contact cooling water is utilized by the mill for equipment cooling and is discharged to the launder ring of the secondary clarifier.

1.2 Baseline Operating Conditions:

The IEP wastewater treatment plant is a state-of-the-art facility consisting of a group of unit operations and processes performing flow and load equalization, super oxygenation, primary solids settling, activated sludge treatment, moving beds, secondary solids settling, and sludge dewatering. The typically treated flow through the system is 3.0 million gallons per day.

All process waters on-site are collected in the pump house sump pit before moving into the effluent system where electric, positive-suction pumps send water to the Speece Cone system. The Speece cone system super-oxygenates the water to approximately 50-60 mg/L O₂ to mitigate odor and begin BOD₅ removal. The super-oxygenated water then enters the Primary clarifier for solids settling. To mitigate flow spikes through primary treatment, a variable slip stream of flow off the pump house is diverted to equalization tanks. Additionally, during anticipated heavy BOD loading conditions, the 75 ft surge tank is used to mitigate loading spikes frequently observed during specialty paper production. Once mill production returns to low flow, low BOD conditions, the sequestered water is metered back into the head of the WWTS.

After primary treatment, wastewater collects in a clear well that feeds both secondary treatment as well as the Connustrenner. The Connustrenner system removes approximately 1.6 MGD of water for reuse within the mill, the remaining primary treated water is sent to the MBBR system to begin the activated sludge process. Three MBBRs are used in IEP's WWTS; No. 1 is in series while No. 2 and 3 are in parallel (§3.2, Figure 1). Number 1 MBBR treats slip stream average of 1000 gpm, while No. 2 and 3 MBBRs split the total treated flow (~1.5MGD each). Following the MBBRs, the wastewater gravity flows into the Orbal oxidation ditch.

The MLSS within the Orbal oxidation ditch is altered between 4000-6000 mg/L depending on anticipated BOD₅ loading due to the current production schedule. Weirs can be raised or lowered to alter the residence time in the Orbal should it be required. The dissolved oxygen (DO) concentration in the inner Orbal has a target of at least 3.0 mg/L-DO, with large negative deviations resulting in revised production schedules (e.g. low DO results in revised production to reduce BOD₅ loading to system). After exiting the Orbal system the wastewater gravity flows to the secondary clarifier.

The secondary clarifier functions as a solids settler and thickener. Sludge is removed from the clarifier and either sent to waste (WAS) or to re-seed the Orbal (RAS), while clarifier water overflows and is mixed with non-contact cooling water prior to final discharge.

Sludge drawn from the primary clarifier together with WAS from the secondary clarifier and DAF sludge from the TMP mill are combined and processed through IEP's sludge handling system. The sludge is dewatered and is either further processed through the fluidized bed combustor and/or sent to the sludge barn for disposal.

1.3 Reduced Production Operating Procedures:

Should loading to the wastewater treatment system become substantially lower resulting in an overall reduction in system water quality, several options are available to address the problem.

For short term loading changes e.g. extend machine down (2-5 days) starch may be directly added to the MBBR system and the Orbal to maintain adequate food for the system biology.

For longer term loading changes it may be possible to remove one or more MBBRs from the overall system flow. This would result in a lowered overall system capacity for BOD₅ removal but would better align the system for treating a reduced load. By balancing the aerobic treatment capacity to the BOD₅ load the system would function better and a healthier more stable population of bacteria in the activated sludge would be possible.

1.4 System Upset Operating Procedures:

The IEP wastewater treatment plant consists of a group of unit operations and processes performing super oxygenation, primary solids settling, activated sludge treatment, moving beds, secondary solids settling, and sludge dewatering. These unit operations and processes are composed of the major equipment and systems described in the subsequent sections. The applicable description can guide troubleshooting for unit specific issues.

1.5 Site Activities that Affect the Volume or Character of Wastewater:

The volume and character of an industrial effluent are extremely volatile by nature. Differing production schedules can cause large loading changes to the effluent system. Surge tanks are used on-site to buffer flow swings from affecting the WWTS and alteration of the MLSS in the Orbal allows for assimilation of different levels of BOD₅ loading.

2. FACILITY INFORMATION AND RESPONSIBLE INDIVIDUALS

2.1 General Information: §173-240-150(2)(a)

Facility Name Inland Empire Paper Company

Type of Facility Newsprint Manufacturing

Facility Address 3320 N Argonne Rd

City Spokane State WA ZIP 99212

County Spokane Tel. Number (509) 924 - 1911

Operator Name Kevin Rasler – IEPCO President and General Manager

Operator Address 3320 N Argonne Rd

City Spokane State WA ZIP 99212

County Spokane Tel. Number (509) 924 - 1911

2.2 Key Personnel Contact Information: §173-240-150(2)(a)

Contact List	
Key Facility Personnel	
Doug Krapas <i>Environmental Manager</i>	Office: (509) 720 - 5815
	Emergency: (208) 661 - 5526 (cell phone)
Kevin Davis <i>Production Manager</i>	Office: (509) 720 - 5826
	Emergency: (509) 448 - 8824
Luke Huntley <i>Paper Machine Superintendent</i>	Office: (509) 720 - 5819
	Emergency: (509) 998 - 1334
Cody Murdock <i>Pulp Mill Superintendent</i>	Office: (509) 720 - 5824
	Emergency: (509) 386 - 0551
John Nelson <i>Maintenance Superintendent</i>	Office: (509) 720 - 5864
	Emergency: (509) 936 - 5898
Ben Carleton <i>Process Engineer</i>	Office: (509) 720 - 5827
	Emergency: (208) 505-4503
David Demers <i>Operations Technician</i>	Office: (509) 720 - 5831
	Emergency: (208) 659 - 3530
TJ Eixenberger <i>Project Engineer</i>	Office: (509) 720 - 5862
	Emergency: (509) 386 - 0551
Kevin Rasler <i>President & General Manager</i>	Office: (509) 924 - 1911
	Emergency: (509) 710 - 9236

3. WASTEWATER TREATMENT PLANT SYSTEM DESCRIPTION

3.1 Introduction and Overview:

The IEP wastewater treatment plant is a state-of-the-art facility consisting of a group of unit operations and processes performing flow and load equalization, super oxygenation, primary solids settling, activated sludge treatment, moving beds, secondary solids settling, and sludge dewatering. These unit operations and processes are composed of the major equipment and systems described in the subsequent sections; where applicable design criteria and capacity are presented.

