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Environmental Protection Technology Series

Pilot - Demonstration Project For Industrial Reuse Of Renovated Municipal Wastewater

Office of Research and Development
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PILOT-DEMONSTRATION PROJECT FOR INDUSTRIAL REUSE OF RENOVATED MUNICIPAL WASTEWATER

By

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ABSTRACT

Three pilot plant treatment sequences were operated during this study to produce various grades of effluent for subsequent testing as industrial water sources. The testing was conducted in pilot-sized test loops consisting of small cooling towers and heat exchangers. At the same time the renovated waters were tested, Contra Costa Canal water, which is presently used by industry in the study area, was also investigated in a test-loop identical to those used for the renovated water.

The study results illustrated that the wastewater investigated can be treated satisfactorily for reuse in industrial applications. Corrosion rates and fouling factors observed with renovated water were equal to or less than found with the canal water. Precipitation of phosphorous was the major source of scale formation while using renovated water for cooling purposes, thus indicating the need for phosphorous removal.

Biological oxidation of organic materials and ammonia in a multistage treatment system resulted in renovated water suitable for industrial reuse. Filtration and phosphorous removal in association with biological treatment were also advantageous. Physical-chemical treatment processes can produce suitable renovated water provided that a suitable method is developed to prevent the generation of noxious odors in the activated carbon process.

Costs for the alternative treatment systems ranged from approximately 25¢/1000 gal for conventional activated sludge treatment to 50¢/1000 gal for a three-stage biological treatment system with filtration and alum addition for phosphorous removal. Physical-chemical treatment costs, including the cost for nitrogen removal, ranged from about 38 to 45¢/1000 gal. The costs for renovated water should be significantly less than the above costs in those cases where high degrees of treatment are required to discharge the treated effluent to a receiving body of water.

This report was submitted in fulfillment of Project Number 17080 FSF, by the Contra Costa Water District and the Central Contra Costa Sanitary District, under the partial sponsorship of the Environmental Protection Agency.

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Section I

INTRODUCTION

This report presents the results of a pilot-demonstration investigation on wastewater renovation conducted by Bechtel Corporation under a contract dated July 27, 1970, with the Contra Costa County Water District (CCCWD) and Central Contra Costa Sanitary District (CCCSD). The investigation was partially funded by a Class IV Research and Development Grant from the Environmental Protection Agency. Figure 1-1 shows the project area location. This location is adjacent to industries generally located along the south shore of Suisun Bay.

MOTIVATION

In 1969, during a feasibility study conducted by Bechtel for the CCCWD, it was concluded that "it is technically feasible to produce renovated water for the CCCWD for a wide range of irrigational and industrial uses" (Reference 1). As a result, it was recommended that (1) a sampling and analysis program be established to characterize local waters and wastewaters, (2) a pilot-demonstration program be initiated, and (3) a master plan for water renovation be developed. It was also recommeded that an agreement be established for cooperation between the CCCWD and CCCSD in undertaking additional wastewater renovation investigations.

The CCCWD and CCCSD, on December 3, 1969, entered into a memorandum of understanding which:

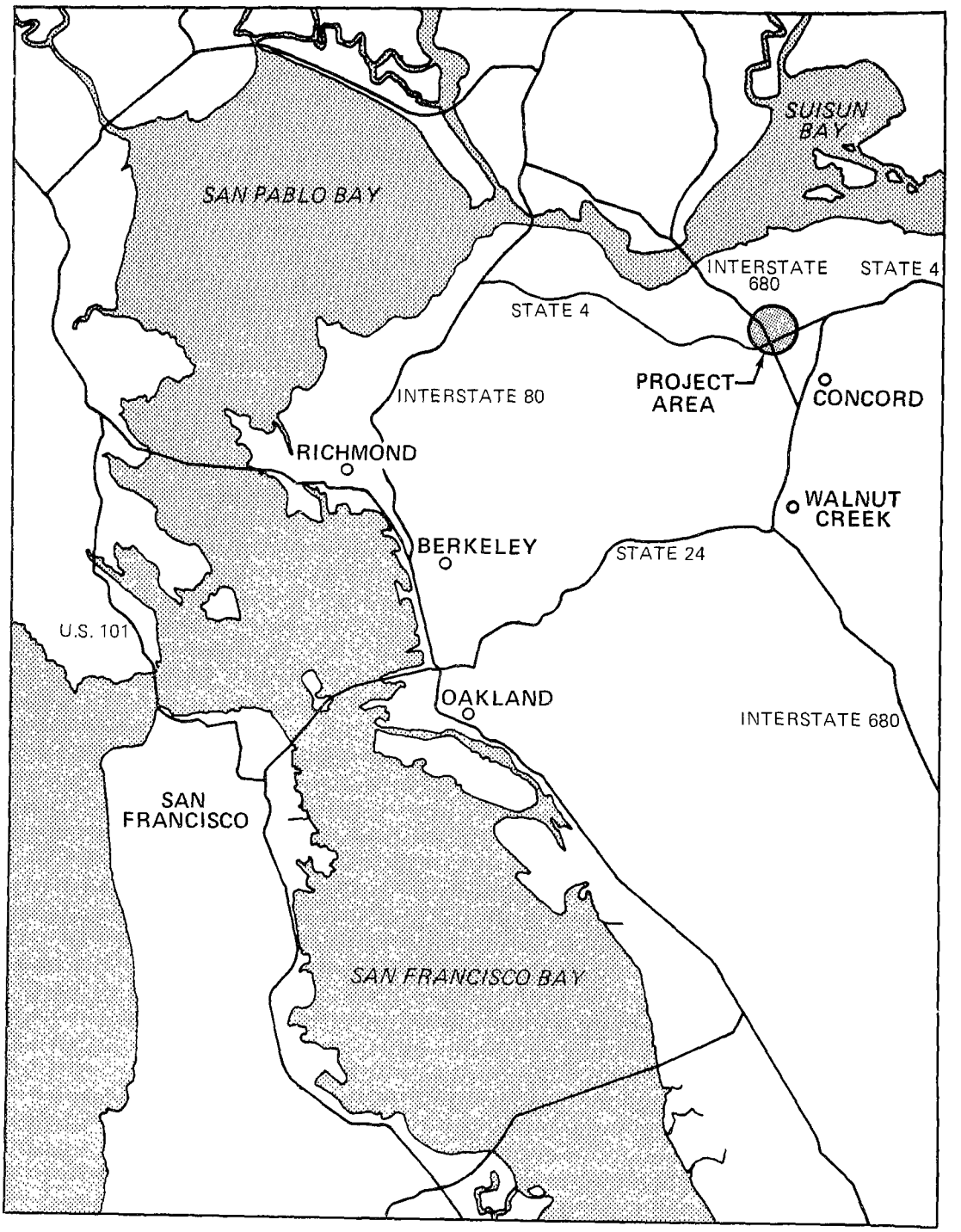

Figure 1-1. Geographic Location of Project Area

- Recognized the basic water supply function of the CCCWD and water pollution control function of the CCCSD
- Established the intent of both Districts to cooperate in future water renovation and reuse activities
- Outlined the following six-phase program:

Phase	Activity
I	Water Renovation Feasibility Study (sponsored by CCCWD and completed September 1969)
II	Solid Waste Disposal Investigation (sponsored by CCCSD and completed March 1970)
III	Sampling and Analysis Program (jointly sponsored by CCCWD and CCCSD and completed November 1971)
IV	Pilot Plant Program (jointly sponsored by CCCWD and CCCSD)
V	Demonstration Program (jointly sponsored by CCCWD and CCCSD)
VI	Implementation Plan for wastewater renovation and solid wastes disposal

Because of the national significance associated with Phases IV and V, the CCCWD and the CCCSD applied for, and were granted on February 19, 1970, a Class IV research and demonstration grant from the Federal Water Pollution Control Administration (now Environmental Protection Agency) to carry out the pilot-demonstration program.

The motivation for this investigation stems from the activities listed above, which reflect the CCCWD's and the CCCSD's desire to conserve our water resources by water reuse.

OBJECTIVES

Objectives of the pilot-demonstration program using various wastewater treatment process sequences were to:

- Allow investigation of the removal of various impurities in wastewaters by certain treatment processes
- Produce various grades of renovated waters whose properties will be tested for factors of importance in industrial water use
- Provide process data that, along with other available information, will be used in order-of-magnitude cost comparisons of various water renovation processes

Section II

CONCLUSIONS AND RECOMMENDATIONS

Three pilot plant treatment sequences were investigated during this study to determine the suitability of various grades of renovated wastewater for industrial reuse (Figure 2-1). In the Phase I treatment sequence, primary effluent from the existing CCCSD treatment plant was given biological treatment in the pilot plant. The biologically treated effluent, before and after filtration, was chlorinated and used as cooling water makeup for the industrial test loops.

In the Phase II pilot plant sequence, primary and activated sludge treatment was provided in the CCCSD treatment plant, while ammonia was biologically oxidized to nitrate in the pilot plant nitrification process. The nitrate was subsequently removed biologically as nitrogen gas in the denitrification process (Figure 2-1). Filtration and activated carbon adsorption were used as additional treatment steps on a portion of the effluents from the nitrification and the denitrification processes. Nitrified, filtered nitrified, and activated-carbon-treated nitrified effluents were used as makeup water for the industrial test loops. Similar combinations of denitrified effluents were also used in the cooling towers.

The Phase III treatment sequence incorporated only chemical and physical processes. Wastewater was given preliminary treatment for the removal of grit, rags, and large suspended materials in the existing CCCSD plant before being conveyed to the pilot plant, where lime was added to remove suspended solids and phosphorous in the first stage flocculation-sedimentation processes. Carbon dioxide was then added to reduce the pH

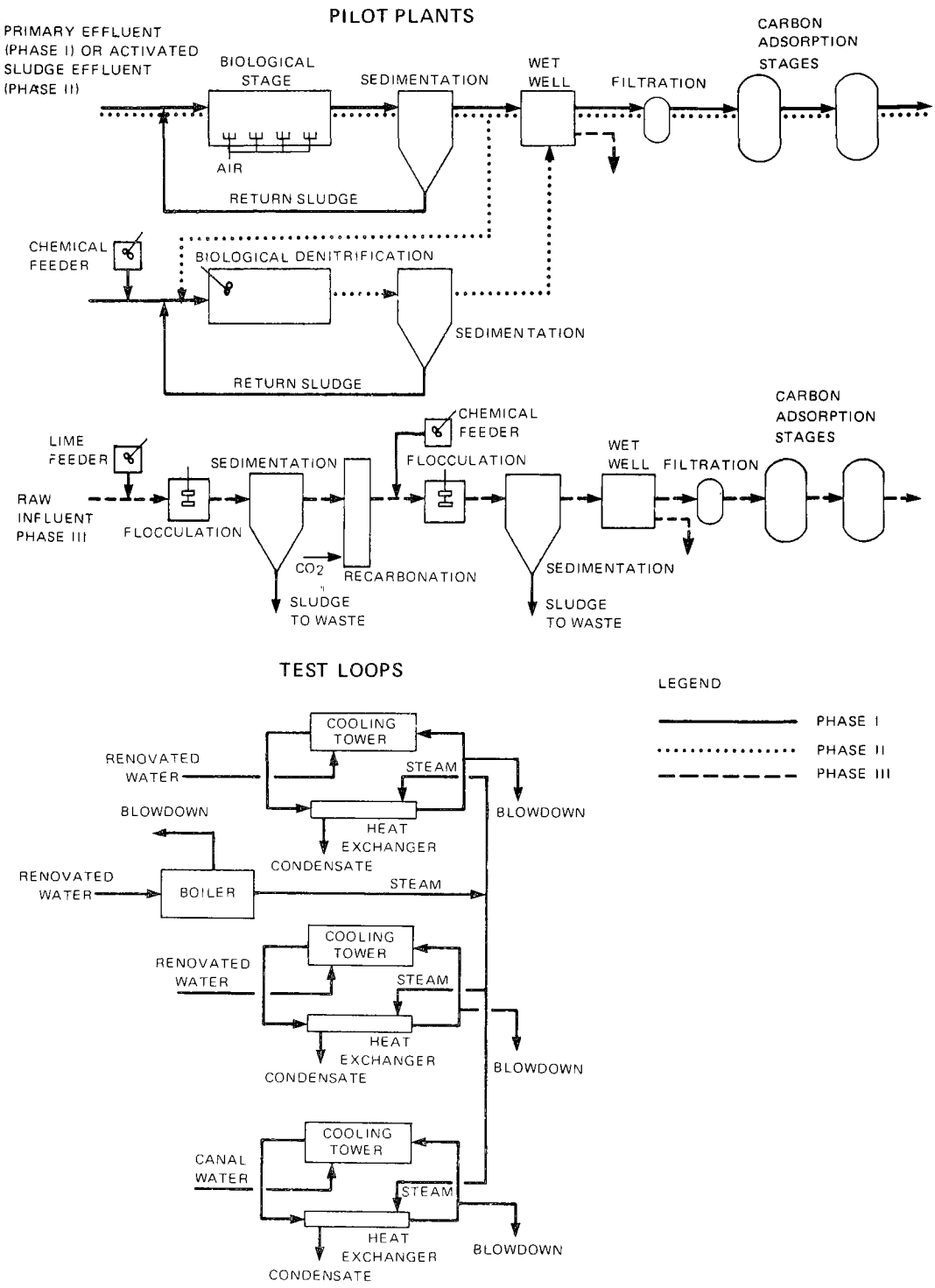

Figure 2-1. Process Flow Diagram for the Pilot – Demonstration Facility

prior to the removal of excess calcium in the second stage flocculation-sedimentation processes. Filtration followed for the removal of the remaining suspended solids, and activated carbon treatment was used to reduce the organic carbon content of the filtered effluent. After chlorination, a portion of the treated wastewater was used as makeup water for the industrial test loops. Filtered chemically treated effluent was also used as cooling water makeup.

In addition to testing various grades of renovated water in the industrial test loops, the Contra Costa Canal water that is presently used by industries in the area was also tested. Such tests permitted direct comparison of the renovated waters with the existing water source.

Conclusions regarding industrial reuse and the various treatment sequences investigated in this study are discussed in this section. Recommendations based on the pilot-demonstration investigations are also presented.

