



## ENVIRONMENTAL FOCUS

### Meet new NPDES permit limits

**For most U.S. plants, treatment modifications will be required for additional removal of TSS, heavy metals, organics and aquatic toxicity.**

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Many refiners will need improved technology to meet new permit limits of the National Pollutant Discharge Elimination System (NPDES). Ashland Petroleum's 68,000-bpd Canton refinery applied such technology in reconfiguring its wastewater treatment plant (WWTP). A plant description and other information on factors leading up to the Canton changes are outlined below.

**Fresh approaches.** Alternative approaches were developed for each type of pollutant. Treatment options considered for ammonia-nitrogen were improved raw waste collection and in-plant source control at the steam stripper or biological nitrification and de-nitrification in the activated sludge system. Aquatic toxicity analysis indicated that heavy metals and adsorbable organics contributed to the toxicity observed. Therefore, activated carbon treatment was evaluated to provide organic polishing and effective removal of toxic organic constituents. Carbon treatment was evaluated by end-of-pipe carbon column adsorbers, which would be preceded by granular media filtration for suspended solids removal and the activated sludge process with additions of powdered activated carbon (PAC) to the aeration basin. PAC addition provides for adsorption of biofactory organics. It is also used to adsorb inhibitory organics, thereby allowing survival of nitrifying bacteria. Powdered carbon also increases the settling velocity and thickening characteristics of the mixed liquor suspended solids. This process can achieve lower effluent TSS levels required for TSS compliance.

**Heavy metals removal** was examined because a positive means for removal of all metals of concern was needed. Several options were developed. The base-case one involved a new metal-hydroxide precipitation step and primary clarifier following the existing DAF (dissolved air flotation) units. This process would be supplemented with iron and/or sulfide additions to improve performance. It would also be supplemented with a coagulant (iron, alum or sulfide) addition system prior to the secondary clarifiers. If necessary, this would be followed by final filters.

Options included chemical addition ahead of the existing DAF units, with reliance on flotation of the precipitated solids rather than settling (for the base case). Final polishing was thought to be a more likely requirement for these cases than for the base case. Option 1 involves a two-stage sulfide and iron addition, co-precipitation and removal system. The first stage would involve a new precipitation step after the API unit with floc removal in the existing DAF units.

The second stage would involve a new precipitation and flocculation system prior to the activated sludge clarifiers, followed by final filters, if necessary. Option 2 was a new two-stage system with only sulfide addition in each step. Treatability studies were then performed to obtain performance data on the various options.

### The Canton plant . . .

Ashland Petroleum's Canton plant produces gasoline, diesel fuel, jet fuel, heating oil and asphalt. The wastewater treatment plant (WWTP) consists of API separation, dissolved air flotation (DAF), equalization, activated sludge aeration and clarification.

Typical wastewater and final effluent characteristics for the WWTP are in the table below.

As can be seen, raw waste levels are similar to those of other refinery wastewaters, with the possible exception of the O&G level after flotation treatment, which is somewhat high. This was found to be due to mechanical problems in the DAF units. These were corrected. This resulted in improved DAF performance and DAF effluent O&G level of about 30 to 50 mg/L. The plant produces a good quality effluent. Aeration Basin Food/Microorganism (F/M) Ratio averages about 0.35 lb COD/day-lb MLVSS, a value at which good biological treatment would be expected. The process design for the upgraded WWTP was developed for an average COD loading of 4,800 lb/day. This corresponds to a concentration of 575 mg/L at a flow of 1.0 mgd, and a maximum COD loading of 10,000 lb/day.

### Impact of new water quality-based permit limits

Parameters anticipated for inclusion in the new water quality-based final permit are in Table 1. Where applicable, these limits were developed based on receiving stream water quality criteria and appropriate wasteload allocation procedures. Not all parameters will have limits. Provisions for monitoring only of selected parameters are anticipated.