3.2 Wastewater Treatment System Schematic: §173-240-150(2)(b),(d)

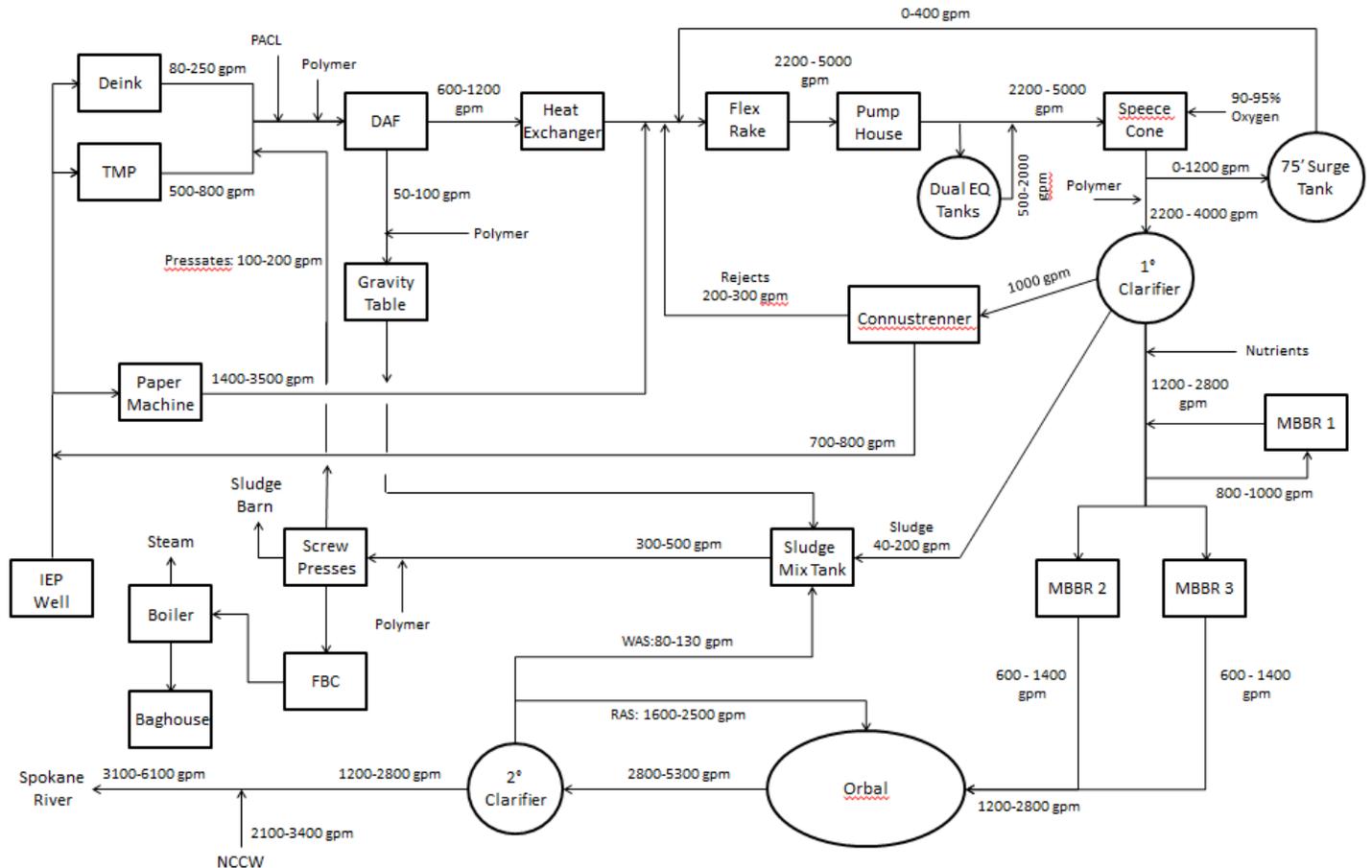


Figure 1: Wastewater Treatment Schematic

3.3 Process Unit Descriptions and Design Criteria: §173-240-150(2)(b),(c),(d),(f)

3.3.1 Flex Rake

The flex rake is a mechanically cleaned bar rack and triggers based on sump pit level. During operation this coarse screen removes large chips and debris from the influent wastewater prior to entering the effluent pumps.

3.3.2 Pump house

The wastewater pumping system includes three 885-rpm positive suction pumps, each capable of providing flow capacity up to 4500 gpm. One pump is powered by the mill's 63-kV power supply; another by the mill's 110-kV power supply; and the third is diesel powered for standby in the event of a total power failure. The separate electrical feeds are supplied to the two electrical pumps to lessen the impact of power outages.

All the mill sewer trenches are channeled to the effluent pump house sump. The effluent pumps transfer the collected sewer flows to the 100ft Primary Clarifier. The sump has no overflow. The pumping system is operated so that one pump

is the lead pump and the other two are standby backups. The lead pump is on a VFD and is controlled by a level transmitter measuring the sump pit. The second pump is controlled by a mechanical hi level float switch and the third pump (diesel) is controlled by a high high level float switch. Therefore, if the first pump does not keep up with the flow from the mill, the second pump will start pumping and if the two pumps do not keep up, the third will start pumping. If the secondary or tertiary pump startup audio and visual alarms are triggered within the mill to alert crews of high flows.

In case of a power failure, the diesel pump will start up when the high-level sump probe is triggered. All three pumps have a combined capacity of 13,500 gpm. The highest recorded flow from the mill was approximately 6,000 gpm.

3.3.3 Equalization (EQ) Tanks

A portion of the flow sent forth from the influent pumps at the pump house is diverted to the EQ tanks. The tanks are 100-foot diameter and 20-foot deep to contain roughly 1.2 million gallons each. The tanks are designed to operate with three 15 HP floating mixers and one 50 HP floating aerator to keep the tank contents well-mixed and aerated. From the EQ tanks, wastewater will be pumped by one of two 3,500 self-priming, centrifugal pumps back to the pump house and move forward with the rest of the water feeding the Speece cone. This is the newest addition to IEP's WWTS, and optimization of operations remains ongoing.

3.3.4 Speece Cone

The Speece cone system consists of a hollow stainless steel cone with no internal mixers, baffles or moving parts. The influent and effluent piping is 12" in diameter and capable of passing dirty wastewater without clogging. The cone super oxygenates the water that passes through by creating an intense bubble swarm at the inlet of the cone. The geometry of the cone and the buoyant force of the bubbles do not allow any the bubbles to exit, thereby ensuring complete dissolution. An onsite vacuum swing absorption oxygen generator is used to provide a nearly pure oxygen source from ambient air. The combination of a pure oxygen stream and a slight pressure increase in the cone make it possible to increase the dissolved oxygen concentration to above 60mg/L in the wastewater (up to 2100lb of O₂ per day).

The Speece cone system is located just downstream of the effluent pumps and oxygenates 100% of the water that leaves the effluent pump house. This includes all flows to the primary clarifier, reclaimed effluent waster, and all water directed to surge tanks used on-site for surge control.

The system was installed in a proactive effort to increase BOD₅ removal in the primary clarifier and to offset any septic conditions that may develop in the primary clarifier due to IEP's ongoing effort to reduce wastewater discharge flows.

3.3.5 100ft Primary Clarifier

The entire mill sewer flow is handled by the 100ft diameter x 12ft side water depth primary clarifier. The main purpose of the primary clarifier is to remove suspended solids from the wastewater prior to aerobic treatment. The tank has a 24-inch inlet pipe and two 8-inch diameter sludge withdrawal pipes. The clarifier mechanism rakes sludge to a center draw-off. The mechanical rake arms include a surface skimmer to remove floatable materials for return to the bar racks at the pumping station. The clarifier rake arm mechanism travels at 0.04 rpm and plows sludge to a center draw-off pipe. Sludge is removed from the clarifier by two pumps - one operating as the primary pump and the other as a standby pump; while sludge withdrawal is dependent on the solids inventory in the clarifier. A sludge depth of 4 to 6 feet is desirable for good sludge compaction in the clarifier. A cationic polymer is added at the clarifier inlet to flocculate the suspended particles to promote settling and sludge solids removal.

The clarifier rake arm is equipped with torque switches that provide alarms due to high sludge loads or problems with the rake arm drive. The torque switches are set to alarm at 70% of load and fail at 80% maximum rake arm drive load. Motor loading of the rake arm is also monitored on the DCS system and programmed to alarm at 65% of maximum load.

Table 1: Primary Clarifier Design Criteria

Parameter	Units	Value	
Diameter	ft	100	
Water Depth	ft	12	
Planar Surface	ft ²	7,850	
Volume	gal	705,000	
Parameter	Units	Value	
		Average	Max
Flowrate to clarifier	MGD	5.5	6.3
Overflow rate	gal/ft ² /d	700	800
Hydraulic Retention Time (HRT)	hrs	3.0	

Influent and effluent solids are monitored five days per week through lab analysis, and an on-line pH meter is located at the outfall of the primary clarifier. Upper control limits are established for these solids to trigger corrective action. At high solids loading of the influent, a mill search is initiated to find the solids losses (i.e.: leaking packing on pumps, agitators, or process equipment). High primary clarifier effluent solids are indicative of excess sludge inventory in the clarifier requiring that the sludge withdrawal rate be increased.