CONCLUSIONS

Industrial Reuse

- Based on the industrial test loop studies, wastewater can be treated satisfactorily for reuse in industrial applications. Major areas of concern for cooling water reuse are calcium, sulfate, phosphate, and silicate as they relate to scaling, and the maximum cycles of concentration one can maintain without excessive maintenance costs. Organics and total dissolved solids (TDS) are the most important factors affecting boiler feed water treatment costs.
- The corrosion rate of carbon steel as determined with corrosion coupons, for all renovated waters tested without the use of corrosion inhibitors, ranged from 2.0 to 31 mils/year, which was less than that for untreated canal water (12.5 to 37 mils/year). Canal water with the addition of a

corrosion inhibitor was observed to have a corrosion rate of 2.7 mils/year. Based on these results, it can be concluded that renovated water can be produced that is substantially less corrosive than untreated canal water.

- Fouling of heat exchanger surfaces by renovated wastewater was approximately equal to that of the untreated canal water. The fouling factors ranged from 0.0005 to 0.004 hr-^{o}F-sq ft/Btu for the renovated waters and 0.0005 to 0.003 hr-^{o}F-sq ft/Btu for the untreated canal water. Based on these results, it can be concluded that renovated water can be produced that will not cause any greater fouling problems than untreated canal water.

- Precipitation of phosphorous was the major source of scale formation while using renovated wastewater for cooling purposes, thus indicating the need for phosphate removal. The phosphorous concentration in the renovated water should be 0.5 mg/l as P or less in order to minimize scaling problems.

- Where chlorine is used in cooling water systems for the control of biogrowth, it would be desirable to remove ammonia from renovated water used for makeup purposes in order to reduce chlorine requirements. An effective removal procedure is the biological nitrification process in which ammonia is converted to nitrate.

- Since nitrate does not limit the cycles of concentration attainable in the operation of cooling towers, the need for denitrification cannot be justified on such a basis. However, lower algal growth anticipated with denitrified effluent compared to nitrified effluent suggests that denitrification may be helpful.

- Filtration of renovated water not only improves the water quality but also reduces potential problems arising from the deposition of suspended solids in industrial heat exchanger equipment. With filtered renovated water, this problem would not be as great as normally encountered during the use of canal water.

- Activated carbon treatment would not be required for biologically treated wastewater for its reuse as industrial cooling water. If renovated water is to be used for boiler feedwater, it may be more economical for industry to provide any additional treatment, such as activated carbon, for that portion of the water used for such purposes.

Biological Treatment

- Biological nitrification in a conventional single-stage activated sludge system can be achieved provided the sludge age is greater than 10 days and the dissolved oxygen is maintained at greater than 2.0 mg/l. A two-stage biological system designed to achieve nitrification is significantly more reliable and less difficult to control than a single-stage system.
- The presence of a limited concentration of organic materials in the nitrification reactor influent of a two-stage biological system is necessary to promote a required synergistic relationship between the nitrifying bacteria and the heterotrophic bacteria. These organic materials should have a total organic carbon (TOC) concentration of approximately 30 to 50 mg/l. Reduction of organic materials in typical municipal wastewaters to this level can be achieved with a high rate activated sludge system, a trickling filter, or a chemical-primary treatment process.
- The pilot plant denitrification data were quite variable due to difficulties encountered with the methanol feed system and solids separation. Effluent nitrate concentrations ranged from less than 1 mg/l to 20 mg/l and averaged about 3.2 mg/l, indicating an average removal of 82 percent. With an improved methanol feeding system and an aeration step between the denitrification reactor and the final clarifier, as found necessary in subsequent studies to improve solid separation, the denitrification process can be expected to perform satisfactorily.

Physical-Chemical Treatment

- Chemical treatment resulted in the most consistent effluent quality of all the pilot plant processes investigated during the study. Total phosphorous removals averaged about 97 percent, and about 70 percent of the total organic carbon (TOC) was removed in the chemical treatment system.

- Ferric chloride at a concentration of approximately 15 mg/l enhanced the coagulation of insoluble precipitates formed in the second-stage flocculation unit. In a large-scale treatment plant, recycling lime sludge or using coagulant aids would be expected to perform as well as the ferric chloride for this purpose and would thus minimize potential sludge handling problems.

- Because of the organic carbon concentration remaining after chemical treatment, either activated carbon or biological oxidation is required to lower the concentration of total organic carbon in the effluent to about 10 mg/l.

- The chemically treated effluent turbidity was approximately 5 JTU. This turbidity was reduced by only 20 to 40 percent by filtration. Improved filter performance may be expected if a polyelectrolyte or alum is added to the filter influent.

- Filtration following biological treatment increases process reliability, improves effluent quality, and should enhance disinfection as a result of significant decreases in the effluent suspended materials.

- Chlorination of the filtration process influent should be provided to prevent biological growths and subsequent clogging of the filter media.

- The data obtained in this study indicate that if activated carbon treatment is employed, a carbon contact time of approximately 15 minutes would be suitable for biologically treated effluents and 30 minutes for chemically treated effluents. Organic carbon removals averaged only about 2 or 3 mg/l in the second stage of the activated carbon process when the contact time in each carbon stage was 25 to 30 minutes.

- The pilot plant data indicated that approximately 410 pounds of activated carbon per million gallons of water treated would be required for biologically treated effluents, and 690 pounds per million gallons would be required for chemically treated effluents.

- While activated carbon reduced the total organic carbon concentration of the biological effluents to values as low as 2.0 mg/l, the reduction generally represented less than 10 percent of the total organic carbon removed from the raw wastewater. Since only 70 percent of the total organic carbon is removed by chemical treatment, activated carbon treatment of chemically treated effluents would be required.

Related Studies

- Various units in the treatment sequences of the pilot plant were spiked with poliovirus and monitored to determine the degree of inactivation resulting from each treatment step. Essentially all of the poliovirus was inactivated within five minutes of exposure in the first-stage lime flocculation unit when the pH was 11 or above. Contact with the lime sludge at an operating pH between 10 and 11 and with biological sludges reduced the active viral count by three to four orders of magnitude. Activated carbon treatment removed about 75 percent of the virus, whereas little or no viral removal occurred during filtration or sedimentation.
- Results of Provisional Algae Assay Procedure (PAAP) tests demonstrated that there was little or no growth of the test algae for at least 15 days in the chemical treatment effluent, indicating that algal growth can be minimized by the removal of phosphorous. Denitrified effluent resulted initially in a logarithmic growth of algae, although after approximately five days the rapid growth stopped, thus suggesting that nitrogen became the limiting nutrient.
- Cooling tower circulating water which had approximately five cycles of concentration was not toxic to fish (stickleback) after 96 hours of exposure, when canal water, nitrified effluent, filtered nitrified effluent, filtered chemical treatment effluent, and activated carbon chemical treatment effluent were used as cooling water makeup.
- Heavy metals and trace organics, including pesticides, phenols, and detergents, are generally reduced by 50 to 99 percent in the biological, chemical, and physical treatment-renovation processes.
- Treatment costs for the various sequences investigated are summarized in Figure 2-2 for 30-mgd plants. All of the costs are based on mid-1972 prices as discussed in Section 6. The treatment costs range from approximately 25¢/1000 gal for conventional activated sludge treatment to 50¢/1000 gal for a three-stage biological treatment system with filtration and alum addition for phosphorous removal. Chemical treatment preceding biological nitrification, biological denitrification, and filtration costs approximately 41¢/1000 gal. This treatment cost was within the range of costs associated with physical-chemical treatment including nitrogen removal (i.e., approximately 38 to 45¢/1000 gal).

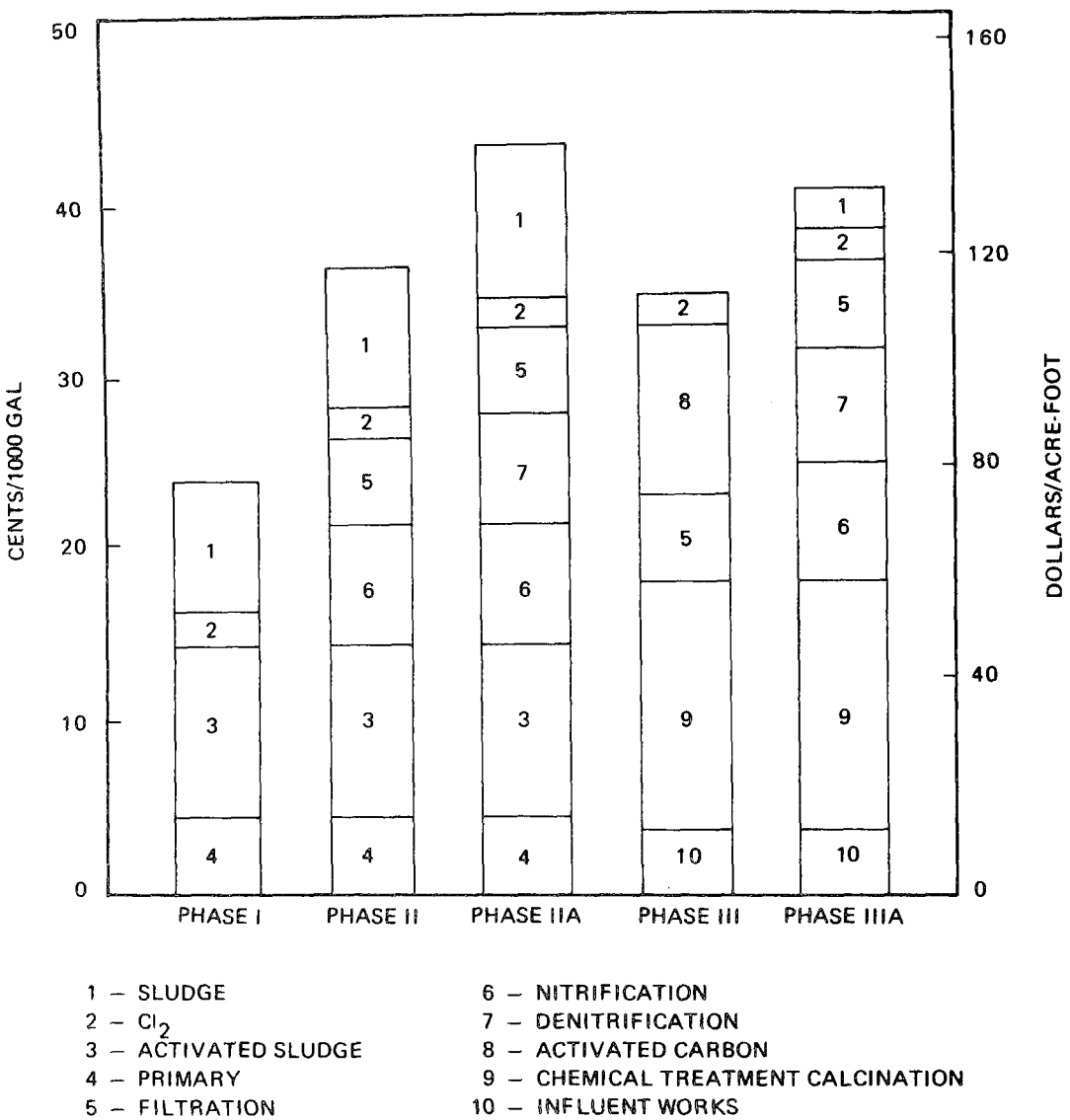

Figure 2-2. Approximate Treatment Costs for 30-mgd Plants

RECOMMENDATIONS

The following recommendations are offered for consideration:

- Additional investigations should be conducted on the biological denitrification process in order to develop:*
  - Improved control of the methanol feed. (A continuous TOC analyzer or a selective nitrate probe may be satisfactorily used for this purpose.)
  - Improved solids separation of mixed liquor suspended solids. (Intermediate aeration between the denitrification reactor and the sedimentation basin may be desirable. Dissolved air flotation may also be worthy of further study.)
- Further studies on the activated carbon process are recommended to:
  - Optimize the carbon contact times required for biologically and chemically treated effluents
  - Evaluate the necessity of filtration preceding activated carbon treatment
  - Further investigate methods to eliminate the generation of noxious odors in carbon columns
  - Investigate the advantages and disadvantages of using two carbon stages in series versus a single stage, as well as the expanded bed mode of operation versus the packed bed
- Sludge disposal methods should be evaluated to:
  - Determine optimum dewatering methods such as vacuum filtration and centrifugation
  - Ascertain fuel requirements and product recovery values associated with incineration and recalcination
  - Establish the market value of digested sludge as a liquid fertilizer and, after drying, as a soil conditioner

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*Since the time the pilot plant results presented in this report were completed, CCCSD has conducted additional large-scale studies on the denitrification process.

Section III

EXPERIMENTAL PROGRAM

DESCRIPTION OF FACILITIES

A process flow diagram of the wastewater renovation pilot plant facilities and the industrial test loops are presented in Figure 2-1. The pilot plant facilities consist of biological processes (activated sludge, nitrification, and denitrification), physical treatment processes (filtration and activated carbon adsorption), and the chemical treatment processes of lime coagulation and flocculation. The industrial test loops used during this study were designed to simulate the use of cooling water in industrial applications. Figure 3-1 is a perspective drawing showing the relative locations of the pilot plant facilities and the industrial tests loops.

Pilot Plant Facilities

In Figure 3-1, the rectangular basin labeled biological stage was 8 feet wide, 16 feet long, 11 feet deep, and contained 32 submerged air diffusion units (Chicago Pump). This vessel was initially used as a conventional activated sludge basin treating primary effluent from the CCCSD plant. Later the active volume of this vessel was reduced by baffling, and the vessel was used as a nitrification basin treating activated sludge effluent from the CCCSD 1-mgd plant.