As can be seen, conventional parameters, such as BOD, COD, TSS, O&G and total phenolics will be regulated. Also, new water quality-based limits will be imposed for heavy metals and ammonia-nitrogen. Seasonal ammonia-nitrogen limits were developed. Treatment for this parameter was

judged to be necessary. The capability to remove all of the heavy metals was judged to be of primary concern in the treatment plant upgrade. Both acute and chronic toxicity water quality-based limits will apply to this discharge. These limits will apply to both Daphnia and Fathead Minnows.

Effluent monitoring data collected over the last two years, including aquatic toxicity data with Daphnia and Fathead Minnows, indicated need for aquatic toxicity control. Note that TDS levels were high enough in some of these samples to exhibit or cause some of the chronic toxicity values observed. In other words, had TDS levels been lower, chronic toxicity values could have been lower. However, the periodic presence of acute toxicity and high levels of chronic toxicity indicated the presence of toxic agents other than salt. Initial fractionation test work indicated that adsorbable organics and heavy metals were the other major toxic constituents.

Equalization tank effluent (mg/L)* Effluent (mg/L)				
Parameter	Range	Typical	Range	Typical
BOD	200-600	350	5-100	15
COD	400-1,200	600	50-150	100
TSS	50-250	150	50-150	75
O&G	75-200	150	2-10	5
NH <sub>3</sub> -N	<1-20	10	0.2-5	1-2
Tot.phenolics	2-50	5-15	0.01-10	0.2-0.5
Sulfide	0.1-1.5	1.0	<0.01	<0.01

**TREATABILITY STUDIES**

These studies employed samples of wastewater from the equalization tank and API effluent at the Canton plant. Grab samples were collected weekly in 55-gal drums, transported to Nashville within 8 hrs of collection, stored at 4°C and used for testing over the next week.

**Activated sludge test work.** Activated sludge treatability test work evaluated possible modifications to the existing system. Goal: improve removal of specific constituents and aquatic toxicity and evaluate biological nitrification. Four test units (Units A through D) were operated over a 10-week period using equalization tank effluent as feed. The continuous-flow treatability systems are in Fig. 1

**TABLE 1 - Summary of regulated parameters**

Parameters	
BOD	<b>Metals*</b> Antimony Cadmium Chromium, hexavalent Chromium Copper Lead Mercury Nickel Selenium Silver Thallium Zinc
COD	
TSS	
O&G	
NH <sub>3</sub> -N	
** June-September	
** March-May, October-November	
** December-February	
Sulfide	
pH	
total phenolics (4AAP)	

Toxicity  
 Acute toxicity, Daphnia and Fathead Minnows  
 Chronic toxicity, Daphnia and Fathead Minnows

\* Some metals are monitor only.

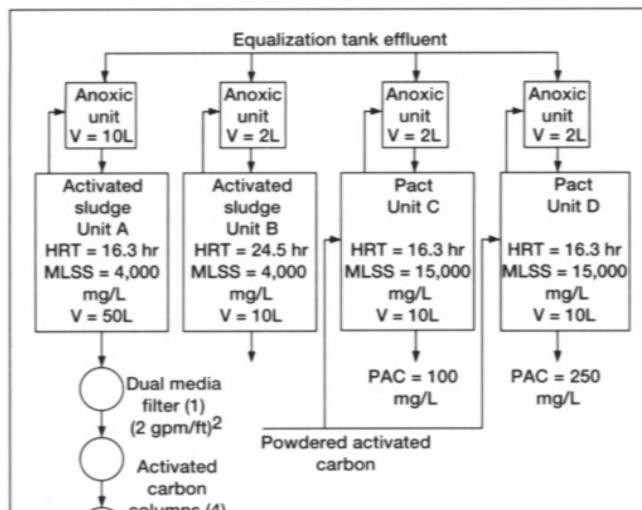




Fig. 1 - The Canton refinery's treatability units.