The effluent from the 100-ft primary clarifier flows into a clear well tank where it is then pumped forward in the system by either an electrically powered pump or a diesel back up in the event of power failure. From the clear well pumps water is sent to either the Moving Bed Biofilm Reactors (MBBRs) or the Connustrenner. Since the diversion to the Connustrenner is downstream from the primary clarifier, the only components treating the full waste flow are the wastewater pump station and the primary clarifier. The biological portion of the treatment system is subject to significantly lower flow and loading than the primary clarifier because of removal in the primary clarifier and the diversion to recycle.

Because the flow to the primary clarifier is limited (through carefully operated surge control) to a maximum of 5.5 MGD, after diverting the maximum amount of 2.3 MGD to the Connustrenner, the maximum flow that would be seen by the subsequent secondary system is 4.0 MGD (instantaneous flow of about 2,800 gpm).

3.3.6 Connustrenner

The Connustrenner is a compact, highly efficient, self-cleaning fractionating type filter device used to reclaim primary clarifier effluent for recycling in the pulp mill. Approximately 2.3 MGD of effluent from the primary clarifier is diverted to the Connustrenner resulting in approximately 35% clean filtrate, or 0.8 MGD. This practice reduces water use by taking advantage of an available water stream with satisfactory quality for some uses within the plant.

Since the diversion to the Connustrenner is downstream from the primary clarifier, the only components treating the full waste flow are the wastewater pump station and the primary clarifier. The biological portion of the treatment system is subject to significantly lower flow and loading than the primary clarifier because of removal in the primary clarifier and the diversion to recycle.

3.3.7 Moving Bed Biofilm Reactors (MBBRs)

An MBBR system consists of an activated sludge aeration system where the sludge is concentrated on recycled plastic carriers. These carriers have a large surface area that provides optimal contact between water, air, and bacteria. The bacteria/activated sludge grow on the internal surface of the carriers and break down the organic matter from the waste water. The aeration system keeps the carriers with activated sludge in motion. Only the excess bacteria growth will separate from the carriers and flow with the treated water forward in the effluent treatment system. The MBBRs installed at IEP are intended to be a “roughing” BOD₅ treatment that works in tandem with the aeration basin directly downstream.

At IEP the effluent from the 100ft primary clarifier is directed to one of three MBBR systems for enhanced BOD₅ removal and minimization of filamentous bacteria formation. MBBR #1 withdraws 800 – 1,000 gpm (1.2 - 1.5 MGD) off a side stream of the primary clarifier effluent as a pre-polishing step. The flow is then returned to the main line where it is then split in parallel to MBBR #2 and #3 (Figure 1). The MBBR feed stream is fed with a proportional share of nutrients as

required for optimum BOD₅ removal, and each tank includes a defoamer system to minimize foam generation during operation.

Table 2: MBBR Design Criteria

Parameter	Units	MBBR 1		MBBR 2 & 3	
		Value		Value ¹	
Media Surface Area	m ² /m ³	660		660	
Tank Media Fraction	%	70		44	
Tank Volume	gal	116,000		250,000	
Aeration Rate	SCFM	2,000		5000	
Parameter	Units	MBBR 1		MBBR 2 & 3	
		Average	Max	Average ¹	Max ¹
Influent Flow	gpm	1,000	1,000	1040	1600
BOD ₅ Loading	lbs/d	4,150	5,190	8,950	11,200
BOD ₅ Effluent	mg/L	660	1,100-1,600	660	1,100-1,600
BOD ₅ Effluent	mg/L	400	660-960	400	660-960

1 – Values are for each MBBR

3.3.8 Orbal Aeration Basin

The aeration basin system consists of a 2.1 million gallon (MG) tank, divided into three concentric oxidation ditch channels (“Orbal” configuration). Each oxidation ditch channel is 20-foot wide with a 14.2-foot normal water depth. The volume of the outside channel is 0.98 MG, while the middle and inside channels are 0.70 MG and 0.42 MG, respectively. Aeration of the tank sections is by disk aerators manufactured by Evoqua Water Technologies. The discs aerate the tank and provide mixing by rotating on horizontal shafts. The outer two (2) Orbal channels have six (6) shafts and the inner Orbal channel has four (4) shafts. The shafts are driven by six (6) 50-hp motors and four (4) 30-hp motors. There are a total of 564 aeration disks installed on the aeration shafts in the Orbal channels. The drive system has been optimized to turn the rotors at a speed that imparts the maximum amount of aeration possible for this type of aerator according to manufacturer recommendations. The activated sludge system operates with recycle activated sludge (RAS) and combined effluent from MBBR #2 and MBBR #3 feeding into the outside channel. Flow progresses through ports successively into the middle and inner channels. The inner channel discharges over a manually-adjustable overflow weir. There is a defoamer application on the discharge from the Orbal to the secondary clarifier to reduce excessive DO that may limit the ability of the sludge to settle.

The outer channel of the aeration basin is operated with a low dissolved oxygen (DO) concentration of less than or equal to 1.0 mg/L. The middle channel is operated at about 1.6 to 2.0 mg/L DO and the inner channel is operated at 2.5 to 3.5 mg/L.

Improvements to the aeration basin in the past 15-20 years have included increasing the aeration capacity by adding aeration disks and increasing aerator shaft rotational speed up to the maximum speed that can be handled by the drive motors. Originally, the outer channel was designed to operate in anoxic or semi-anoxic conditions (0 mg/L DO). The additional aerators in the outer channel improve BOD₅ removal. The modifications have effectively maximized the amount of aeration possible for the existing configuration.

Table 3: Orbal Design Criteria

Parameter	Units	Value	
Solids retention time (SRT)	day	5 – 8	
RAS flow rate	%	40-100% (of flow to basin)	
Volume of basin	gal	2,100,000	
DO concentration	mg/L	0.9 (outer ring) – 3.0 (inner ring)	
Parameter	Units	Average	Maximum Day
Influent BOD ₅ from MBBRs	mg/L	400	660-960
Max BOD ₅ Removal Rate	lbs/day	15,500	17,900
Target MLSS	mg/L	4,500	5,500

3.3.9 120ft Secondary Clarifier

The aeration basin effluent is discharged to the secondary clarifier for removal of biological solids. The secondary clarifier is a 120-ft diameter circular concrete tank with a side water depth of 16 feet. The secondary clarifier was constructed in 2000, replacing the 100-ft diameter clarifier that had previously been used in the secondary process. The mixed liquor from the aeration basin enters the tank through the center, with clarified effluent overflowing the peripheral weir. Settled solids are removed with the rotating mechanisms, using hydraulic head differential to pull sludge through full-radius suction hoods rotating with the mechanisms around the floor of the tank. The rotating mechanisms rotate at approximately 0.3 rpm. The NCCW is also discharged to the launder ring of the secondary clarifier.

The secondary clarifier was designed with an increased vertical drop over the weir to elevate the dissolved oxygen concentration in the effluent and reduce the impact of IEP effluent on the river dissolved oxygen concentrations.

The limiting characteristic for most secondary clarifiers is the solids loading rate, defined as the mass of solids entering the clarifier per surface area per day. IEP has experienced difficulty in the past with sludge settleability. The clarifier effluent flow rate is the flow entering the clarifier minus the underflow (the RAS and WAS (Waste Activated Sludge)). Operationally, this is the same flow as the MBBRs and aeration basin influent.