The biological denitrification basin was 5 feet 6 inches in diameter and 11 feet deep. During the testing period on biological denitrification, this basin was continuously stirred with a submersible pump. Methyl alcohol

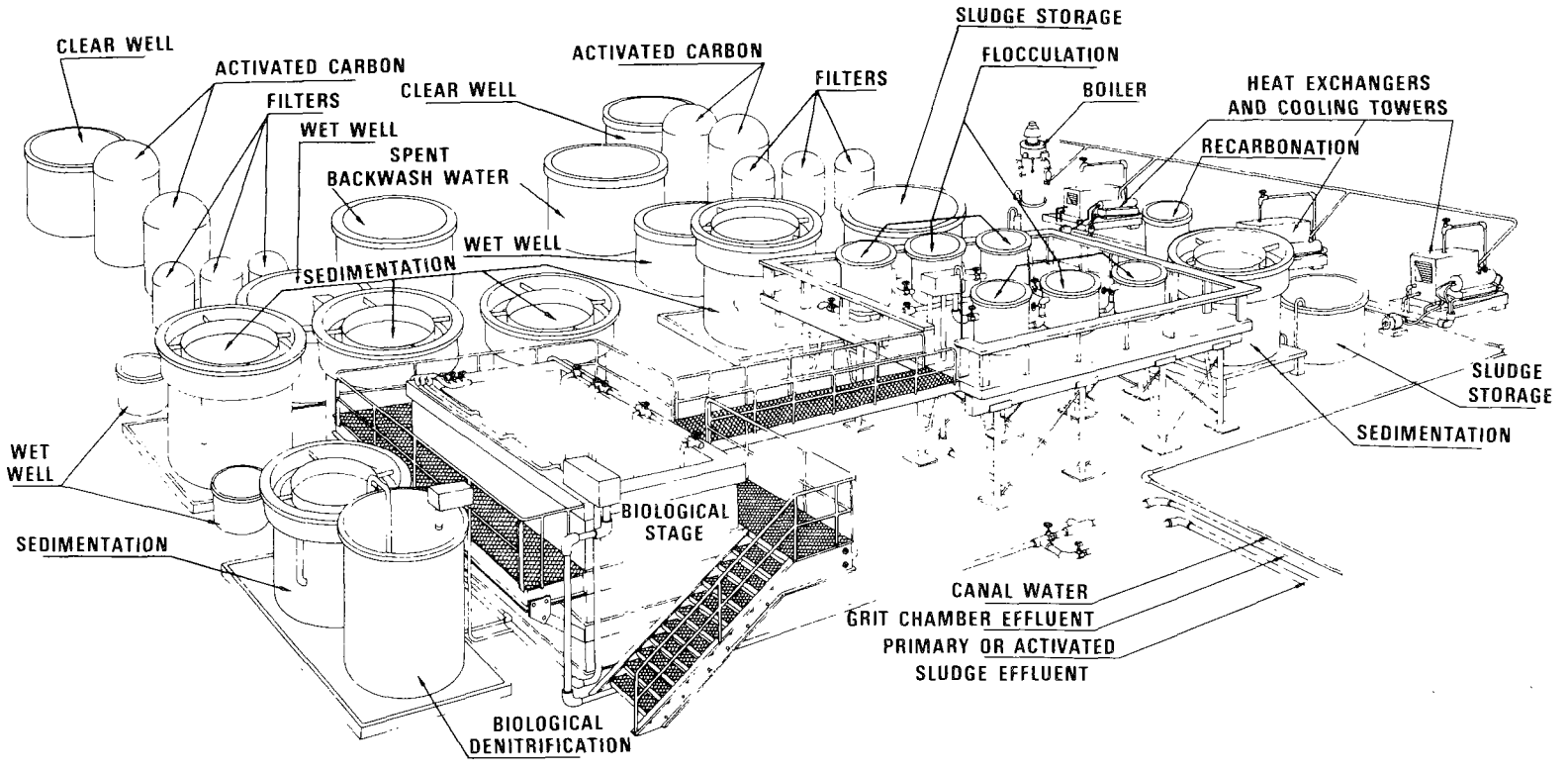

Figure 3-1. Perspective Drawing of Pilot-Demonstration Facilities

was added continuously to the denitrification basin using a variable-speed diaphragm pump. For a short period of time, the denitrification basin was used for biological nitrification since it was initially equipped with air diffusers.

All sedimentation basins used in the pilot facilities were designed identically. These basins were 6 feet in diameter and 9 feet 11 inches deep with an external peripheral effluent channel near the overflow weir. A 60-degree cone bottom, constructed in each basin, was connected to a horizontal pipe located near the bottom of the basin for sludge withdrawal.

Lime addition to the physical-chemical pilot plant influent (i.e., partially settled raw wastewater) was regulated by a preset pH controller (Uniloc Model 1000). The pH sensor was located in the first stage flocculation tank, and the pH was continuously recorded in the laboratory trailer.

The flocculation tanks in the physical-chemical system were each 2 feet 6 inches in diameter and 3 feet 4 inches deep. Three flocculation tanks were provided for each stage of treatment. Inlet and outlet piping was provided to facilitate removing any one or all of the flocculation tanks from the operation. Each operating flocculation tank was equipped with a clamp-mount variable-speed mixer (Lighting Model ND-1YM) with a flat blade turbine impeller.

Recarbonation between the first and second stage of the chemical treatment system was provided in a tank 2 feet 6 inches in diameter and 10 feet deep. Bottled carbon dioxide gas was introduced through a diffuser into the bottom of the recarbonation tank, countercurrent to the liquid flow. Following recarbonation, precipitated calcium carbonate was flocculated with ferric chloride used as a coagulant. The ferric chloride was added continuously (using a diaphragm pump) to the flocculator in the second stage of the chemical treatment system.

The effluents from the biological processes and the chemical treatment process were given additional physical treatment of filtration followed by two stages of activated carbon adsorption. The filtration equipment consisted of three 20-inch diameter pressure filters (Bruner, Model AG20), each containing different media, as illustrated in Figure 3. Filters No. 1 and No. 2 contained anthracite coal (0.68 to 0.77 mm effective size, 1.75 uniformity coefficient) and graded sand (0.45 to 0.55 mm effective size, 1.50 uniformity coefficient). Filter No. 3 contained mixed media (Neptune Microfloc; MF-152) consisting of anthracite, sand, and garnet. Each stage in the activated carbon system included a pressurized 36-inch diameter vessel (Bruner, Model AC36) containing granular carbon (Filtrasorb 400, Calgon Corporation) at a depth of 2.5 feet. Graded gravel was used to support the filtration media as well as the activated carbon. Backwash rates of approximately 20 gpm/sq ft and 6 gpm/sq ft were used for the filters and the carbon columns, respectively. Surface wash mechanisms were installed in each filter and carbon column.

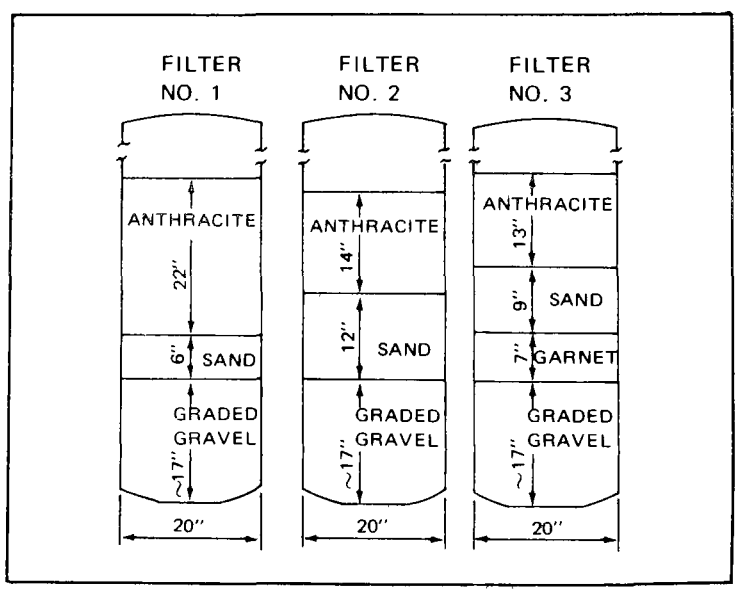

Figure 3-2. Pilot-Plant Filtration Media

Existing Treatment Plant

The existing treatment plant at CCCSD provides primary treatment to the incoming wastewater preceded by screening and grit removal. Approximately 30 minutes detention time is provided in the aerated grit chamber thus providing pre-aeration as well as grit removal. The design detention time in the primary sedimentation tanks for a 30-mgd average daily flow is 1-1/2 hours.

Sludges from the primary sedimentation tanks are anaerobically digested, dewatered on drying beds, and sold as a soil conditioner. The grit removed from the wastewater is dewatered and buried on the plant site. Gas produced during anaerobic digestion is used for fuel to operate gas engines used for generation of electricity. Digester supernatant is returned to the plant influent.

Pre-chlorination and post-chlorination of the wastewater is practiced, and the chlorinated effluent is discharged to Suisun Bay through an outfall pipeline. The primary effluent used in the pilot plant study was pumped to the pilot facilities before post-chlorination.

In addition to the 30-mgd primary treatment plant, a conventional activated sludge plant having a nominal capacity of 1 mgd is located at CCCSD. Primary effluent is treated in this plant and used for surface sprays and irrigational needs at the plant site. The activated sludge basin is rectangular in shape and is aerated using blowers connected to a diffused aeration system. A detention time of approximately 6 to 8 hours is provided in this basin. The sedimentation basin is also rectangular in shape providing a detention time of about 2 hours.

Industrial Test Loops

Three industrial test loops, each consisting of a small cooling tower, heat exchanger, and appurtenances, were included in the pilot-demonstration facilities. The cooling towers were 3 feet by 4 feet in plan with 20 inches of packing, which was constructed of 1/4 inch by 1-1/2 inch redwood splash bars having 2 inch horizontal spacing and 1-5/16 inch vertical spacing (Marley Model 4411).

Each heat exchanger consisted of a steel shell, with 1/2 inch steam inlet and outlet, having an outside diameter of 4-1/2 inches (Schedule 40 pipe) and a length of 3 feet. The heat exchanger shell contained two 5/8 inch OD carbon steel removable tubes, each having a wall thickness of 0.065 inch (Calgon, Model Test Heat Exchanger).

The industrial test loops were equipped with appropriate valves, steam traps, strainers, rotometers, pressure gauges, and thermometers to facilitate their operation. Also, a corrosion probe (Magna) for instantaneous corrosion rate measurements and a corrosion test specimen bypass loop (Betz, Model No. 35D000110) were located in the circulating water on the heat exchanger tube outlet of each industrial test loop.

Two sources of steam were used in the industrial test loops. Excess steam available at 15 psi from the CCCSD plant was piped to the pilot plant area to serve as one source. The other source was produced at the pilot plant site using a vertical firetube boiler (Eclipse, Type Z). This boiler was rated at 173 lb steam/hour with a maximum operating pressure of 150 psig. Boiler feed water was filtered using cartridges and softened using ion exchange (Culligan Mark 5 Model CF-28-40).

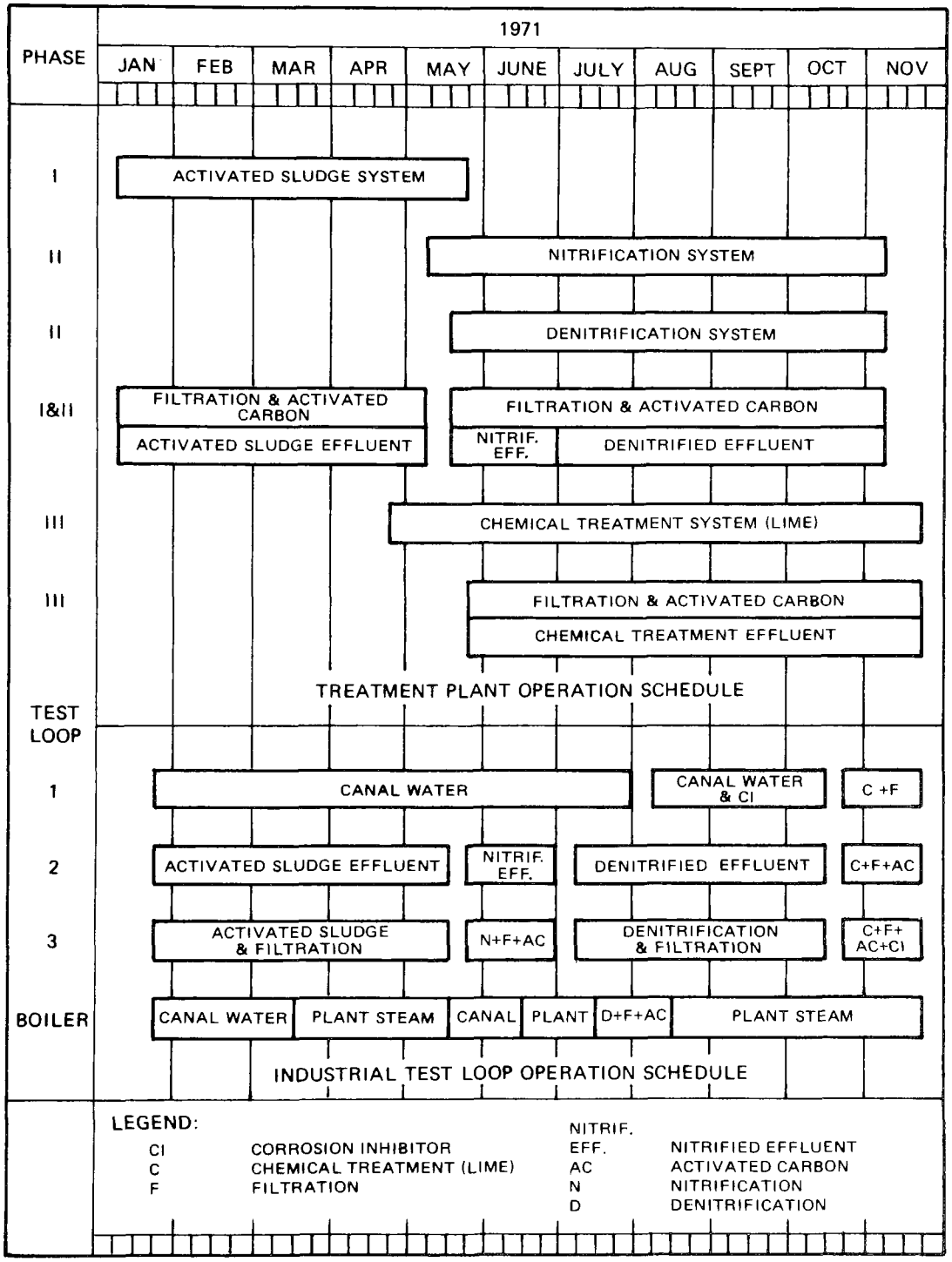

Figure 3-3. Pilot-Demonstration Plant Operating Schedule

OPERATING PROGRAM

Operation of the pilot facilities was divided into three phases. In addition to process evaluation, the major purpose of the experimental program was to test the use of the various renovated product waters in the industrial test loops. Activated sludge, nitrification-denitrification, and physical-chemical treatment processes were tested and evaluated, in Phases I through III, as discussed below. Figure 3-3 summarizes the operating schedule for the entire period of the pilot plant study. The upper portion of the figure refers to the operation of the treatment plants and the lower portion refers to the periods when treated wastewaters were fed to the industrial test loops.