Unit A was a simulation of the existing activated sludge system modified to include sufficient aeration and phosphorus addition. Unit B had a 50% greater aeration basin hydraulic retention time. It was operated to determine if a third aeration basin in parallel with the existing basins would significantly improve system performance and achieve consistent biological nitrification. Units C and D were operated like Unit A, with PAC additions to these aeration basins. PAC dosages of 100 and 250 mg/L were used to operate Units C and D, respectively. Granular activated carbon (GAC) columns were operated using the Unit A effluent, to determine if granular carbon usage was warranted and which contacting mode (PAC or GAC) was most beneficial. The size of Unit A (50-liter aeration basin) was large enough to generate the effluent flow required to operate the 1-in.-diameter carbon columns. Conversely, 10-liter volumes were used on all other units. A commercial regenerated powdered carbon was used in the powdered carbon activated sludge unit operations.

**Test results** are in Table 2. The units were operated at 20 to 22° C, slightly above the minimum basin temperature expected. Biological treatment using the activated sludge process was very effective. Biological nitrification occurred in all test units, including those without PAC additions. However, nitrifiers are sensitive organisms. They may require additional controls to prevent shock upsets. Also, a new anoxic basin for denitrification would be required. Denitrification using an anoxic reactor would be required if ammonia was removed at the full-scale wastewater treatment plant (WWTP). In-plant ammonia control, with collection and stripping of all high-strength ammonia discharges, would be more reliable and cost-effective, because sour water stripping is presently used. Based on this and other factors, in-plant control of ammonia discharges was selected to achieve effluent permit limits for ammonia.

Test work indicated that PAC significantly reduced the effluent soluble TOC and COD values and increased the sludge settling rates. The system with the higher PAC dosage (Unit D) obtained the lowest soluble effluent TOC and COD levels. A 50% increase in aeration basin retention time (Unit B) did not result in a significant improvement in parameter removals.

Excellent O&G removals were observed in all systems. Excellent removals of total phenolics and sulfide were also observed in all systems. However, data indicate that unit performance was susceptible to high influent levels of these parameters. Therefore, control of sour water discharges and spent caustic containing phenolics was implemented to assure effluent compliance. A PAC dose of 250 mg/L (Unit D) would be used in subsequent carbon treatment evaluations.

**TABLE 2 - Activated sludge treatability data**

Parameter	Unit A Full-scale simulation (D.T.=16 hours)	Unit B (D.T.= 24 hours)	Unit C PACT @ 100 mg/L (D.T.= 16 hours)	Unit D PACT @250 mg/L (D.T. = 16 hours)
Aeration basin				
** MLSS, mg/l	3,540	2,190	5,980	10,160
** MLVSS, mg/l	2,900	1,920	4,990	7,130
** ZSV, ft/hr	8.3	11	23.1	19.3
Effluent (mg/L)				
** BOD <sup>5</sup> - Total	11	15	13	13
***** Soluble	8	10	10	7
** COD <sup>5</sup> - Soluble	91	82	41	23
** TOC <sup>5</sup> - Soluble	29	26	13	7
** Oil & grease	7.5	6.5	3.0	2.0
** TKN <sup>5</sup> - Soluble	3.7	2.4	5.7	2.1
** NH <sub>3</sub> -N -Soluble	3.3	2	2.7	1.6
** Sulfides	<0.1	<0.1	<0.1	<0.1

**Final filtration and carbon column tests.** A pilot-scale dual-media filter and granular carbon column system were operated using the activated sludge effluent. The major objectives of these tests were to evaluate toxicity and specific parameter removals and also to collect process design information. One downflow dual-media filter and four activated carbon columns were tested. Unit A effluent was filtered using a 1.5-in.-diameter downflow dual-media filter at a hydraulic loading rate of 2.0 gpm/sq ft. The filter contained 12 in. of sand (0.50 +/- 0.05 mm effective size and uniformity coefficient of 1.5) and 18 in. of anthracite coal (1.00 +/- 0.05 mm effective size and 1.7 uniformity coefficient).

The carbon columns were 1.0 in. in diameter. Each was packed with 3.5 ft. of a commercial-grade reactivated carbon. A target hydraulic loading rate of 2.0 gpm/sq ft was also used for operating the carbon columns. This provided an empty bed contact time of approximately 15 min/column for a 60-min total contact time. Operation of two, 3.5-ft-deep carbon columns in series provided about the same contact time as one full-scale carbon column that contains 20,000 lb of carbon. Four pilot carbon columns were operated, thus, two full-scale columns (in series) were simulated.