Table 4: Design Criteria and Capacity for Secondary Clarifier

Parameter	Units	Value	
Diameter	ft	120	
Planar surface area	ft ²	11,300	
Parameter	Units	Average	Maximum Day
TSS from clarifier	mg/L	4,500	5,500
Overflow rate	gal/ft ² /d	400	600
Solids loading rate (SLR)	lb/ft ² /day	12	16
Max clarifier effluent flow rate	MGD	3.6	4.0

3.3.10 Dissolved Air Flotation (DAF)

Dissolved air flotation (DAF) is a water treatment process that clarifies wastewaters by the removal of suspended matter such as oil or solids. The removal is achieved by dissolving air in the water or wastewater under pressure and then releasing the air at atmospheric pressure in a flotation tank basin. The released air forms tiny bubbles which adhere to the suspended matter causing the suspended matter to float to the surface of the water where it may then be removed by a skimming device

The DAF installed at IEP is a 40' x 18' x 5.2' Permutit DAF clarifier that is utilized to remove suspended solids and ink from the De-ink Plant and #5 TMP Plant wastewater. The combined wastewater is pretreated with poly-aluminum chloride (PAX) in a retention tank to coagulate the ink. From the retention tank, the wastewater is pumped into the DAF aeration tank to become saturated with dissolved air. Between four to five SCFM of plant air at 60 psig is metered into

the pressurized aeration tank. From the aeration tank, the air-saturated liquid flows through the distribution header of the clarifier. An anionic polymer is added at the clarifier inlet to flocculate the suspended particles in the liquid. When the aerated liquid is released through the distribution header to atmospheric pressure, micro-bubbles form. These bubbles attach themselves to the flocculated particles in the liquid and then float to the surface. The float is moved along the surface, delivered up a ramp, and discharged into the solids compartment by a continuously operating flight of scrapers.

Recovered solids are removed from the solids compartment by a pump and sent to the Andritz gravity table for further thickening before being fed into the Andritz screw press for final dewatering.

Clarified effluent from the DAF is uniformly withdrawn at the far end of the flotation compartment. The clarified effluent is sent to the heat exchangers for temperature reduction prior to being sent to the mill's wastewater treatment system.

3.3.11 Heat Exchangers

To produce pulp IEP utilizes a thermo-mechanical refiner system. The resulting wastewater from this process is often in excess of 150°F (65°C). Wastewaters at this temperature can cause difficulties in the activated sludge system used to treat the water. To mitigate these issues IEP utilizes two plate-type heat exchangers to cool effluent from the #5 TMP system and float cell rejects prior to discharge to the WWTS.

Approximately 600 to 800 gpm of 150±20°F effluent from the DAF is processed through the heat exchangers for reduction to approximately 85 to 90°F prior to discharge to the primary clarifier. Approximately 1,000 to 1,700 gpm of 55°F non-contact cooling water (NCCW) is used on the cold side of the heat exchangers. NCCW from the heat exchangers is discharged to IEP's secondary clarifier launder ring.

3.3.12 Effluent Metering and Clear Well

A Parshall flume measures effluent from the final clarifier to a clear well using a redundant level transmitter system. Flow information is recorded on a strip chart as well as within the DCS system.

The clear well consists of a concrete vault that serves as a stilling chamber for the outfall prior to introduction to the Spokane River. From the clear well an automatic composite sampler collects final effluent for analytical testing as required per NPDES permit no. WA-000082-5.

3.3.13 Outfall

Treated wastewater is discharged to the Spokane River at river mile 82.6. The outfall pipe consists of a 24-inch line extending from the clear well to the Spokane River. At the river, the outfall decreases in diameter to 18 inches. The 18-inch outfall line extends approximately half way across the Spokane River and has multiple outlet ports for dispersion of the treated effluent into the Spokane River. The outfall was modified slightly in 2001 to eliminate restrictions caused by holdup from Spokane's Upriver Dam (a few miles downstream of IEP). The modification, along with the installation of new effluent metering and a manhole, resulted in an estimated capacity of 11.4 MGD.

3.3.14 Sludge Dewatering

Sludge from the primary clarifier is pumped to the sludge mix tank. This sludge is then combined with waste activated sludge from the secondary clarifier prior to thickening. The sludge is thickened and dewatered on one of two systems: the Andritz gravity table and screw press, or the FKC rotary drum thickener and screw press. A maximum of 50 dtpd (limitation specified by the Air Operating Permit) of dewatered sludge is conveyed to a fluidized bed combustor (FBC) for energy recovery in the form of steam. Any dewatered sludge generated above 50 dtpd is sent out for composting or to landfill. Ash generated from the FBC is utilized as a cement admixture, as a mineral enhancement to compost, or hauled wetted to landfill.

3.3.15 75ft Clarifier (Surge Tank)

A 75' diameter x 14' side water depth clarifier is utilized for flow surge control of the mill's wastewater treatment system. Excess wastewater flow from the mill due to process changes or abnormal conditions is sent to the 75ft clarifier. After the flow surge has diminished, the volume in the 75-ft clarifier is slowly returned to the wastewater treatment system via the mill's sewer system.

4. OPERATIONAL OBJECTIVES AND PARAMETERS

4.1 Operating Parameters: §173-240-150(2)(e)

The operation of the wastewater treatment system is controlled and adjusted by utilizing the following measured operational parameters.

Table 5: WWTS Operating Parameters

Parameter	Method	Location(s)	Frequency	Typical Range
Sludge Volume Index (SVI)	Standard Methods 2710-D	Inner Orbal	Daily	120 – 220
Dissolved Oxygen (DO)	Standard Methods 4500-OG	1° Clarifier	Daily	1 – 15 mg/L
		MBBR Discharges	3 per week	1 – 3 mg/L
		Inner Orbal	Continuous	1 – 3 mg/L
Total Suspended Solids (TSS)	Standard Methods 2540D	Final Effluent	3 per week	6 – 8 mg/L
		1° Clarifier inlet	5 per week	800 – 5000 mg/L
		1° Clarifier outlet	5 per week	50– 400 mg/L
		DAF effluent	2 per week	90 – 600 mg/L
RAS			5 per week	8000 – 14000 mg/L
		Final Effluent	7 per week	2 – 25 mg/L
Mixed Liquor Concentration (MLSS)	Standard Methods 2540D	Orbal	Daily	4000 - 7000 mg/L
Oxygen Consumption Rate (OUR/SOUR)	Standard Methods 2710-B	Inner Orbal	2-3 per week	20 – 40 mg/L/hr
		Outer Orbal	2-3 per week	50-100 mg/L/hr
pH	Electrode	MBBR discharges	3 per week	6.5 – 8.0
		Orbal	3 per week	6.5 – 8.0
		Final effluent	Continuous	6.5 – 8.0
BOD ₅	Standard Methods 5210-B	1° Clarifier in/out	5 per week	800 – 2500 mg/L
		MBBR discharges	3 per week	300 – 500 mg/L
		DAF effluent	2 per week	1000 – 1600 mg/L
		Orbal effluent	3 per week	5 – 15 mg/L
		Final effluent	5 per week	5 – 15 mg/L
Total Phosphorus (TP)	Standard Methods 4500-PE	MBBR inlet	3 per week	0.5 – 3 mg/L
		MBBR discharges	3 per week	0.01 – 0.5 mg/L
		Orbal	5 per week	0.01 – 0.15 mg/L
		Final effluent	2 per week	0.05 – 0.15 mg/L
Total Kjeldahl Nitrogen (TKN)	Standard Methods 4500-N _{org}	MBBR inlet	3 per week	1-16 mg/L
		MBBR discharges	3 per week	1-16 mg/L
Ammonia (NH ₃)	Standard Methods 4500-NC	MBBR inlet	3 per week	3 – 8 mg/L
		Orbal	5 per week	0.01 – 0.2 mg/L
		Final effluent	At min. 1 per month	0.1 – 0.25 mg/L
Temperature	RTD	1° Clarifier influent	Continuous	85 – 115 °F
		DAF Heat exchanger	Continuous	85 – 100 °F
		Final effluent	Continuous	70 – 85 °F

4.2 Observations and Control §173-240-150(2)(c),(e),(f)

Given the lag time necessary for many laboratory tests general observations of the wastewater treatment plant help to provide an indicator of system help and operational success. During the week every morning the wastewater treatment system is inspected by the Technical Superintendent and/or the Pulp Mill Superintendent.