Phase I

The treatment system adopted during Phase I consisted of activated sludge, filtration, and activated carbon adsorption, as shown by the solid line in Figure 2-1. The operation extended from January 6, 1971, through May 18, 1971. On May 3, 1971, effluent from the activated sludge process was used to start the second-stage biological (nitrification) culture. At the same time, the detention time in the activated sludge aeration basin was reduced by approximately one-half. The test loop operation started on January 20, 1971, with Loop 1 receiving the Contra Costa Canal water, Loop 2 receiving activated sludge effluent, and Loop 3 receiving filtered activated sludge effluent. The canal water was also used for boiler feed after it was treated with cartridge filters and softened by zeolite resin.

Phase II

During the operation of Phase II, the CCCSD activated sludge effluent was used as the influent to the pilot plant facilities (dotted line Figure 2-1). The Phase I activated sludge basin was used for the biological nitrification basin, and the denitrification reaction was carried out in the basin that was used for nitrification during Phase I. Nitrified effluent from the Phase II treatment system was filtered and treated with activated carbon from May 20 through June 30, 1971. From July 1 until November 4, 1971, the filters and activated carbon units were used for treatment of the denitrified effluent.

Chemical addition for phosphate removal was investigated during this phase. The denitrification reactor was dosed with ferric chloride from June 16 through September 2, 1971 and with alum from September 3 through October 26, 1971. In addition, during the week of September 20, polymer was added to the influent of the denitrification sedimentation basin.

During this phase, Loop 1 continued to receive Contra Costa Canal water and, until June 30, Loops 2 and 3 received nitrified effluent before filtration and after carbon adsorption, respectively. On July 1 and until October 29, Loops 2 and 3 received the denitrified effluent before and after filtration, respectively. The boiler continued operation as in Phase I until June 15, at which time CCCSD plant service steam was used in the industrial test loops. From July 15 through August 15, denitrified effluent after carbon adsorption was used as the boiler feedwater. The 15-psi CCCSD plant service steam was utilized during the remainder of the project.

Phase III

An evaluation of the physical-chemical treatment system was made from March 15 through November 23, 1971. After approximately 10 minutes of sedimentation, the partially settled raw wastewater was flocculated with lime at a pH value of approximately 11.0 and recarbonated to pH 9 to 9.5. The calcium carbonate formed after recarbonation was flocculated with approximately 30 to 40 mg/l of ferric chloride to improve floc formation before final sedimentation. During the period from October 29 through November 23, the industrial test loops were used to investigate effluents from the physical-chemical treatment system.

The filtered effluent from the two-stage chemical treatment plant was fed directly to Loop 1, whereas Loops 2 and 3 received the filtered water that had the additional benefit of treatment with activated carbon. An average of about 25.0 mg/l of zinc-chromate (Nalco 370) was added to the influent water of Loop 3 to determine its impact on any fouling or corrosion that might develop.

SAMPLING AND ANALYSIS PROGRAM

A sample collection and analysis program was established to monitor the performance of the pilot facilities. Descriptions of the laboratory facilities, sampling program, and analytical methods are presented below.

Laboratory Facilities

A trailer laboratory located at the pilot plant site served as the center for the sample preparation and analysis. The CCCSD Laboratory and Bechtel Environmental Laboratory at Belmont were used for special analyses such as fish toxicity studies and algae growth tests. The trailer laboratory was equipped to handle the routine analyses.

Sampling Program

Samples were collected at several points in the treatment system during each phase of the pilot plant studies, as indicated in Figure 3-4. The composited samples were collected daily and were analyzed for specific constituents on a daily basis. Additional analyses were performed either three times a week or weekly. Appendix A summarizes the sampling frequencies. Automatic samplers, which collected approximately 250 ml of sample each hour, were generally used to provide the daily composited sample for all analyses except for mixed-liquor solids in the biological units. The latter samples were daily "grab" samples since the concentration of these solids did not greatly change during a 24-hour period.

Because of clogging problems encountered with the automatic samplers handling the influent raw waste to the physical-chemical pilot plant, samples were taken manually every 8 hours and composited for analyses for this particular stream.

Analytical Procedures

Analytical methods used in the program generally followed References 2, 3, and 4 (the 12th Edition of Standard Methods and ASTM procedures). Table 3-1 summarizes the methods used and the source references for the procedures. Some modification of certain methods was necessary to assure thorough analytical coverage. Appendix A gives a detailed description of the procedures and instrumental methods used in this study that are not available in the 12th Edition of Standard Methods. Rigorous sample preparation techniques stressing acid digestion and blending were developed for the heavy metal analyses.

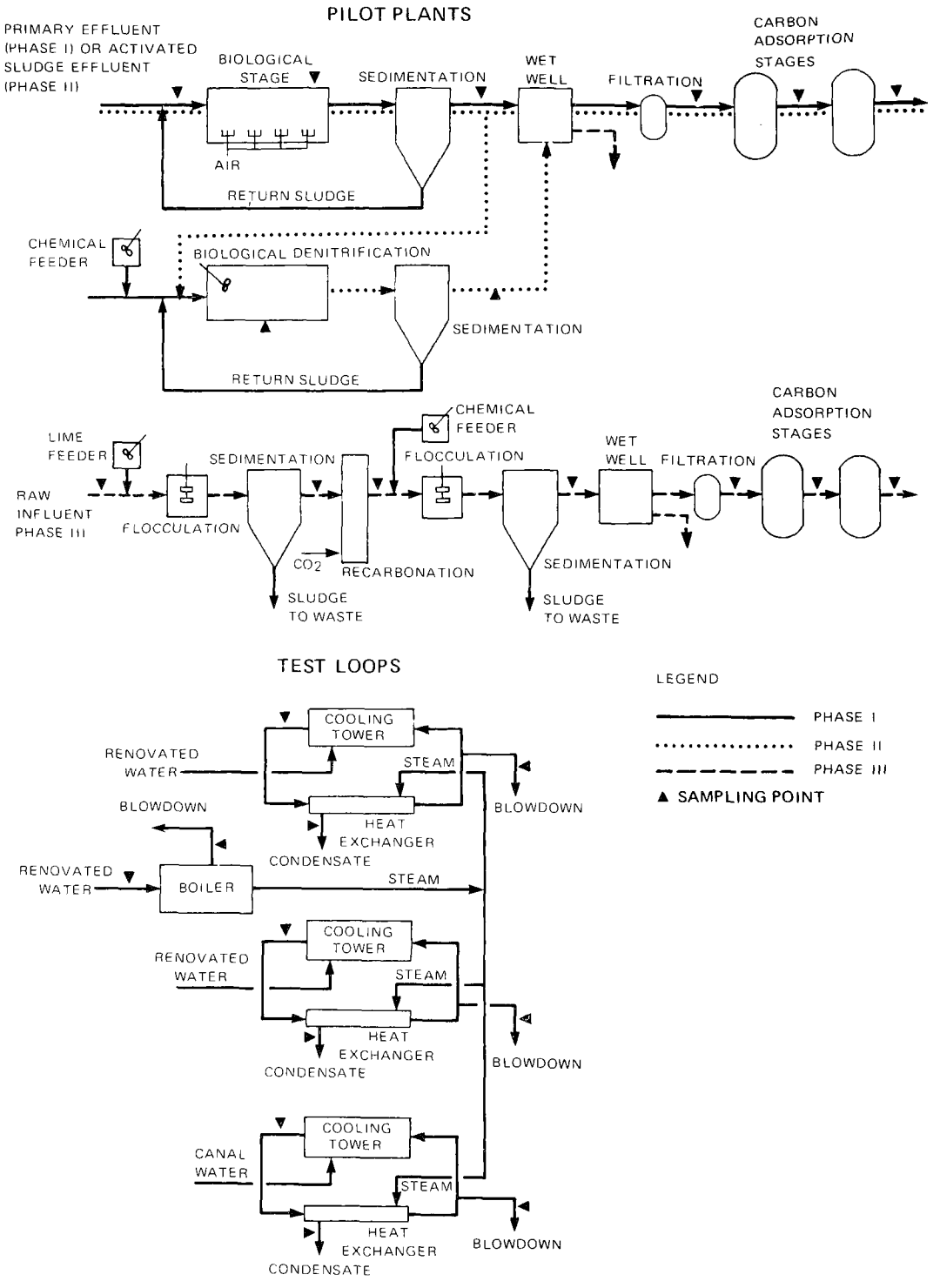

Figure 3-4. Sampling Points in the Pilot-Demonstration Facility

Table 3-1

ANALYTICAL PROCEDURES

Determination	Method	Reference
Conductivity	Wheatstone Bridge	2
pH	Glass Electrode	2
Turbidity	Photometric	2
Total Solids	Gravimetric	2
Dissolved Solids	Gravimetric	2
Suspended Solids	Gravimetric	2
Biochem Oxy. Dem. (BOD)	5-day Incubation	2
Detergents (MBAS)	Methylene Blue	2
Carbon Chloroform Extr.	Direct $CHCl_3$ Extr.	2
Total Organic Carbon (TOC)	Combustion-IR (Sample Unfiltered)	2
Soluble Organic Carbon (SOC)	Combustion-IR (Sample Filtered)	2
Organic Nitrogen	Kjeldahl	2
Phenol	Colorimetric-Distillation	2
Pesticides	Electron Capture G/l Chrom.	2
Alkalinity	Titration	2
Ammonia	Nessler	2
Boron	Curcumin	2
Calcium	EDTA Titration	2
Chloride	Mohr	2
Chromium, Hexavalent	Colorimetric	2
Copper	Cuprethol	2
Hardness	EDTA Titration	2
Iron	o-Phenanthroline	2
Magnesium	EDTA Titration	2
Nitrate	Reduction	2
Phosphate, Ortho	Colorimetric	2
Phosphate, Total	Colorimetric	2
Potassium	Atomic Absorption	2
Silica, Dissolved	Colorimetric	2
Silica, Total	Emiss. Spectrograph	4
Sodium	Atomic Absorption	3
Sulfate	Gravimetric	2
Trace Metals (Dissolved)*	Atomic Absorption	4
Trace Metals (Total)*	Emiss. Spectrograph	4
Mercury	Colorimetric	2
Selenium	Colorimetric	2

*Trace Metals include aluminum, arsenic, barium, boron, cadmium, chromium (tot; chromium (hex.), cobalt, copper, iron, lead, lithium, manganese, mercury, molybdenum, nickel, silver, strontium, tin, titanium, vanadium, zinc, and zirconium.

Section IV

RESULTS OF WASTEWATER TREATMENT STUDIES

The results from Phases I, II, and III of the pilot plant studies at the CCCSD treatment plant are presented and evaluated in this Section. A description of the experimental results from the industrial test loops is presented in the following section.

PHASE I: BIOLOGICAL-PHYSICAL TREATMENT SYSTEM

The biological treatment system was operated initially as a conventional activated sludge plant and later as a multistage plant for biological nitrification and denitrification (Figure 2-1). Physical treatment consisted of filtration through dual or mixed-media filters followed by activated carbon adsorption. At different times during the study, the effluent from the various biological-physical process steps was conveyed to the industrial test loops for evaluation of its suitability for industrial use.

Pilot Plant Activated Sludge

The activated sludge process was in operation from January 6 through May 18, 1971. Figure 4-1 indicates the flow and general characteristics of the activated sludge system. The mixed-liquor suspended-solids concentration during the first month was approximately 2000 mg/l and then increased to a steady-state concentration of about 3000 mg/l during the remaining periods. The volatile suspended solids (VSS) in the mixed liquor averaged 80 percent, whereas the sludge volume index (SVI) averaged 250 ml/gm during the test period.

The net growth rate in the system, defined as the mass of solids produced per unit time per unit mass of solids in the reactor, was quite variable during the first part of January and then remained at a steady-state value of approximately 0.04 day^{-1} during the latter part of January and February (Figure 4-1). Although the net growth rate was relatively low for activated sludge (typical values are 0.05 to 0.2 day^{-1}) during this period, the degree of nitrification was also low, as indicated by the influent and effluent ammonia concentrations shown in Figure 4-2. This is believed to have resulted from the 0.5 mg/l minimum DO concentration maintained in the activated sludge mixed liquor.