Table 3 summarizes filtration and carbon column data. The dual-media filter reduced suspended solids from an average of 66 mg/L to 6 mg/L without polymer addition. Individual feed values ranged from 4 to 206 mg/L. Effluent values ranged from 1 to 14 mg/L, indicating excellent performance. The filter was backwashed daily (23 to 25-hr run times) with a pressure buildup of about 3 to 5 ft of water. This indicates that run time to 24 hrs or greater may be expected on the average from a full-scale filter at terminal headloss values of about 10 ft of water. The solids removal per sq ft of filter surface area was about 1.5 lb/sq ft.

Batch-activated carbon isotherm tests using pulverized regenerated carbon were conducted several times during the investigation on Unit A effluent. For an average Unit A effluent TOC of 31 mg/L in these tests, a carbon usage rate of 0.32lb TOC/lb carbon was observed. For the average filter effluent TOC of 26.5 mg/L (Table 3) the carbon usage rate would be 0.25 lb TOC/lb carbon. These rates are on the high end of the range for refinery wastewaters. They indicate excellent adsorption characteristics.

The carbon columns were operated for 30.4 days. TOC was monitored daily to observe breakthrough and determine carbon adsorption capacity.

Due to high pressure, carbon columns were backwashed several times during testing. Although no gas bubbles or odors were noted, a buildup of biological solids in the first carbon column was thought to have occurred. This increased headloss in the columns and necessitated backwashing.

**TABLE 3 - Filtration/carbon column treatability data**

Parameter	Biological effluent	Filter effluent	Carbon effluent
TSS	66	6	-
BOD	-	12	4
TOC	43.8	26.5	<5
O&G	-	6.2	<1.0
TKN	-	6.0	2.7
NH <sub>3</sub> -N	-	1.5	1.0
Total phenolics	-	<0.050	<0.050

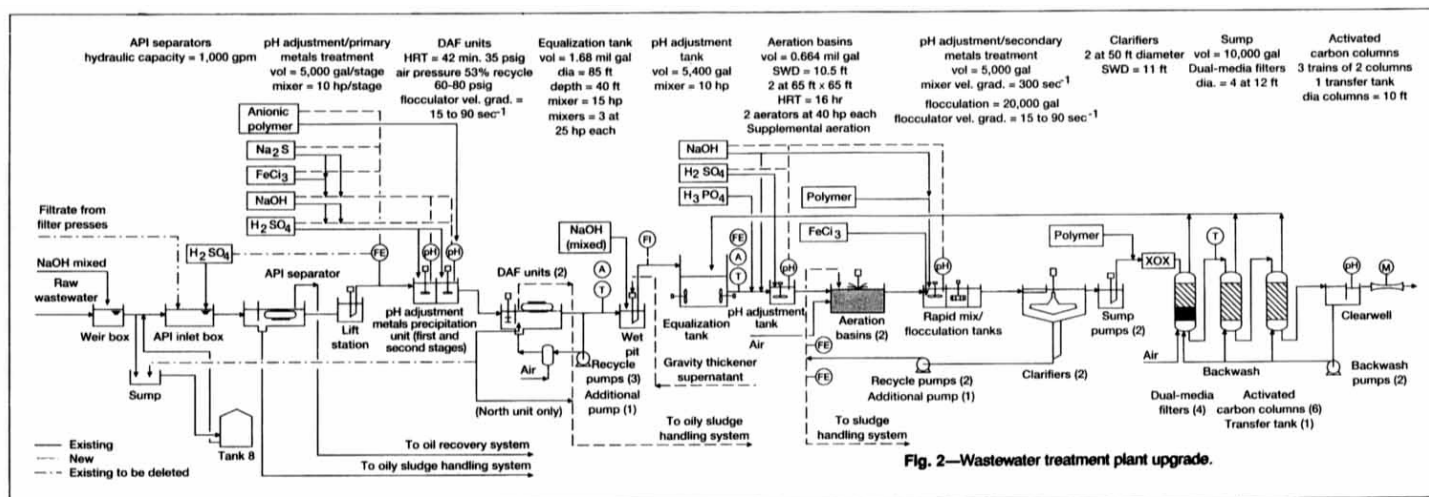
**TOC.** The ratio of the column effluent TOC divided by the feed TOC concentration was analyzed. Breakthrough plots were extrapolated to estimate the carbon usage rate for two full-scale columns operated in-series at a hydraulic loading rate of 2.0 gpm/sq ft. These curves were extrapolated for an effluent TOC/influent TOC ratio of 0.9, corresponding to 90% saturation or exhaustion of the carbon in the column. The 0.9 ratio is used for design when effective use of the carbon is possible. Projected run times for a 90% utilization at 2.0 gpm/sq ft loading rate are as follows:

Column no.	Liters	Days
1	2,100	35.5
2	3,500	59.1

Thus, at these test conditions a run time of about 60 days is projected to achieve 90% saturation of the carbon in the first set of full-scale standard carbon adsorbers. Each has a carbon depth of 7 to 8 ft. Based on the 30-day pilot run, an average carbon usage rate of 0.215 gm/L, or 1,790 lb/day, was projected assuming a 1.0 mgd wastewater flow rate at the feed TOC concentrations observed. The column carbon usage rates were lower than the rates from the batch isotherm tests. But they were considered a better estimate of actual usage rates. Based on removal of 30 mg/L TOC from the filtered activated sludge effluent at a flow of 1.0 mgd (700 gpm) and the pilot carbon usage rates, a full-scale granular-carbon usage rate of about 2,000 lb/day was projected.

**Metals removal test work.** To investigate metals removal, a series of tests involved metals-spiked samples of the API effluent and equalization tank effluent. The API effluent sample was used in batch tests for the first-stage metals removal system using a bench-scale DAF unit. In these tests, the polymer used at the refinery and a 53% recycle rate were used.

To evaluate second-stage metals removal, the equalization tank spiked sample was fed to the continuous-flow activated sludge units shown in Fig. 2. For Units D, E, and H (powdered carbon activated sludge unit), chemical coagulants were fed to the mixed liquor before the external final clarifier. Clarifier effluent samples were analyzed to determine metals removals from the feed. Tests F and G involved separate jar testing of the unit effluent to evaluate a tertiary coagulation and clarification step.



**Fig. 2—Wastewater treatment plant upgrade.**

Test work indicated that co-precipitation with iron and sulfide and removal using the DAF process was the most effective primary metals treatment approach. Chemical doses of 50 mg/L as Fe and 5 mg/L as sulfide at a pH of 7.5 were selected. Excellent removals were obtained, based on anticipated influent levels. However, the expected removals would not achieve final permit limits for cadmium, copper and selenium. Therefore, additional removals would be required in the remainder of the WWTP.

Based on test results, it was recommended that the second stage consist of the addition of iron only as a dose of up to 30 mg/L to the mixed liquor, as required, to meet permit limits. Second-stage treatment will be needed for polishing of cadmium, copper and selenium and possibly for silver, nickel and mercury. Based on these tests, additional full-scale monitoring data for these parameters, and the very stringent permit limits anticipated, it was decided to include final filters in all upgrade options. This was to assure a comfortable margin of safety in meeting effluent metals limits.

In summary, a two-stage type metals coagulation approach and final filters will be used. Primary metals by precipitation with sulfide (5 mg/L as sulfide), ferric chloride (50 mg/L as Fe), and pH adjustment to 7.5 prior to DAF treatment will provide significant metals removals prior to activated sludge treatment. Metals polishing as required, by ferric chloride addition at a dose of up to 30 mg/L as iron just ahead of the activated sludge clarifiers will provide secondary control. Effluent filters for final polishing will also be included.