In addition seven days per week each mill operations supervisor also performs a thorough walkthrough of the system at least once per shift to check for any potential issues, while the Chip handler inspects the clarifiers twice per shift and the ONP handler inspects the sludge press operations twice per shift. Regardless of who is inspecting the WWTS common observations include the following:

- Color of the MLSS in the Orbal
- Foam in the Orbal
- Color and foam of the DAF sludge blanket

- Odor on the mill site
- Clarity of Secondary clarifier
- Clear water depth measurement of secondary clarifier
- Sludge quality and concentration on the gravity table and screw presses

During the inspections any change in observed conditions is communicated to the Technical superintendent, mill operations supervisors, and pulp mill operators so that any changes that are necessary can be made.

4.3 Operational Objectives §173-240-150(2)(e)

4.3.1 Treatment Plant Permit

Inland Empire Paper Company discharges wastewater to the Spokane River under NPDES Permit WA-000082-5. Table 6 and Table 7 show the effluent limitations for the critical season (March to October) and the off-season, respectively.

Table 6: NPDES Permit Limits for Critical Season (March through October)

Parameter	Unit	Average Monthly	Maximum Daily
BOD ₅	lbs/day	1,101	1,555
TSS	lbs/day	4,525	8,450
Total Zinc	µg/L	203	296
Total Lead	µg/L	20.0	29.1
Total Cadmium	µg/L	2.8	4.1
Total Phosphorus	lbs/day	24.7	49.7
pH	S.U.	Daily Minimum ≥ 5.0 and Daily Maximum ≤ 9.0	

Table 7: NPDES Permit Limits for the Off-season (November through February)

Parameter	Unit	Average Monthly	Maximum Daily
BOD ₅	lbs/day	3,530	6,655
TSS	lbs/day	6,392	12,070
Total Zinc	µg/L	203	296
Total Lead	µg/L	20.0	29.1
Total Cadmium	µg/L	2.8	4.1
pH	S.U.	Daily Minimum ≥ 5.0 and Daily Maximum ≤ 9.0	

Total phosphorus listed as a seasonal limit in Table 6 and will be replaced by the DO TMDL waste load allocations (WLAs) at the end of the 10-year DO TMDL implementation phase. Due to the chronic nature of dissolved oxygen impairment and the unprecedented level of nutrient treatment, Ecology has allowed the WQBELs for the parameters regulated by the DO TMDL to be expressed in the same units as the WLAs, that is, as seasonal averages. Table 8 shows the final WQBEL limits in IEP's NPDES permit that must be achieved by November 1, 2021.

Table 8: NPDES Final WQBEL Limits to be Achieved by 2021

Parameter	Unit	Seasonal Average
Final WQBEL for Outfall #001 March through October		
Ammonia	lbs/day	24.29
CBOD ₅	lbs/day	123.2
Final WQBEL for Outfall #001 February through October		
Total Phosphorus	lbs/day	2.39

Note that the critical season for ammonia and CBOD₅ differs from that of total phosphorus. This is divergent from the DO TMDL where all nutrients WLAs were set based on the same critical season of March to October (originally 1.23 lbs/day

Total Phosphorus (TP) for IEP during March through October). This modification was allowed and approved by Ecology following a modelling scenario indicating equivalency with the base DO TMDL scenario.

5. LABORATORY PROCEDURES AND SAMPLING

5.1 General Sample Handling and Preservation Requirements: §173-240-150(2)(h)

This section describes both general and specific methods to be used by field personnel when collecting and handling wastewater samples. On the occasion that field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a wastewater sample, the variant procedure will be documented along with a description of the circumstances requiring its use.

Laboratory standard operating procedures for each testing method are included in Appendix A.

1. All sample collection and preservation procedures will comply with the requirements outlined in 40 CFR §136.3(e), Table II.
2. Wastewater samples will typically be collected either by directly filling the sample container or by using an automatic sampler or other device.
3. During sample collection, if transferring the sample from a collection device, make sure that the device does not come in contact with the sample containers.
4. Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace; all other sample containers must be filled with an allowance for ullage.
5. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid. For all other chemical preservatives, IEP will use the appropriate chemical preservative generally stored in an individual single-use vial. The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not adequately preserved, additional preservative should be added to achieve adequate preservation.
6. All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed either into refrigeration or on ice immediately. All composited samples will be collected into a refrigerated container for the duration of the composite.

5.2 Sample Types: §173-240-150(2)(h)

For WWTS sampling, two types of sampling techniques are used: grab and composite. For these procedures, the NPDES permit specifies the appropriate sample type, while both may be used for routine monitoring of current operational criteria. A complete list of testing types, frequency, and location may be found in section 4.1.

5.2.1 Grab Samples

Grab samples consist of either a single discrete sample or individual samples collected over a period of time not to exceed 15 minutes. The grab sample should be representative of the wastewater conditions at the time of sample collection. The sample volume depends on the type and number of analyses to be performed.

5.2.2 Composite Samples

Composite samples are collected over time, either by continuous sampling or by mixing discrete samples. A composite sample represents the average wastewater characteristics during the compositing period. A time composite sample consists of equal volume discrete sample aliquots collected at constant time intervals into one container.

5.3 Record Keeping: §173-240-150(2)(i)

All laboratory results are recorded first by hand on bench sheets and then transferred to Excel spreadsheets for formal reporting and electronic archiving. IEP's network currently has ten years of historical laboratory data readily available for review. Hand copies are filed and eventually archived for later use; at a minimum all sheets are retained for three years. Additionally, print outs of the effluent system HMI screens are revised on a continual basis in the unlikely event that set points are lost and are stored in the Pulp mill operator's station.

Copies of all laboratory bench sheets can be found in Appendix B.

6. WWTS MAINTENANCE

6.1 General Inspection and Maintenance: §173-240-150(2)(j)

An oiler/inspector is responsible for the preventative maintenance of the wastewater treatment system. All effluent pumps and sludge pumps are checked daily. Aerators are lubricated weekly and checked for tightness monthly. Rake arms are inspected, greased, and oiled weekly. Oxygen system components are inspected and filters are replaced semi-annually. The oiler collects all the waste oil and grease from the preventative maintenance program at the wastewater plant for disposal in the designated waste oil and grease containers.

All major maintenance work required on the effluent treatment plant is coordinated with a scheduled mill shutdown, which normally occurs every four weeks. During these scheduled shutdowns, the wastewater flow is at a minimum, which allows for repair work on the plant without sacrificing wastewater treatment quality.

6.2 Preventative Maintenance Schedule: §173-240-150(2)(j)

Part	Action	Interval
Speece Cone O ₂ Generator	Change air filters	3 months
	Change oil	6 months
	Change Compressor Seals	Annually
Effluent Pumps (all)	Grease & Inspect	2 weeks
Clarifier Weirs	Clean and remove slime	1/month during summer
Orbal Aerator Disks	Clean and remove slime	1/month during summer
Clarifier Drives	Grease & Inspect	2 weeks
Flex Rake	Grease& Inspect	2 weeks
Sludge presses	Grease& Inspect	2 weeks
pH probes	Calibration	4 weeks
Effluent flow meters	Calibration	4 weeks
Temperature probes	Calibration	4 weeks
Dissolve Oxygen Probes	Calibration	4 weeks

6.3 Spare Parts: §173-240-150(2)(l)

On-site availability of spare equipment is handled on a case by case basis. The majority of pumps have existing spares, and spare motors, located on-site. Fortunately, the small range of pump models utilized in the WWTS are also used in other applications within the mill. This results in an increased availability of on-site replacements and offering a wide-range of flexibility for scheduled and emergency equipment replacement.