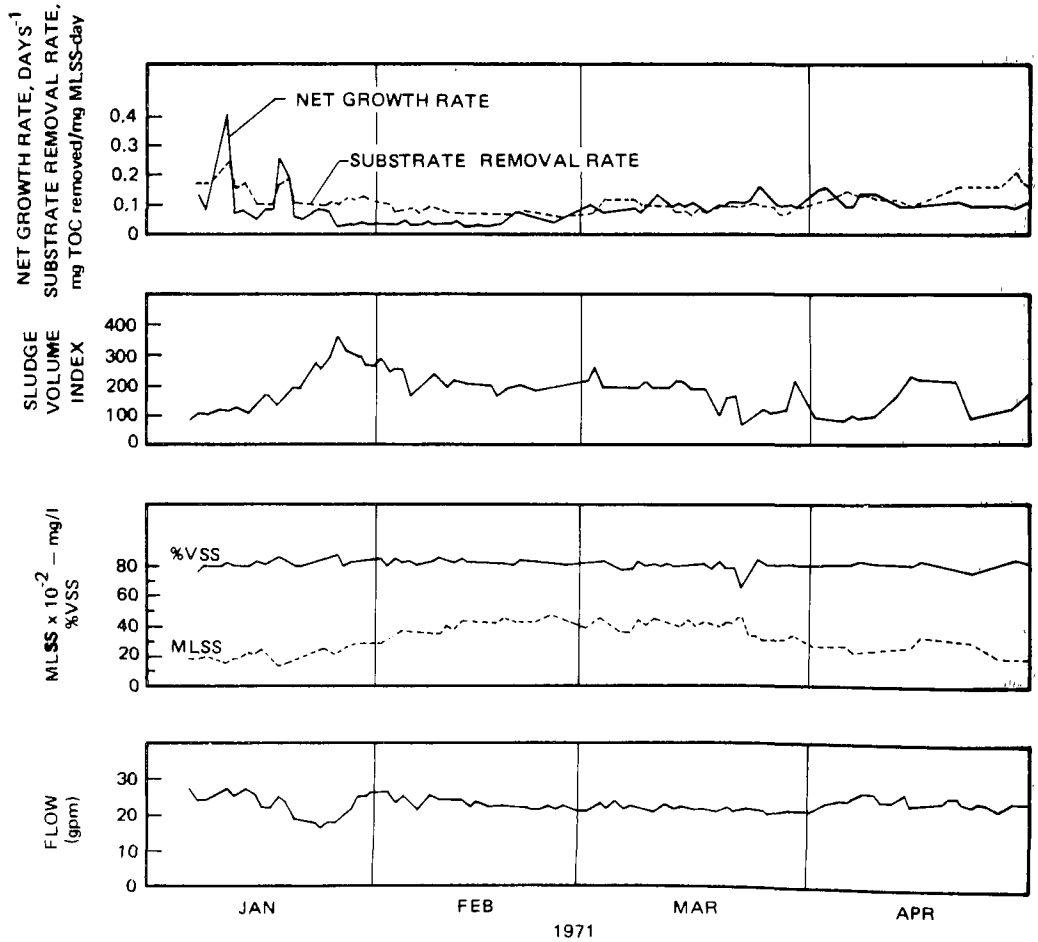

Figure 4-1. Activated Sludge Process Operating Characteristics

As indicated in Figure 4-2, the activated sludge influent ammonia concentration was usually between 20 and 30 mg N/l. Some nitrification did occur in the activated sludge unit as indicated by the increase in nitrate-N during January and again in March. However, this single-stage system was not consistently nitrifying, and thus rather high effluent ammonia concentrations were observed for a significant amount of the time. During the latter part of March and early April, the DO was increased to 2.0 mg/l and the effluent ammonia-N concentration was very low. However, the concentration increased somewhat by early April and remained at approximately 10 mg/l N thereafter. The data plotted in Figure 4-2 suggest that by maintaining a DO of about 2.0 mg/l, in

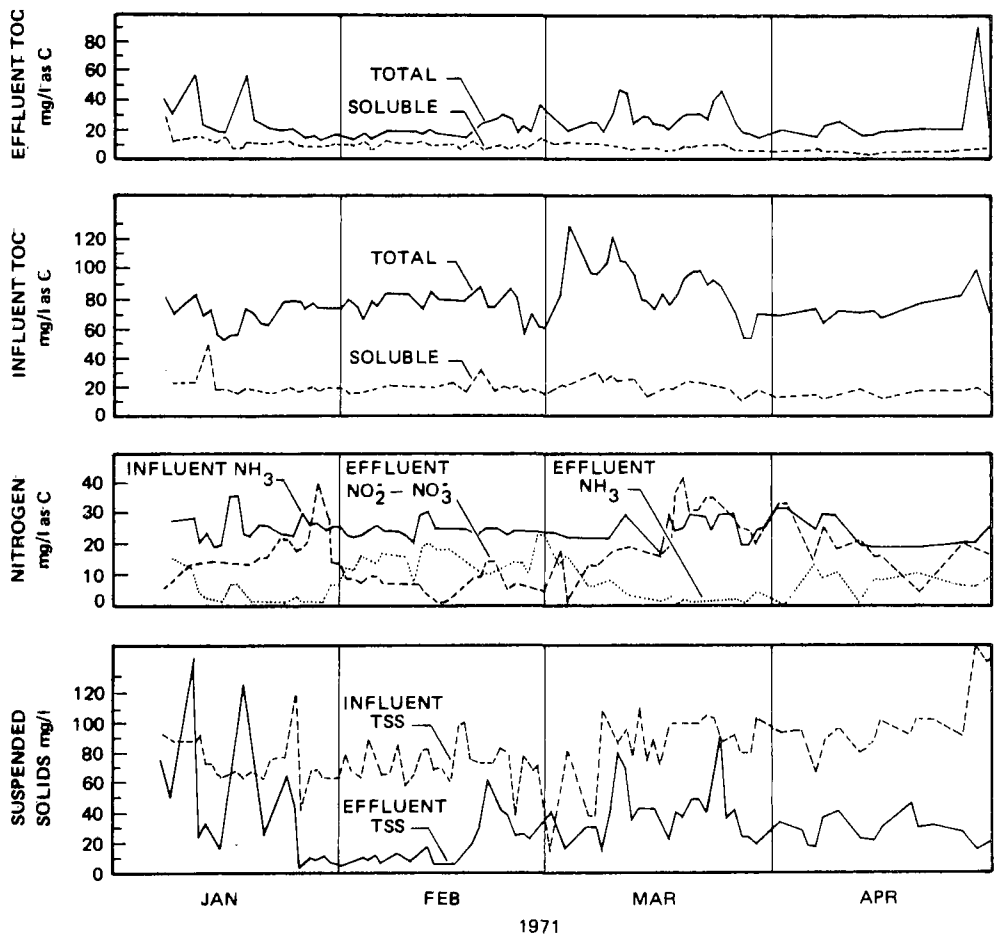

Figure 4-2. Activated Sludge Process Quality Characteristics

conjunction with a net growth rate of about 0.1 days^{-1} (Figure 4-1), it is possible to obtain consistently a 50 percent reduction in the influent ammonia-N concentration with the activated sludge process.

The effluent suspended solids concentrations were found to be quite variable, primarily because of floating materials in the secondary sedimentation tanks. These materials did not appear to be typical floating activated sludge. Instead, the materials appeared to consist of small globules with attached activated sludge floc and filimentous organisms. This problem indicated the need for permanent skimming equipment on the secondary sedimentation tanks. The influent suspended solids averaged 80 mg/l and the effluent 33 mg/l during this testing period.

The activated sludge influent TOC concentration averaged about 80 mg/l with a minimum of 55 mg/l and a maximum of 130 mg/l (Figure 4-2). While the SOC concentrations in the activated sludge effluent were nearly constant at about 9 mg/l, the effluent TOC varied in relationship to the effluent suspended solids and averaged about 26 mg/l.

In May, the loading on the activated sludge system was increased by blocking off a portion of the aeration basin volume. The effluent was then split with one stream going to the filtration and activated carbon systems and the other stream to a separate nitrification basin. A nitrifying culture had been established a few weeks earlier.

The activated sludge and nitrification process data for this operating period are summarized in Table 4-1. The degree of nitrification obtained during this short period of time indeed suggested that multistage biological treatment had much greater potential of nitrification than a single-stage biological process. The pilot plant operation was thus modified to a three-stage system to provide carbonaceous BOD removal in the first stage, nitrification in the second stage, and denitrification in the third stage, as described in Section 3 for the Phase II operation.

Table 4-1

PRELIMINARY ACTIVATED SLUDGE/NITRIFICATION DATA

	May 10, 1971	May 12, 1971	May 13, 1971	May 14, 1971	May 17, 1971	May 18, 1971
Flow, gpm	16	16	16	16	16	9.3
Activated Sludge						
Influent						
SS, mg/l	163	128	148	118	77	
TOC, mg C/l	102	82.5	92	80	64	
SOC, mg C/l	22	20.3	20	16	16	
Ammonia, mg N/l	21	26	20	23	29	
MLSS, mg/l	1840	1750	1980	2180	1930	2230
Effluent						
SS, mg/l	12.8	12.6	62.5	51.0	35.6	
TOC, mg C/l	16	20	35	29	33	
SOC, mg C/l	9.5	11.6	7.0	6.2	24	
Ammonia, mg N/l	13	20	11	9	24	
Nitrate, mg N/l	3	4	8	9	2	
Nitrification Effluent						
TOC, mg C/l		15	17	11	13	10
SOC, mg C/l		7.2	5.3	4.6	5	4.8
Ammonia, mg N/l	4	0.6	1.5	0.6	8	5
Nitrate, mg N/l	23	24	30	31	23	21

Filtration

Effluent from the biological treatment processes generally contained 10 to 30 mg/l of suspended solids. These solids contributed significantly to the effluent total organic content. Filtration of this effluent can, however, increase the plant reliability and result in a much more consistent effluent quality. Figure 4-3 summarizes the influent and effluent TOC and turbidity data for the three types of filter media investigated during this study at a filtration rate of 5.0 gpm/sq ft. The TOC concentration of the filtered effluent approached that of the SOC (10 to 20 mg/l), indicating that the filters were removing most of the suspended organic material. The filtered effluent turbidity was generally less than 2 JTU, and it was much better in quality than the unfiltered activated sludge effluent.

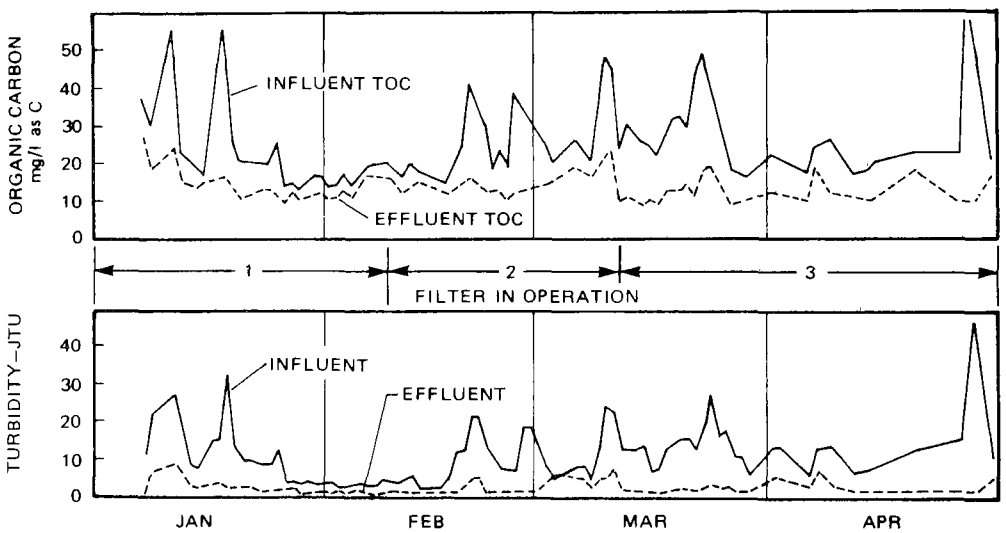

Figure 4-3. Filtration Data Using Activated Sludge Process Effluent

The average percent removals of TOC and turbidity are summarized in Table 4-2. The removals observed indicate very small difference in the three types of filter media tested. Filter No. 3, the mixed media, appeared to have somewhat higher removal efficiencies than Filter Nos. 1 and 2. However, the influent TOC and turbidity concentrations to

Table 4-2

SUMMARY OF FILTRATION PERFORMANCE USING ACTIVATED SLUDGE PROCESS EFFLUENT

Filter	Average Percent Removal	
	TOC	Turbidity JTU
No. 1 (Dual Media) 20 Percent Sand 80 Percent Anthracite	35	75
No. 2 (Dual Media) 45 Percent Sand 55 Percent Anthracite	39	73
No. 3 (Mixed Media) 25 Percent Garnet 30 Percent Sand 45 Percent Anthracite	50	79

Filter No. 3 during the testing period were generally higher than for Filter Nos. 1 and 2. Thus, one would anticipate somewhat higher removals. The cycle time between backwashings for a 20 psi pressure drop was approximately the same for Filter Nos. 1 and 3, with shorter times observed for Filter No. 2. For an influent suspended solids concentration of about 100 mg/l, the time between backwashings was only one to three hours. At an influent suspended solids concentrations of about 40 mg/l, filter runs were 10 to 15 hours, and, with an influent suspended solids of 10 mg/l, filter runs of approximately 30 hours were observed. Based on this information, normal filter runs for a typical plant may be in the range of 12 to 24 hours. However, during periods of relatively high solids in the filter influent (i.e., approximately 50 mg/l), filter runs as low as four hours may be expected.

Although the filtration process generally worked satisfactorily, problems were encountered with biological growth in the filter media. This growth resulted in what appeared to be channeling through the filter along with excessive head losses. Extended backwashing and the addition of heavy doses of chlorine did not eliminate the problem, and, consequently, the filter media were replaced. As a result, facilities were installed to chlorinate the feedwater to the filtration process. This procedure eliminated biological growth problems. Approximately 5 mg/l of chlorine was fed into the water before filtration, resulting in a post-filtration residual of approximately 0.5 mg/l Cl_2. Based on these observations, the ability to chlorinate the filter influent in a full-scale filter plant would be advantageous.

Activated Carbon

Two identical activated carbon columns were operated in series to treat the filtered effluent from the activated sludge units from January 6 through May 25, 1971. The empty-bed contact time for each column, calculated from the total bed volume and the volumetric flow rate, was about 11 minutes. Backwashing every four or five days at about 6.5 gpm/sq ft was required to keep the head loss below 10 psi. Figure 4-4 indicates the TOC and SOC concentrations for the first-stage influent and effluent and the second-stage effluent. The average activated carbon influent TOC was about 14 mg/l, while the activated carbon effluent TOC averaged about 8 mg/l. The data plotted in Figure 4-4 indicate that the first stage of the activated carbon system was removing the bulk of the material during most of the treatment period. The higher TOC removals observed in the first stage can be attributed partly to the filtration of suspended materials remaining in the filtered influent feed.