**Toxicity Reduction tests.** Both acute and chronic aquatic toxicity tests were performed on the effluent from the activated sludge and carbon column test units three times during tests. In each of these tests, toxicity was observed at the dilution that salt toxicity would be exerted based on the TDS level in the samples. Thus, a reduction in salt would be required to achieve consistent compliance with bioassay limits. All biological treatment options investigated could achieve the permit limits as long as dissolved-solids levels were acceptably low. And limits were met as long as the maximum organics removal, achieved by activated carbon treatment, and heavy metals removal capability, were provided in the upgraded treatment system. Salt control stemmed from regulation of cooling tower and boiler operations to avoid excessive dissolved-solids levels in the treatment plant, as required.

**Activated carbon evaluations.** Activated carbon treatment was incorporated into the wastewater management plan for Canton based on available treatability data, other refinery experience that shows improved effluent toxicity levels with carbon treatment and the additional treatment afforded by carbon for total phenolics and other organics. For aquatic toxicity reduction, the basic activated carbon options were 1. a biological system with PAC additions at a carbon dose high enough to achieve effluent polishing and 2. an end-of-pipe granular carbon column adsorption system that would follow the existing activated sludge system. Filters to control effluent heavy metals and suspended solids were included in each option.

**TABLE 4 - Modifications needed for conversion to PACT process**

Item	Comment
1. PAC storage shed and feeder system	1. Needed for PA addition
2. Surface aerator modifications	2. Change in impellers
3. Additional subsurface aeration	3. Higher power level required for mixing
4. Polymer addition system	4. Cationic polymer at 0.5 to 1.0 mg/L
5. Clarifier modifications	5. Conventional clarifier
** a. Replace mechanism	** a. Heavy duty plow-type mechanism required
** b. Increase slope of bottom	** b. Hydraulic take off systems have relatively flat bottoms
** c. Higher walls	** c. Depends on bottom slope
** d. Wastewater pumping	** d. Depends on Item C and minimum SWD
6. Recycle/wastage pumps	6. Replace with rubber lined equipment
7. Gravity thickener mechanism	7. High torque rake mechanism required

**Powdered carbon system requirements.** A PAC carbon dose of 2,100 lb/day (250 mg/L at a 700 gpm flowrate) was selected to achieve anticipated discharge limits. Powdered regenerated service carbon at a delivered purchase cost of \$ 0.60/lb was considered for costing purposes. This dosage was based on the Unit D wastewater treatability results. At these rates, PAC purchase costs would be \$ 450,000/yr. A summary of WWTP modifications needed for PAC additions is in Table 4. These costs include increased aeration basin mixing to maintain the heavier PAC biological solids in suspension and conversion of the existing suction-type clarifiers to conventional scrapper units or possibly to other suction-type units with a lower potential for plugging problems. Note that conversion of the existing clarifiers would require each clarifier to be out of service for two to three months if converted to conventional clarifiers or up to two to three weeks if converted to other suction-type units. Once the first clarifier is converted, however, PAC addition could begin. This would improve operations while the second clarifier is being modified. For economic evaluation purposes, final filters were sized at a hydraulic loading rate of 3 gpm/sq ft at a flow of 1.00 mgd. A conventional high-pressure, dual-media filter system, with anionic polymer addition capability, was costed.

A wet-air regeneration system was considered for regeneration of the powdered activated carbon/waste sludge mixture from this system. However, due to low carbon doses and low sludge disposal costs, this was not economically attractive.

**Granular carbon system requirements.** This system would consist of a filtration and carbon column adsorption system. A granular carbon usage rate of 2,000 lb/day was determined for an end-of pipe carbon column system. Regenerated service carbon at a delivered cost of \$1/lb including transportation and off-site regeneration of spent carbon was considered. At these rates, carbon costs of \$720,000/yr were projected. Capital costs for the carbon system were based on three sets of 10-ft-diameter, 8-ft-deep columns. Each set would include two carbon columns operated in series. A transfer tank was also included.