There are however, several unique units used within the WWTS; spares for these units are kept on-site at all times e.g. Orbal aerator discs drive shafts and motors. Additionally other high frequency maintenance items including blowers and aerators have at least two rebuilt spares kept on-site.

6.4 Local Suppliers: §173-240-150(2)(i)

The list below provides vendor names and contact information which supply and service a broad spectrum of equipment in the WWTS.

Part	Vendor	Phone Number
Gould's Pumps	Pumpstech Inc.	509-766-6330
ITT Pumps	Beckwith and Kuffel	800-767-6700
Gardner Denver Blowers	Beckwith and Kuffel	800-767-6700
Orbal Motors and Aerators	Dykman Electrical	509-536-8787
	Kaman Industrial	509-535-1611
Variable Frequency Drives	Dykman Electrical	509-536-8787

6.5 Equipment List: §173-240-150(2)(j)

Equipment Number	Description	Equipment Number	Description
301-002	Screen – Duperon Flex Rake Screen	301-165	Agitator - Sludge Mix Tank
301-003	Pump - Shower Water, Flex Rake Screen	301-170	Pump - East, Sludge Mix Tank
301-005	Conveyor - Flex Rake Screen	301-172	Pump - Polymer to Day Tank Makedown
301-010	Pump - No.3 Diesel, Pump House Effluent	301-173	Pump - West, Sludge Mix Tank
301-011	Pump - No.4 110 kV, Pump House Effluent	301-174	Pump - Polymer to FKC Press
301-012	Pump - No.5 63 kV, Pump House Effluent	301-175	Tank - Conditioning Tank, FKC Press
301-013	Speece Cone Booster Pump	301-180	Mixer - Conditioning Tank
301-014	Speece Cone	301-185	Thickener - Rotary Sludge Thickener, FKC Press
301-015	O ₂ Generator	301-190	Press - FKC Press
301-025	Clarifier - Surge Control, 75' Diameter	301-191	Pump - FKC Pressate to Orbal
301-030	Drive - Rake Arm, 75' Clarifier	301-195	Conveyor - No.1 Sludge Discharge, FKC Press
301-040	Pump - Sludge, 75' Clarifier	301-200	Conveyor - No.2 Sludge to Ground, FKC Press
301-045	Tank - Ammonium Phosphate	301-205	Conveyor - No.3 Sludge to Barn, FKC Press
301-050	Pump - Ammonium Phosphate	301-250	Tank - No.1 MBBR
301-XXX	Tank - Phosphoric Acid	301-251	Screen - Barrier Screen Centrisorter, No.1 MBBR
301-XXX	Pump, #1 Metering - Phosphoric Acid	301-252	Blower - Aerator, No.1 MBBR
301-XXX	Pump, #2 Metering - Phosphoric Acid	301-253	Pump - No.1 MBBR, Primary Effluent
301-XXX	Tank - Urea	301-254	Pump - Foam Abatement System, No.1 MBBR
301-XXX	Pump, #1 Metering - Urea	301-302	Tank - No.2 MBBR
301-XXX	Pump, #2 Metering - Urea	301-303	Tank - No.3 MBBR
301-065	Basin - Orbal	301-306	Screen - Barrier Screen, No.2/3 MBBR
301-070	Aerator No.1 - Orbal	301-307	Pump - Foam Abatement System, No.2/3 MBBR
301-075	Aerator No.2 - Orbal	301-311	Blower No.1, No.2/3 MBBR
301-080	Aerator No.3 - Orbal	301-312	Blower No.2, No.2/3 MBBR
301-085	Aerator No.4 - Orbal	301-313	Blower No.3, No.2/3 MBBR
301-090	Weir Adjustable Level - Orbal	301-314	Blower No. 4, No.2/3 MBBR
301-095	Weir Manual Level - Orbal	301-316	Sump Pump - No.2/3 MBBR
301-100	Aerator No.5 - Orbal	291-240	Tank - Alum Retention Tank
301-105	Aerator No.6 - Orbal	291-245	Pump - Alum Retention Tank to DAF
301-110	Aerator No.7 - Orbal	291-250	Tank - Air Mix Tank, DAF
301-115	Aerator No.8 - Orbal	291-255	Clarifier - Dissolved Air Flotation Clarifier
301-120	Aerator No.9 - Orbal	291-256	Pump - DAF Sludge (East)
301-125	Aerator No.10 - Orbal	291-257	Pump - DAF Sludge (West)
301-130	Clarifier - Primary 100' Diameter	291-260	Pump - DAF Clearwater to Wash Water Tank
301-135	Drive - No.1 Rake Arm, 100' Clarifier	291-264	Makedown Skid - DAF Dry Polymer
301-136	Drive - No.2 Rake Arm, 100' Clarifier	291-266	Agitator - Polymer Mix Tank, Makedown Skid
301-141	Pump - No.1 Sludge, 100' Clarifier	291-267	Pump - Polymer to DAF (East)
301-142	Pump - No.1 Diesel, Primary Effluent	291-268	Pump - Polymer to FKC Press (West)
301-143	Pump - No.2 Electric, Primary Effluent	291-269	Mixer - Venturi Mixer, Gravity Table
301-144	Pump - No.2 Sludge, 100' Clarifier	291-270	Filter Press - Gravity Table
301-146	Pump - Primary Effluent Reclaim	291-273	Makedown Skid - Sludge Press Dry Polymer
301-147	Barrier Screen Centrisorter, Reclaimed Effluent	291-274	Pump - Polymer to Andritz Sludge Press
301-148	Filter - Conustrenner, Reclaimed Effluent	291-275	Press - Andritz Press
301-152	Agitator - Reclaimed Effluent Chest	291-276	Tank - Andritz Pressate Collection Tank
301-153	Pump - Mill Return, Reclaimed Effluent	291-277	Pump - Andritz Pressate to Orbal
301-155	Tank - Clearwell, Primary Effluent	291-280	Conveyor - Andritz Press Sludge Discharge
301-156	Clarifier - Secondary 120' Diameter	291-285	Conveyor - Andritz Press Sludge Screw Lift
301-157	Pump - Return Activated Sludge, 120' Clarifier	291-287	Conveyor - Andritz Press Sludge Reverse Screw
301-158	Flume - Parshall, 120' Clarifier	291-306	Tank - Poly Aluminum Chloride (PAX)
301-159	Tank - Clarified Secondary Effluent Tank	291-307	Pump - No.1 PAX Pump
301-160	Tank - Sludge Mix Tank	291-308	Pump - No.2 PAX Pump

7. EMERGENCY OPERATING PROCEDURE

7.1 Power Outage: §173-240-150(2)(m)

Overall treatment plant bypass cannot occur. There is no wet well overflow to the river. Two effluent pumps are supplied by separate power feed to minimize the impact of electrical outages. In case of a total power outage, the diesel pump would continue controlling the effluent sump level. A total power outage would also cause the water supply and the process facilities, including all pumps, to fail, which would shut off the wastewater flow. Should there be a pump house failure during normal operations, the mill would be shut down to prevent any flooding from the mill sewer trenches or the pump house wet well.