By May, the removal efficiency of the first stage in the activated carbon process had decreased significantly. Its effluent concentrations of TOC

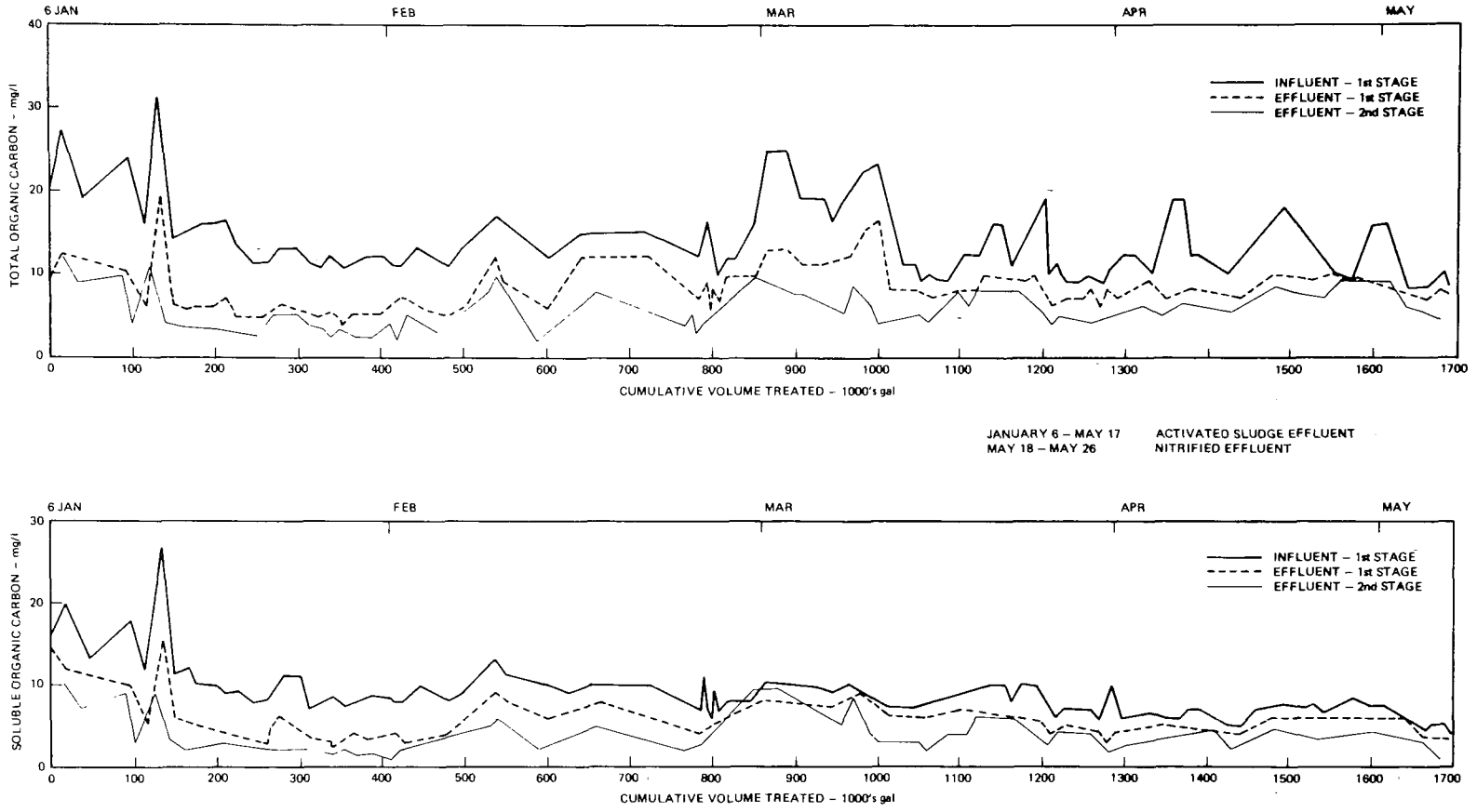

Figure 4-4. Performance Data of Activated Carbon Treatment of Activated Sludge and Nitrification Process Effluents

and SOC were nearly the same as the influent concentrations. On May 25, 1971, the first-stage column was replaced with fresh carbon and re-installed as the second stage of treatment.

Approximately 1.7×10^6 gallons of filtered activated sludge effluent were treated by activated carbon before the first stage approached exhaustion. At this point, the activated carbon removed 0.17 lb TOC/lb activated carbon in the first stage. Based on an influent TOC concentration of 14 mg/l, a 60 percent removal, and a removal of 0.17 lb TOC/lb carbon, approximately 410 lb of activated carbon would be required for each million gallons of water treated.

Average performance data for the carbon units are summarized in Table 4-3. The first-stage carbon unit removed approximately 40

Table 4-3

SUMMARY OF ACTIVATED CARBON PERFORMANCE DATA DURING TREATMENT OF FILTERED ACTIVATED SLUDGE EFFLUENT

Item		First Stage Organic Carbon		Second Stage Organic Carbon		Overall Carbon Removal	
		Total	Soluble	Total	Soluble	Total	Soluble
Average Influent	mg/l	14.0	8.6	8.3	5.8		
Average Effluent	mg/l	8.3	5.8	5.7	3.6		
Total Pounds Applied	lbs	192	120	116	81	192	120
Total Pounds Removed	lbs	77	40	36	30	111	69
Removals	$\frac{\text{lbs OC removed}}{\text{lbs act. carbon}}$	0.17	0.07	0.081*	0.068		
Average Removal	percent	40	33	31	37	59	58
Average Flow	gpm/sq ft	1.4		14			
Total Throughput Volume	gal	1.7×10^6		17			
Average Empty Bed Contact Time	min	11.2		11.2			

*Carbon adsorption was terminated before breakthrough of the activated carbon column.

Operation of the first stage: January 6 - May 25, 1971

Activated carbon bed repacked: May 25, 1971

and 33 percent of the influent TOC and SOC, respectively, whereas the second stage removed 31 and 37 percent of the remaining TOC and SOC. Removal percentages of the organic carbon were nearly identical in each stage of carbon treatment, indicating that the organic carbon which passed through the first stage was readily adsorbed by the second carbon column. The combined two-stage activated carbon treatment process achieved a 59 percent reduction in the organic carbon concentration of the activated sludge effluent during the five months of operation. These removals, combined with those resulting from the activated sludge process resulted in an overall TOC removal of 93 percent, 82 percent of which occurred in the conventional activated sludge stage. Although the activated carbon treatment removed almost 60 percent of the organic carbon in the activated sludge effluent, this only amounted to about 7.5 percent of the TOC in the raw wastewater. This small but significant increase in effluent quality resulted in an appreciable increase in the complexity and expense of the treatment process. Thus, in cases where biological systems are able to operate at high efficiencies, it may not be necessary to utilize activated carbon treatment unless very high quality effluents are required.

PHASE II: BIOLOGICAL NITRIFICATION-DENITRIFICATION

Phase II of this study started on May 20, 1971, at which time activated sludge effluent from the CCCSD 1-mgd plant was used as the influent to the pilot plant nitrification reactor. The average influent flow rate into the treatment process was approximately 17 gpm, resulting in a four-hour detention time in the nitrification basin followed by two hours of sedimentation. The settled nitrified effluent was then fed into a hydraulically mixed reactor where methanol was added in a 3:1 methanol: nitrate-N weight ratio for biological denitrification under anaerobic

conditions. Flow in this basin averaged 10 gpm initially for a detention time of about three hours and a settling time of two and a half hours. This overall operation resulted in a three-stage biological system which provided carbonaceous BOD removal in the activated sludge, ammonia oxidation in the nitrification basin, and nitrate removal in the denitrification basin.

The main emphasis during this phase of the work was on the nitrification and denitrification studies. However, additional investigations were conducted using the physical treatment processes of filtration and activated carbon adsorption. Nitrified effluent was fed to the filtration and activated carbon units from May 20 until June 30. From July 1 until project complation on November 4, the denitrified effluent was filtrated and treated with activated carbon.

Nitrification

The influent and effluent ammonia concentrations, nitrification mixed liquor suspended solids (MLSS) concentrations, and the ammonia removal rates are presented in Figure 4-5.

The ammonia content in the nitrification process influent was usually 10 to 20 mg/l; however, at times, lower ammonia values were observed. These lower values resulted from varying degrees of nitrification occurring in the activated sludge stage preceding the nitrification stage. Often, the ammonia concentration in the nitrification-stage effluent was less than 1 mg N/l. Ammonia concentrations greater than this value generally corresponded to periods when the MLSS in the nitrification reactor were significantly reduced in concentration.

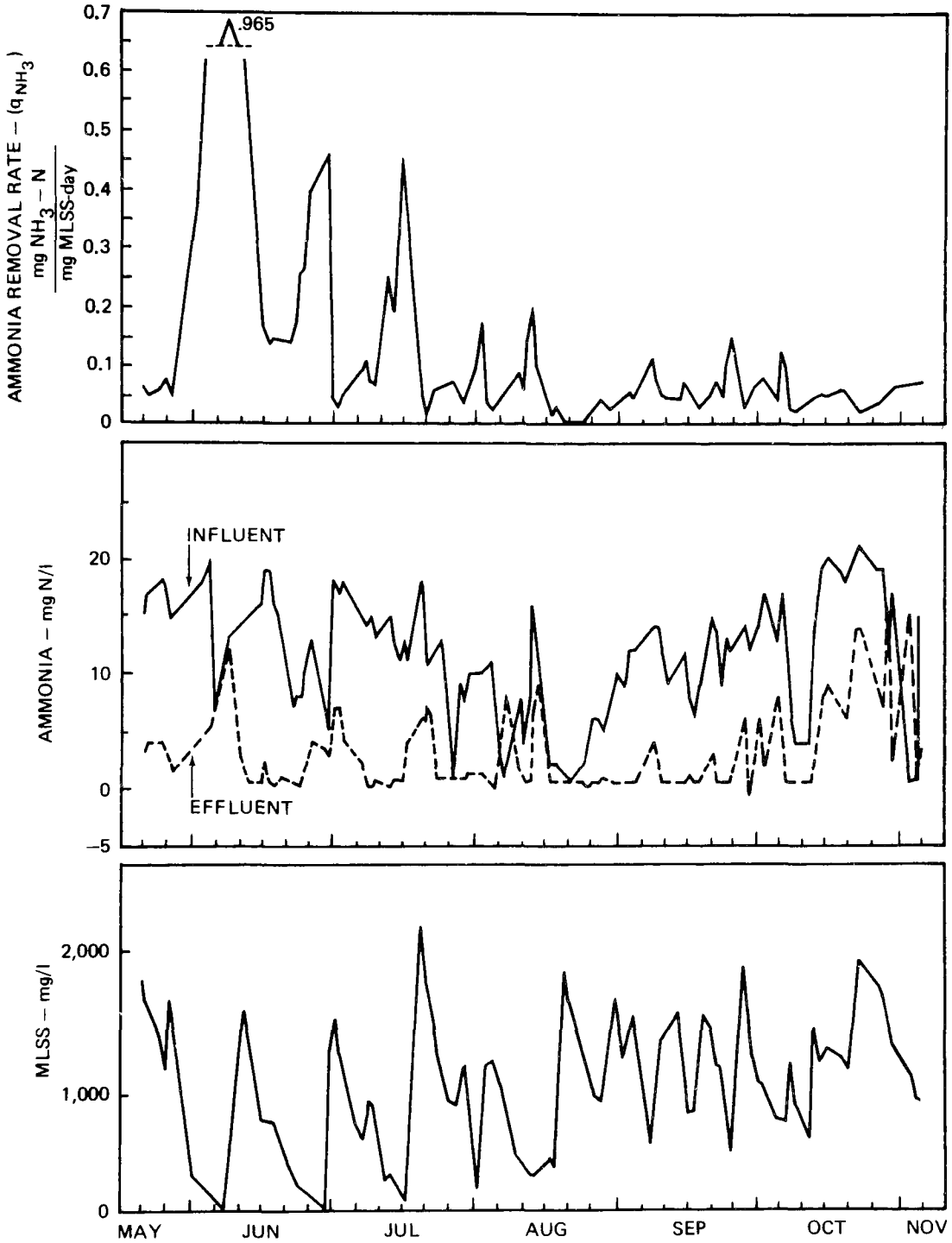

Figure 4-5. Nitrification Process Data

During about the first three months of operation of the nitrification stage, as shown in Figure 4-5, the MLSS concentration was quite variable. The sudden increases in MLSS resulted from the addition of settled activated sludge solids from the activated sludge mixed liquor. Following each addition of solids, however, the nitrification MLSS decreased with time until the nitrification efficiency decreased, and, consequently, the effluent ammonia concentration increased.

During the latter three months of operation, activated sludge solids were added to the nitrification reactor at more frequent intervals so that the MLSS did not decrease below about 500 to 1000 mg/l. The effluent ammonia concentrations observed during this period were consistently quite low. However, in October, the effluent ammonia concentrations increased to values around 10 mg/l. For about one week prior to this period, the influent ammonia concentrations to the nitrification stage were quite low since the ammonia was being oxidized in the activated sludge stage. It is possible that because of the low influent ammonia, the viability of the nitrifying culture decreased resulting in the reduction of nitrification efficiency.

The ammonia removal rates, expressed as the mass of ammonia removed per unit time per unit mass of suspended solids in the nitrification reactor (Figure 4-5), were initially quite variable as a result of the changing MLSS concentrations. These rates, however, were more stable during the latter two months of operation. The data indicate that, with an ammonia removal rate of 0.05 mg NH_3-N/mg MLSS-day, a MLSS of 3000 mg/l, and 20 mg/l of ammonia oxidized, a detention time of 3.2 hours would be required in the nitrification reactor. It should be noted that this ammonia removal rate is based on pH values

of about 7.0. If somewhat higher pH values of around 8.0 to 8.5 could be maintained in the nitrification reactor, then a higher ammonia removal rate would be expected, providing the potential of reducing the required detention time.

The results obtained during this study suggest that high removal efficiencies of organic materials and suspended solids in the first biological stage preceding the nitrification stage are not required, and indeed are not desireable. If relatively high removal efficiencies are obtained in the first biological stage, then, at times, some nitrification may occur which could be detrimental to the nitrifying culture in the second stage. Also, in order to maintain a desireable MLSS concentration in the nitrification reactor, the data indicate that it is necessary to have some carbonaceous oxidation in the nitrification reactor in conjunction with the oxidation of ammonia to nitrate, thus establishing a synergistic effect between the heterotrophic and autotrophic microorganisms.

The average and range of suspended solids observed monthly in the CCCSD 1-mgd activated sludge effluent and the effluents from the pilot plant nitrification and denitrification stages are summarized in Table 4-4. The TOC and SOC concentrations for these effluents are illustrated in Figure 4-6. Suspended solids in the activated sludge effluent averaged 9 mg/l less than observed in the nitrified effluent. The decrease in nitrified MLSS discussed earlier can be largely attributed to this increase in solids concentration.

In the denitrified effluent, the suspended solids averaged approximately 20 mg/l higher than the nitrified effluent. This increase resulted from problems encountered with the denitrification stage MLSS floating in the sedimentation tank. At times, a layer of solids several inches thick would accumulate on the surface of the sedimentation tank. The floating nature of these solids is discussed in more detail later.