**TABLE 5 - PACT vs. GAC cost summary**

Item	PAC	GAC
<b>Capital costs (\$)</b>	°	°
Clarifier modifications	** 350,000	*****0
Final filters/pumps	** 600,000	** 600,000
GAC columns	***** 0	** 750,000
Aeration basin modifications	** 200,000	** 200,000
Polymer feed	**** 20,000	*****0
Zimpro license	** 150,000	*****0
PAC storage/addition system	**** 75,000	*****0
Subtotal capital (\$)	1,395,000	1,550,000
Annual capital (\$/yr)	** 226,688	** 251,875
<b>O&amp;M costs (\$/yr)</b>	°	°
Carbon	*** 455,000	*** 720,000
Electrical	**** 95,000	**** 35,000
Maintenance	**** 36,000	**** 78,000
Sludge disposal/polymer	**** 55,000	**** 10,000
Operating labor	** 200,000	** 200,000
Subtotal O&M	*** 841,000	* 1,043,000
Total annual costs (\$/hr)	\$1,067.688	\$1,294.875
Notes:	°	°
** CRF= 0.1625 (10% for 10 yr)		

**TABLE 6 - Treatment process summary**

Treatment	Parameter treated	Upgrade facilities
API separator	O/G+TSS	-
pH adjustment/primary metals precipitation system	pH + heavy metals	Three-stage pH adjustment including FeCl <sub>3</sub> +Na <sub>2</sub> S addition
DAF units	O/G+TSS+heavy metals	Improved polymer addition+effluent turbidity monitoring + increased air dissolution
Equalization tank	Flow+organics+O/G	Improved mixing +H <sub>3</sub> PO <sub>4</sub> addition + effluent turbidity O&G monitoring
pH adjustment system	pH	single-stage pH adjustment
Aeration basins	BOD+COD+phenolics+O/G+heavy metals+ sulfide+aquatic toxicity	Increased aeration capacity
pH adjustment/secondary metals precipitation system	pH+heavy metals	pH adjustment including FeCl <sub>3</sub> + polymer addition
Clarifiers	BOD+COD+TSS+heavy metals	Improved heavy metals removal+ upgrade sludge recycle/wastage control
Dual-media filters	BOD+COD+heavy metals+TSS	Installation of down flow filters+ effluent turbidity monitoring
Activated carbon columns	BOD+COD+phenolics+heavy metals+aquatic toxicity	Installation of down flow adsorbers

The economic evaluation of activated carbon treatment options included estimates of capital costs, operating and maintenance (O&M) costs, and annual costs (Table 5). The PAC biological system has slightly lower capital costs (about \$1.4 vs. \$1.6 million), slightly lower O&M costs, and slightly lower annual costs, primarily due to its lower carbon costs.

The PAC biological system is projected to be slightly less expensive. However, it will not produce as good an effluent for organics and probably aquatic toxicity, and PAC dosing requires more control than GAC. Also, this system is considered to be less responsive to variations in influent characteristics and organic loadings. Also, it would have an advantage over carbon columns (it can provide protection to the activated sludge biomass from shock loads or toxic spills). Additionally, some time, two to three weeks or two or three months, involving operation with only one clarifier would be required. Therefore, the granular carbon column system was selected for installation at Canton.

**The upgrade plant.** The upgraded treatment facility (Fig. 2) will include two-stage heavy metals precipitation and removal. Sodium sulfide and ferric chloride will be used in the first-stage precipitation system with metals removal in the existing DAF units. A new three-stage wastewater pH control system and new chemical feed system will be added to the DAF influent stream. The existing flocculation and DAF units will be improved, also, The DAF unit modifications include provisions to add more air for removal of the additional solids.

Secondary metals treatment will be provided by ferric chloride addition to the aeration basin mixed liquor, a new rapid-mix and flocculation system and settling in the existing secondary clarifiers. The excess metals from this step will be removed with the waste-activated sludge. New dual-media filters followed by granular activated carbon columns will provide further polishing of TSS, heavy metals, organics, total phenolics and aquatic toxicity.

Mixing in the equalization tank is being improved by three new mixers. The activated sludge system is being upgraded to include additional aeration capacity and phosphoric acid addition. The wastewater management system is being further upgraded by collection of all foal water discharges for ammonia removal in the existing in-plant sour water steam stripping system. Table 6 lists parameters of concern and modifications being implemented for additional control.

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