7.2 Primary Clarifier Failure: §173-240-150(2)(m)

In the event of a major breakdown of the 100' primary clarifier, valves are in place that can divert raw mill effluent to the 75' clarifier. Approximately 2,000 gpm of effluent from the 75' clarifier can be diverted back to the primary clarifier clear well via the Conustrenner piping system. This would allow full secondary treatment to continue through the MBBRs, Orbal, and Secondary Clarifier. Any excess flow not diverted to the clear well from the 75' clarifier would be discharged directly (via overflow) to the Orbal Aeration Basin for treatment. Mill operations would be modified as necessary to assure compliance with all operating permits while the 100' primary clarifier is out of service.

7.3 Extended Down: §173-240-150(2)(g)

During extended downtime of the mill, i.e., more than two days, starch is added to the outer Orbal channel and directly to the MBBR tanks. An addition rate of 80 pounds of starch every four hours is sufficient to maintain biological growth.

8. SAFETY: §173-240-150(2)(k)

All safety requirements for the Mill site are addressed in IEP's Accident Prevention Plan.

Appendix A

Laboratory Standard Operating Procedures

pH Measurement (SM 4500-H+ B-11)

The acidity or alkalinity of a solution is measured on a meter in terms of pH units. A value of 7.0 indicates a neutral solution. Values above 7.0 indicate higher alkalinity, whereas values below 7.0 indicate higher acidity.

The manufacturer's instructions are followed closely for the pH meter use and storage and preparation of the electrode. IEP uses a combination electrode with a built-in thermistor for automatic temperature compensation. The electrode is kept wet by returning it to the storage solution whenever the pH meter is not in use.

To calibrate the electrode before use, remove it from the storage solution, rinse, blot dry with a soft tissue and place in a pH 7.0 buffer solution, and set the isopotential point (adjust to pH 7.0). Remove the electrode from the first buffer, rinse thoroughly, blot dry and immerse into a pH 10.0 buffer solution. Adjust the slope dial so that the meter indicates the pH value of the 10.0 solution. The electrode slope is checked daily by performing this buffer calibration and the slope should be 92 to 102 percent. The buffers of pH 7.0 and 10.0 are used for calibration because IEP's effluent ranges between a pH of 7.0 and 8.5. These standards meet the requirements of being with ± 2 pH units of the sample pH, being 3 pH units apart, and they are not $> \text{pH } 10$.

When reading the pH sample, establish equilibrium between electrodes and the sample by stirring the sample to ensure homogeneity. The limit of accuracy under normal conditions is ± 0.1 pH units; therefore, values are reported to the nearest 0.1 pH unit.

Total Suspended Solids (SM 2540 D-11)

Total suspended solids are determined by filtering a well-mixed sample through a weighed glass fiber filter and weighing the residue after being dried at 103-105 °C. The increase of filter weight represents the total suspended solids.

IEP uses a CoorsTek Buchner Funnel attached to a water aspirator for the filtering apparatus. A Whatman 934 AH filtering disk with wrinkled-side up is set into the funnel. Vacuum is applied and the disk is washed with three (3) consecutive 20 ml portions of distilled water. The crucible is then dried in an oven 103-105 °C for one hour, cooled in a desiccator to room temperature and reweighed. The cycle of drying, desiccating and reweighing is repeated until a constant weight is obtained, or the weight fluctuates less than 0.5 mg between successive weighings.

A sample of 500-1,000 ml of effluent is filtered through the glass fiber disk and washed three (3) times consecutively with 10 ml portions of distilled water. The desiccating and weighing pattern as described above is repeated until a constant weight is obtained, the weight fluctuates less than 0.5 mg or the weight loss is less than four (4) percent. The increase in weight of the crucible represents the total suspended solids.

Calculation:

$$\text{Total Suspended Solids (mg/l)} = \frac{(A-B) \times 100 \text{ (g)}}{\text{Sample Volume (ml)}}$$

A = weight of filter and dried residue

B = weight of filter

Biochemical Oxygen Demand (SM 5210 B-11)

The biochemical oxygen demand (BOD) test measures the relative amount of oxygen required by a waste stream. It specifically measures the oxygen utilized during a specified incubation period for the biochemical degradation of organic material. The BOD measurement is significant in water quality management because it is used to determine the approximate quantity of oxygen that will be required to biologically stabilize the organic matter present. The higher the BOD loading on an ecosystem, the more negative the impact.

Procedure:

Sample Collection

The final effluent sample from the IEP wastewater treatment system is collected using a refrigerated automatic sample. The temperature is maintained at 4 °C.

Sample Storage

The samples are to be stored in the lab refrigerator at 4 °C for a period not to exceed 48 hours. Storage temperature and duration must be recorded. Samples are warmed to 20 °C before analysis.

Glassware Preparation

BOD glassware is washed in a high temperature laboratory dishwasher and allowed to dry before being stored for use. Tubing on the BOD water supply is to be replaced and the dilution water vessel is to be acid washed and rinsed with tap water followed with a triple rinse with distilled water.

Incubator

Incubation occurs in the dark with temperature maintained at 20 ± 1 °C. The temperature is monitored via digital display on the exterior of the incubator.

Dilution Water Preparation

Dilution water is to be kept in a state of constant saturation by the addition of air to the vessel. HACH BOD Nutrient Buffer pillows are added for the preparation of 18 liters of dilution water per BOD set up.

Dilution Ratio

Proper sample dilutions have been learned over a considerable period of time. For the most accurate results, the proper dilution will have a 5-day DO depletion of at least 2 mg/l and a residual DO of not less than 1.0 mg/l. If proper dilution is in question, then is ideal to use several dilutions to determine the proper range.

BOD Set-up

Using a pipet or graduated cylinder, sample volumes are dispensed into a minimum of three (3) BOD bottles. The prepared dilution water is then syphoned to the BOD bottles. Enough dilution water is added to the BOD bottle to make a water seal.

Two bottles are filled with dilution water and BOD nutrient as blank checks for dilution water quality and glassware cleanliness.

DO Measurements

After calibrating the meter, the DO reading is recorded for one bottle of each sample. The bottles are placed in the incubator at 20 °C. After five days, the oxygen meter is calibrated and the DO reading is recorded for each bottle. The final DO should not drop below 1.0 mg/l.

BOD Calculations

BOD (mg/l or ppm) is calculated by subtracting the five day DO residual from the initial DO for each bottle, adjusting for the seed oxygen uptake and multiplying by the dilution factor. The dilution factor is determined by dividing the volume of the sample used into 300 ml (the volume of the BOD bottle).

$$\text{BOD}_5 \text{ (mg/l)} = \frac{(\text{D}_1 - \text{D}_2) - (\text{S})\text{V}_s}{\text{P}}$$

D_1 = DO of diluted sample immediately after preparation (mg/L)

D_2 = DO of diluted sample after 5 d incubation at 20°C (mg/L)

S = oxygen update of seed, $\Delta\text{DO}/\text{mL}$ seed suspension added per bottle. S = 0 if samples are not seeded.

V_s = volume of seed in the respective test bottle (mL)

P = decimal volumetric fraction of sample used; $1/\text{P}$ = dilution factor

Quality Control

A check standard solution of glucose/glutamic acid is run with each batch of samples. The standard check solution is made by combining 150 mg glucose and 150 mg of glutamic acid in one liter of distilled water. A 2% solution of this mixture is set up using the above BOD procedure.

Total Phosphorous (SM 4500-P E-11)

Phosphorous is essential to the growth of algae and other biological organisms. Because of the algae blooms that may occur in surface waters, there is a concerted effort to control the amount of phosphorous compounds that enter surface waters through industrial waste discharges. Inland Empire Paper has the ability to utilize two variants of the standard method. The HACH Method 10209/10210 variant utilizes the same digesting and complexing reaction as is used in the standard method, but allows for the process to be more streamlined by using prepackaged kits and a reactor cell.