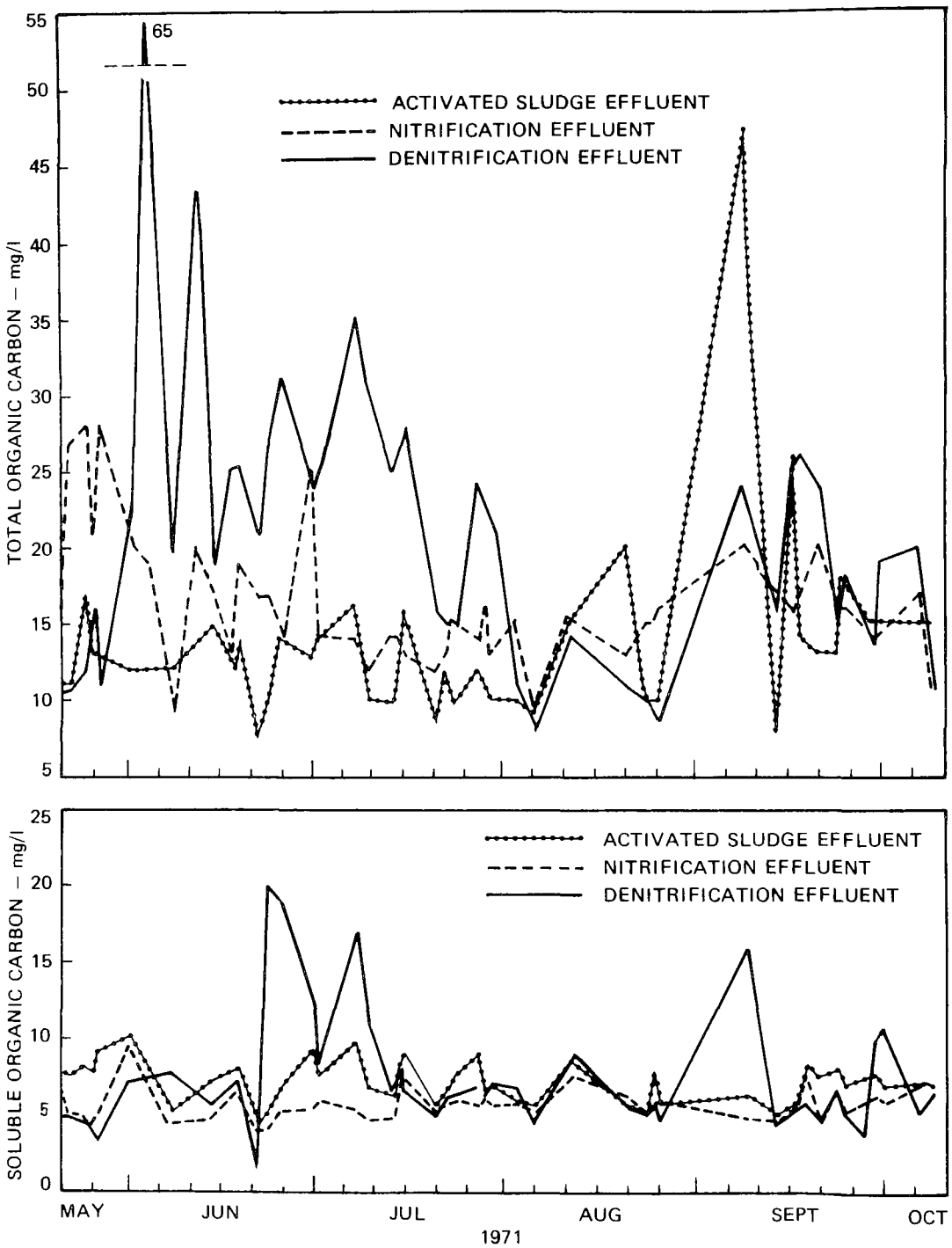

Figure 4-6. Nitrification-Denitrification Process Data

Table 4-4

AVERAGE MONTHLY SUSPENDED SOLIDS CONCENTRATIONS OF THE BIOLOGICAL EFFLUENTS (mg/l)

Month	Activated Sludge*		Nitrification		Denitrification	
	Average	Range	Average	Range	Average	Range
June	10	8-13	34	22-42	45	30-70
July	7	2-10	23	12-50	35	21-62
August	6	2-12	18	8-26	25	5-37
September	14	4-50	19	11-37	56	33-95
October	29	6-87	25	8-56	61	13-116
Average	14		23		42	

*CCCSD 1 mgd plant

Effluent TOC concentrations, as shown in Figure 4-6, averaged 14 mg/l in the activated sludge effluent and 15 mg/l in the nitrified effluent. In the denitrified effluent, the TOC averaged 20 mg/l with this higher value, compared to the nitrified effluent, resulting primarily from the loss of floating solids mentioned above.

The SOC concentrations shown in Figure 4-6 for the activated sludge and nitrification-stage effluents indicate slightly lower values for the latter. Generally, the denitrified effluent SOC concentration was nearly the same as the nitrified effluent except for three peak concentrations. These peaks probably resulted from the methyl alcohol which was not completely metabolized in the denitrification reactor.

Denitrification

Nitrate removal rates expressed as the mass of nitrate-nitrogen removed per unit time per unit mass of suspended solids in the denitrification reactor, influent and effluent nitrate-nitrogen concentrations, and denitrification MLSS concentrations are presented in Figure 4-7.

The nitrate removal rates were quite variable during the first two months of operation, but, during the latter three months, the rates were more stable. The removal rates were quite high during the month of July, primarily as a result of low concentrations of MLSS (Figure 4-7). Very good removals were observed at rates up to about 0.15 mg nitrate removed/mg MLSS-day, indicating that the detention time in the denitrification reactor could be as low as approximately one hour. The residence time in the denitrification reactor may, however, need to be longer in order to assure that sufficient time is available for the denitrification reaction to be completed, thus minimizing any additional denitrification in the final sedimentation tank.

During the pilot plant denitrification studies, problems were encountered with poor settling of the denitrification MLSS. Apparently, either the denitrifying organisms were continuing to produce nitrogen gas in the sedimentation basins or the suspended solids from the denitrification basin were carrying entrained nitrogen gas bubbles into the sedimentation basin, thereby buoying up the settled solids and causing them to float on the surface of the sedimentation tank. The inlet to the sedimentation tank was modified so the mixed liquor would be exposed

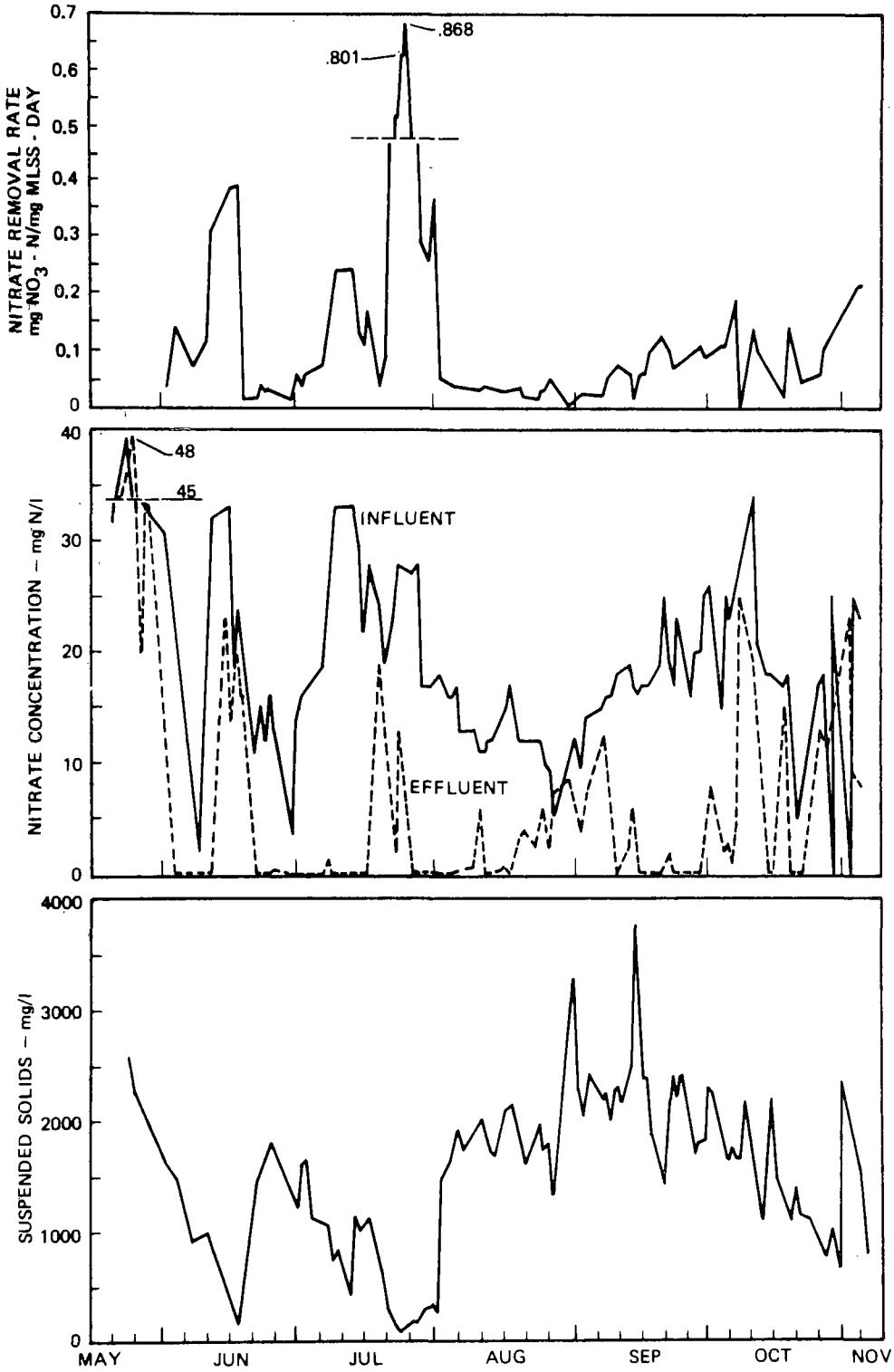

Figure 4-7. Denitrification Process Data

to atmospheric pressure for a short time before entering the settling zone. This modification was employed to release the nitrogen gas entrapped in the biological floc. Some reduction in suspended solids losses was achieved by this technique, but the problem was not eliminated. Air agitation for 3 to 5 minutes in the center of the sedimentation tank inlet was also tested as a means of stripping out excess nitrogen gas and methanol and to provide a residual DO in the sedimentation tank. Since there was no apparent improvement in the effluent quality, this procedure was discontinued. Recent information, from the EPA District of Columbia pilot plant, indicates that aeration of the mixed liquor for about 30 minutes prior to settling reduces the solids separation problems and, at the same time, reduces the excess methanol carried over from the denitrification reaction. Similarly, results obtained from the CCCSD plant, which was modified to further investigate the problems with denitrification discussed above at a scale of about 0.5 mgd, indicate that aeration of the denitrification mixed liquor for approximately one hour prior to sedimentation significantly improves solids separation and effluent quality.

The effluent nitrate concentrations, as shown in Figure 4-7, were quite variable. However, the rather high nitrate concentrations observed during part of July resulted from problems encountered with the methanol feed pump. The effluent nitrate increases observed in August and the first part of September are believed to have resulted from the ferric chloride additions being made to the denitrification reactor during this time, which caused a decrease in the pH of the mixed liquor. When all plant functions were properly operating and the denitrification MLSS concentration was sufficiently high, effluent nitrate-nitrogen concentrations of 1 mg/l or less could be maintained.

Although the denitrification results obtained during this study generally placed doubt on the adequacy of this process, enough good results were

obtained to indicate that, with additional time and development, the process can be made to perform satisfactorily. The results from the EPA District of Columbia pilot plant and the CCCSD "Advanced Treatment Test Facility," reported after the data in this report were obtained, illustrate that the denitrification process can be designed to perform satisfactorily. Perhaps the most significant finding in these more recent investigations was the required aeration of the denitrification MLSS for 30 to 60 minutes prior to final sedimentation.

Phosphorus Removal

Ferric chloride and alum were added during different periods of time to the denitrification system to observe the effect on phosphorus removal. Table 4-5 is a summary of the results.

Ferric chloride was added in three different ratios of iron to phosphorus. Removals of phosphorus for Fe/P weight ratios of 1.9 and 5.6 were 57 percent and 63 percent, respectively. Results during the testing period from June 16 to June 24 indicated only 43 percent phosphorus removal at an Fe/P ratio of 2.5. This lower removal resulted in part from problems encountered with the chemical feeding system. Also, the addition of ferric chloride periodically reduced the pH of the denitrification MLSS to the extent that the denitrification reaction was inhibited.

Alum additions during periods in September and October were maintained at an aluminum-to-phosphorus weight ratio of about 1.2 to 1.3. These ratios resulted in average phosphorus removals of 52 percent across the denitrification system. Based on these results, it appears that alum would be preferred over ferric chloride for the

removal of phosphorus in combination with biological treatment. However, to obtain a high degree of removal, multiple addition points would be required.

Table 4-5

PHOSPHOROUS REMOVAL USING FERRIC AND ALUMINUM ADDITIONS TO THE DENITRIFICATION SYSTEM

Date	Dosage (Weight Ratio)		Effluent mg/l as P	Removal %
	Fe/P	Al/P		
6/16 6/24	2.5	—	5.2	43
7/30 8/20	1.9	—	4.2	57
8/21 - 9/2	5.6	—	3.6	63
9/3 - 9/17	—	1.2	4.2	55
9/20 - 9/27	—	1.3	4.2	52
9/28 - 10/26	—	1.2	4.9	50

Filtration

The filtration process data shown in Figure 4-8 for filtration rates of 5.0 gpm/sq ft indicate that, during the time that nitrified effluent was being filtered, there was a very good removal of the suspended organic materials. Effluent turbidities were quite low and showed only minor variations over the six weeks of operation. Influent turbidities ranged from 2 to 30 JTU, averaging about 14 JTU; whereas effluent turbidities averaged about 2 JTU, for a mean turbidity removal of 85 percent. TOC removals averaged 70 percent through the filtration process resulting in an effluent concentration of about 6 to 7 mg/l. Apparently, the effluent suspended solids in the nitrified effluent formed a strong floc with filterability similar to typical activated sludge floc.

The quality of the filtered, denitrified effluent was considerably more variable than that of the nitrified effluent. In July and early August, when the influent turbidity was relatively low, there was very little removal of turbidity occurring with Filter No. 2. Removals were somewhat better in late August and September, when Filter No. 1 was in operation. This may have been due to the higher solids loading during this period, since turbidity removals of Filter No. 2 appeared to improve somewhat between August 5 and 15 as the loading increased. Effluent turbidities from Filter No. 3 appeared to be consistently lower than observed with Filter Nos. 1 and 2. Table 4-6 summarizes the average removals for the various filters employed.