SM 4500-P E (Standard Method)

Phosphorous analysis requires two general procedural steps:

1. Conversion of the organic phosphorous forms to dissolved ortho-phosphate
2. Colorimetric determination of dissolved orthophosphate

Phosphates present in organic and condensed inorganic forms must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the condition for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to ortho-phosphates by heating with acid and persulfate. Ortho-phosphate reacts with molybdate in an acid medium to produce a phospho-molybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

IEP uses the HACH powder pillows and Spectrophotometer handbook procedures for the Acid Persulfate Digestion Method and the Ascorbic Acid Method to determine the total phosphorous.

Procedure:

Samples must be analyzed immediately

1. Measure 25 ml of sample into a 50 ml Erlenmeyer flask
2. Add the contents of one (1) potassium persulfate powder pillow and swirl to mix
3. Add 2.0 ml of 5.25N sulfuric acid
4. Boil to digest for 30 minutes (maintain volume at 20 ml by adding deionized water)
5. Cool sample to room temperature
6. Add 2.0 ml of 5.0N sodium hydroxide
7. Pour contents into a 100 ml graduated cylinder and bring up to 100 ml with distilled water. Invert 20 times to mix well
8. Pour the sample into two (2) 25 ml sample cells

9. To one cell add all the contents of one phosphate pretreatment powder pillow. Use this solution to zero the spectrophotometer at 700 nm
10. To the other cell add the contents of one Phos Ver 3 powder pillow and swirl immediately. Allow to react for a minimum of two (2) minutes and maximum of ten (10) minutes
11. Place the prepared sample cell into the spectrophotometer and measure phosphate (mg/l)
12. Run a reagent blank to get a reading for deionized water interference (0.02 mg/l)
13. Total phosphate (mg/l) can be expressed as total phosphorous (mg/l) by dividing by 3.07

SM 4500-P E (HACH 10209/10210)

HACH method 10209/10210 utilizes prepackaged kits and a reactor to follow SM 4500-P E and produce similar results to the steps outlined by the standard method previously described.

TNTplus™ 843 kits are used along with the spectrophotometer handbook procedure to determine the Total Phosphate concentration.

Procedure:

Samples must be analyzed immediately

1. Turn on the reactor chamber and set the temperature to 100 °C
2. Measure and dispense the appropriate sample volume into the sample vial
 - a. 2.0 ml for the low range analysis
 - b. 3.5 ml for the ultra-low range analysis
3. Remove the foil from the DosiCap and tighten cap, reagent-side down, on the sample vial.
4. Shake the vial 2-3 times to dissolve the reagent. Inspect the DosiCap to verify all the reagent has dissolved.
5. Insert sample vials into the reaction chamber
6. Allow sixty (60) minutes for digestion to occur
7. Remove the sample vials from the reactor and allow to cool to room temperature
8. Pipet 0.2 ml of Solution B (16% Sulfuric Acid) into the sample vial
9. Apply DosiCap C (Sodium Metaborate) to the sample vial
10. Invert the vial 2-3 times.
11. Allow to react for ten (10) minutes
12. Invert the vial an additional 2-3 times.
13. Place the reagent blank in the spectrophotometer and “zero” the instrument
14. Place the prepared sample cell in the spectrophotometer and measure PO₄-P (mg/l)

Ammonia (as N) (SM 4500-NH₃ F-11)

Ammonia (as N) is a nutrient that can promote the growth of algae in surface waters. Additionally, in its free state it can also be toxic to aquatic life. Efforts are increasing to control the amount of ammonia released by industrial discharges to protect the environment from both the possibility of excessive algae growth and the possibility of aquatic toxicity.

SM 4500-NH₃ F (HACH Method 10205)

SM 4500-NH₃ F was implemented to prevent the generation of hazardous waste during completion of USGS I-3520-85, which was the previously accredited method.

IEP uses the Hach Company Ammonia Method 10205, which utilizes salicylate and is equivalent to the phenate method. TNTplus™ 830/831 kits are used along with the spectrophotometer handbook procedure to determine the Ammonia (as N) concentration.

Procedure:

1. Measure and dispense the appropriate sample volume into the sample vial
 - a. 5.0 ml for the ultra-low range TNTplus™ 830 kit
 - b. 0.5 ml for the low range TNTplus™ 831 kit
 2. Remove the foil from the DosiCap and tighten cap, reagent-side down, on the sample vial.
 3. Shake the vial 2-3 times to dissolve the reagent. Inspect the DosiCap to verify all the reagent has dissolved.
 4. Allow the mixture to react for fifteen (15) minutes.
 5. Invert the vial an additional 2-3 times.
 6. Place the reagent blank in the spectrophotometer and “zero” the instrument
 - a. The reagent blank is prepared by replacing the sample volume with deionized water.
 7. Place the prepared sample cell in the spectrophotometer and measure NH₃-N (mg/l)
-

Total Kjeldahl Nitrogen (SM 4500-N_{org})

For enhanced process control, IEP uses TKN testing to evaluate MBBR nitrogen uptake.

SM 4500-N_{org} (HACH Method 10242)

Total Kjeldahl nitrogen is the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate (NH₄)₂SO₄ and subsequently analyzed colorimetrically using salicylate or phenate chemistry with nitroprusside. Organic Kjeldahl nitrogen is the difference obtained by subtracting the free-ammonia value from the total Kjeldahl nitrogen value. Total nitrogen (TN) is the sum of TKN (free-ammonia and organic nitrogen) plus nitrate and nitrite.

$$\begin{aligned} [\text{TKN}] &= [(\text{NH}_3 + \text{NH}_4^+)] + [\text{Nitrogen}_{\text{org}}] \\ [\text{Nitrogen}_{\text{org}}] &= [\text{TKN}] - [(\text{NH}_3 + \text{NH}_4^+)] \quad [\text{TN}] = [\text{TKN}] + [\text{NO}_3^-] + [\text{NO}_2^-] \end{aligned}$$

Hach Method 10242, also known as TNTplus 880 is a simplified green chemistry alternative to other methods approved at 40 CFR 136 for the purposes of regulatory reporting of TKN. In the simplified total Kjeldahl (s-TKN) method, inorganic (NH₃, NH₄⁺, NO₂⁻) and organic nitrogen are oxidized to nitrate by digestion with peroxodisulfate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulfuric and phosphoric acid to form a nitrophenol. Oxidized forms of nitrogen in the original sample (nitrite + nitrate) are determined in a second test vial and then subtracted, resulting in TKN.

$$[\text{TKN}] = [\text{TN}] - [\text{NO}_3^- + \text{NO}_2^-]$$

Procedure:

1. a) Measure and dispense 1.3 mL of sample into 20 mm reaction tube
b) Measure and dispense 1.3 mL of solution A into 20 mm reaction tube
c) Add 1 tablet from reagent B bottle into reaction tube and cap tube
2. Heat 1 hour at 100 degC in the reactor
3. Cool down reaction tube to room temp
4. Add 1 Microcap from reagent C bottle into reaction tube
5. Cap reaction tube and invert 2-3 times to dissolve Microcap contents
6. Transfer 0.5 mL of digested sample from reaction tube into vial 1 (red label)
7. Transfer 0.2 mL of reagent D into vial 1
8. Immediately cap and invert 2-3 times
9. Transfer 1.0 mL of undigested sample into vial 2 (green label)
10. Transfer 0.2 mL of reagent D into vial 2
11. Immediately cap and invert 2-3 times
12. Allow the mixture to react for fifteen (15) minutes.
13. Place vial 1 in the spectrophotometer to read until the display reads "E1"
14. Place vial 2 in the spectrophotometer to read and record results

Appendix B

Laboratory Bench Sheets