The highly variable turbidities of the filter influent attest to the difficulty of attaining consistent solids removals in the denitrification sedimentation tank. The lower quality of the filtered, denitrified effluent may have been due to the nature of the floc. It appears from the filtration

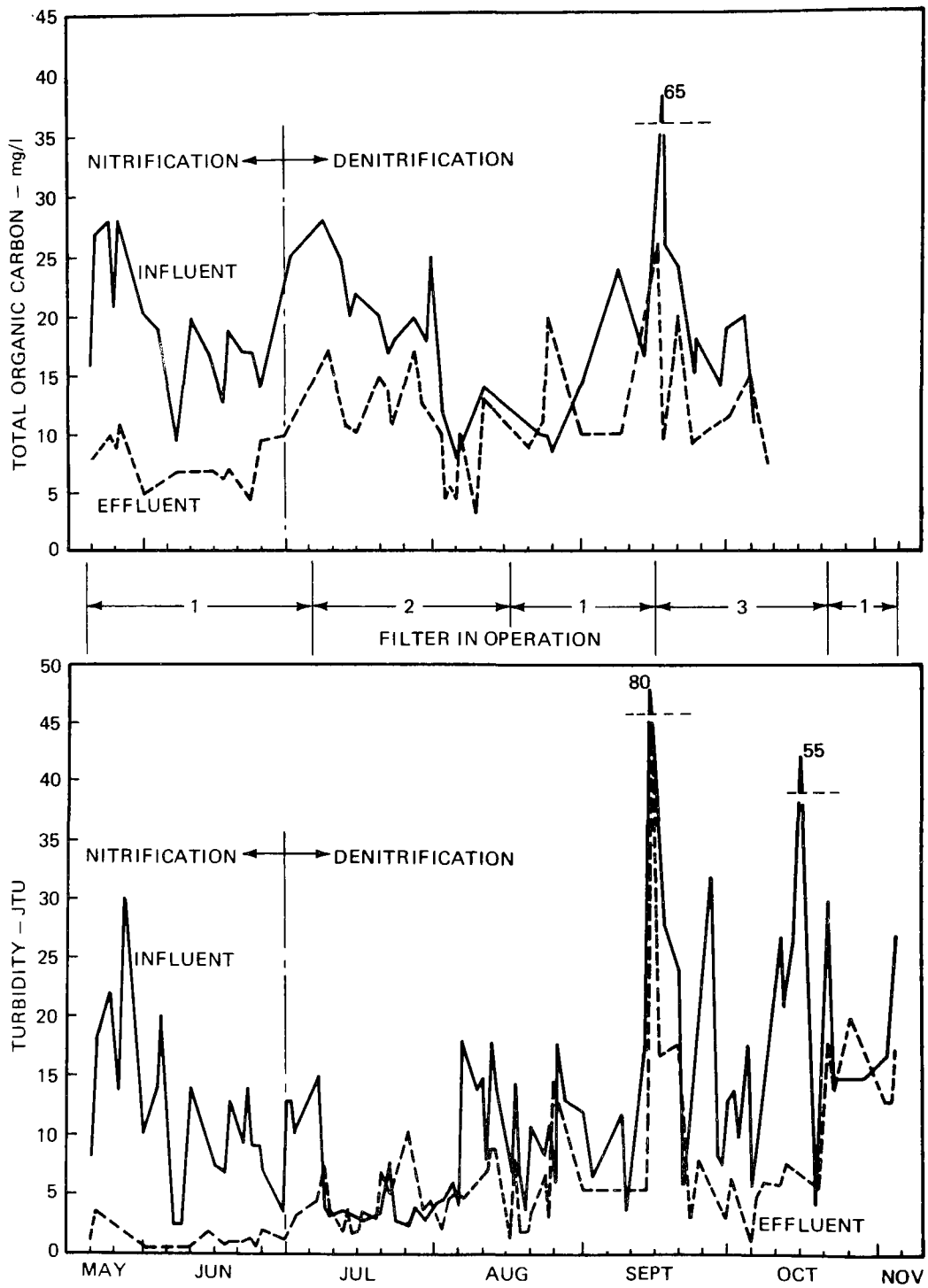

Figure 4-8. Filtration Data Using Denitrification Process Effluent

Table 4-6

SUMMARY OF FILTRATION PERFORMANCE USING DENITRIFICATION PROCESS EFFLUENT

Filter	Average Percent Removals	
	TOC	Turbidity JTU
No. 1 (Dual Media)		
20 Percent Sand 80 Percent Anthracite	58* 37	84* 53
No. 2 (Dual Media)		
45 Percent Sand 55 Percent Anthracite	27	37
No. 3 (Mixed Media)		
25 Percent Garnet 30 Percent Sand 45 Percent Anthracite	38	61

* Nitrified Effluent

data that this floc was more fragile than that developed in the first two biological stages. If this is the case, it may be necessary to reduce the filtration rate when filtering denitrified effluent. Also, aeration of the denitrification MLSS prior to sedimentation, as discussed earlier, may improve the filterability; however, this was not studied.

Activated Carbon

The results of the two-stage carbon adsorption studies for the Phase II pilot plant are shown in Figure 4-9. Nitrified effluent was filtered and passed through the columns from May 20 until June 30, 1971. From July 1 until November 4, the denitrified effluent was used as the influent to the activated carbon adsorption system. The 1.7 million gallons which the first-stage carbon column had been exposed to at the start of the study

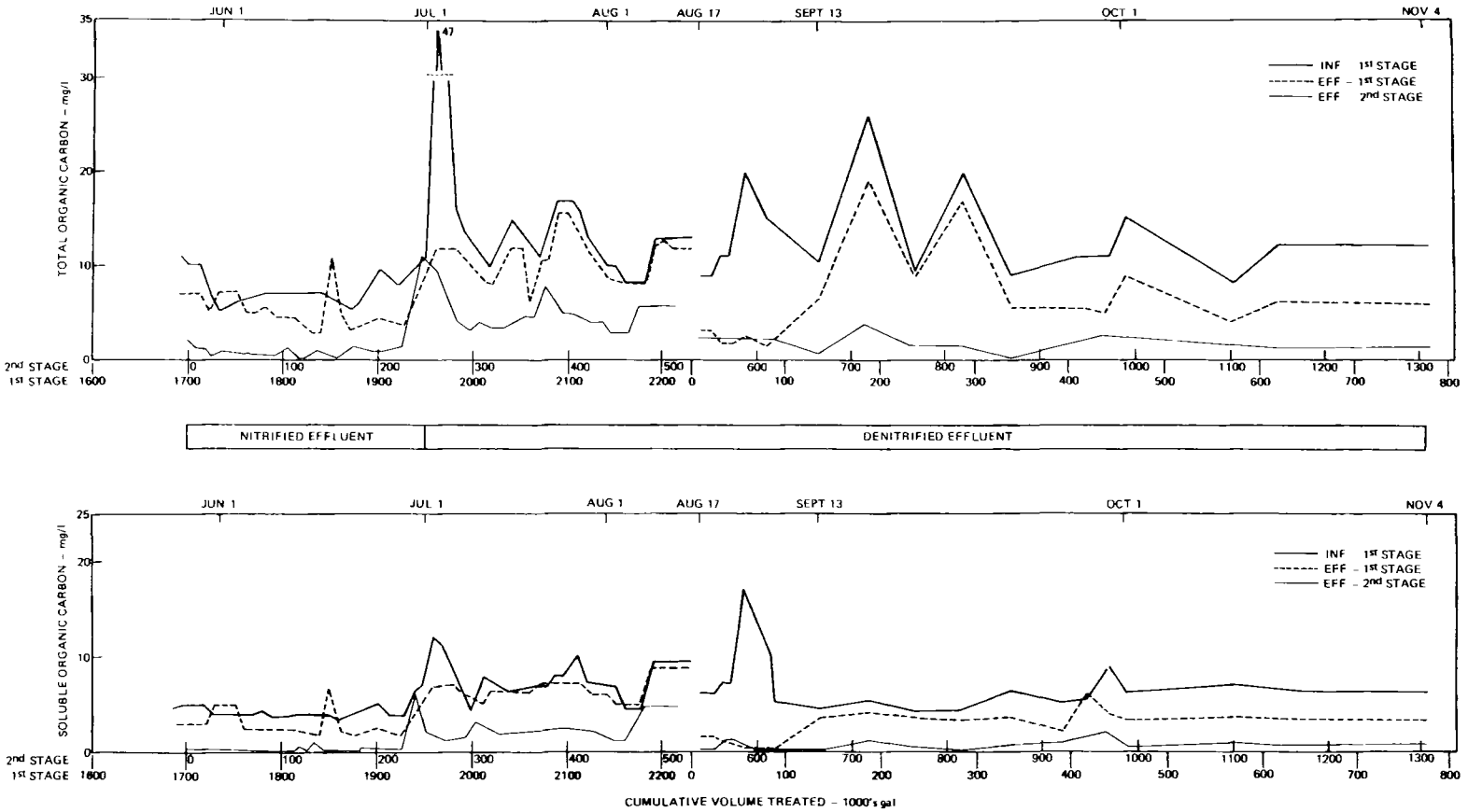

Figure 4-9. Performance Data of Activated Carbon Treatment of Nitrification and Denitrification Process Effluents

represents the amount of water treated during Phase I, when this column acted as the second-stage for the activated sludge effluent. The new second-stage column was freshly repacked activated carbon.

The TOC concentration in the first-stage effluent as shown in Figure 4-9 was generally 2 to 4 mg/l less than the influent TOC, representing about a 30 percent reduction in the TOC concentration. The very high TOC concentrations resulting when the denitrified effluents was fed to the carbon columns were due to suspended solids from the denitrification process. However, these materials were effectively removed in the carbon column. The second stage of the activated carbon system was operating more effectively than the first during this period, as is evident from Figure 4-9. This column was generally removing 3 to 5 mg/l of TOC, or about 50 percent of the influent TOC.

SOC concentrations in the activated carbon influent were usually between 5 and 10 mg/l, while the second-stage effluent was generally between 1 and 2 mg/l. This reduction represented approximately 80 percent removal of the SOC through the activated carbon process.

During much of July and early August, the organic carbon content of the first-stage effluent was nearly the same as the influent. On August 17, the first-stage was removed from service after treating approximately 2.2 million gallons, repacked with fresh activated carbon and reinserted as the second stage of the carbon treatment system. The former second stage was installed as the first stage. (Note in Figure 4-9 the discontinuity in the abscissa on August 17, when the treated volume of the first stage changes from 2.2 million gallons to about 500,000 gallons, signifying the interchange of columns). Removal efficiencies improved appreciably. Except for two high TOC concentrations in the first-stage effluent in late September, each column appeared to remove about the same amount of TOC and SOC, resulting in a treated effluent having a TOC concentration of 2 to 3 mg/l.

The activated carbon adsorptive capacity was determined from the time fresh carbon was initially contacted with the influent until the time the carbon was exhausted. This was done by calculating the masses of TOC and SOC removed and relating them to the mass of activated carbon contacted. A summary of the data is shown in Table 4-7 for the period between January 6 and August 16, 1971. The average removal efficiency during this period was 30 percent and the average carbon loading, through exhaustion, was 0.11 lb total organic carbon removed per pound of activated carbon.

Also shown in Table 4-7 are performance data for the activated carbon column that served as the second stage in the process from May 26 to August 17 and then the first stage from August 17 through October 31, 1971. The data, as shown in Figure 4-9, indicate that this carbon column was not completely exhausted through the end of October. The average TOC removal efficiency for this column was 50 percent and the carbon loading was 0.13 lb TOC removed per pound of activated carbon. Since the carbon was not exhausted, this removal efficiency was greater than observed with the column operated between January 6 and August 16. However, the carbon loading on the column operated from May 26 through October 31, without being exhausted, was actually higher than that of the previous observations. This difference in carbon loading was due to the differences in the influent suspended TOC concentrations. The activated sludge and nitrification process effluents, which were readily filtered, resulted in lower TOC concentrations than the denitrification process effluent.

TOC removals in the activated carbon columns generally ranged between 6 and 12 mg/l. Based on an average TOC concentration of about 80 mg/l in the treatment plant influent, the activated carbon system removed only

Table 4-7

SUMMARY OF ACTIVATED CARBON PERFORMANCE DATA DURING TREATMENT OF THE FILTERED NITRIFICATION AND DENITRIFICATION PROCESS EFFLUENTS

		Organic Carbon	
		Total	Soluble
Jan 6 - Aug 16, 1971			
Total Applied	lbs	166	109
Total Removed	lbs	49	36
Average Removal	percent	30	33
Carbon Loading	$\frac{\text{lbs TOC removed}}{\text{lbs act. carbon}}$	0.11	0.08
Average Flow	gpm/sq ft	0.7	
Detention Time	min	27	
May 26 - Oct 31, 1971			
Total Applied	lbs	112	63
Total Removed	lbs	56	38
Average Removal	percent	50	60
Carbon Loading	$\frac{\text{lbs TOC removed}}{\text{lbs act. carbon}}$	0.13	0.086
Average Flow	gpm/sq ft	0.9	
Detention Time	min	21	

about 10 percent of the organic materials. Thus, although the activated carbon treatment resulted in very low effluent organic carbon concentrations, the preceding biological treatment processes were responsible for the removal of the bulk of the organic materials.

PHASE III: CHEMICAL-PHYSICAL TREATMENT SYSTEM

The two-stage chemical treatment system which began operation in late April and continued until November consisted of lime coagulation and settling in the first stage followed by recarbonation and settling of the calcium carbonate precipitate in the second stage. The effluent from this treatment process was then filtered and treated with activated carbon.

Lime Treatment

Preliminary laboratory data were obtained on the chemical treatment of wastewater to determine the approximate operating pH to use in the pilot plant studies. As shown in Figure 4-10, a pH of 10.5 to 11.0 appeared to be near the optimum value with regard to minimizing the lime requirements and obtaining appreciable reductions in TOC, phosphorus, and turbidity. Approximately 350 to 500 mg/l of lime (as CaO) were required to maintain a pH of 10.5 to 11.0, while only 250 mg/l of lime were required to obtain an operating pH of 10.0.

The lime dosage requirements, and the operating influent and effluent pH values for the chemical treatment system are shown in Figure 4-11. The amount of lime used in the chemical process was controlled by a pH feedback controller to maintain a predetermined pH value. Daily lime requirements ranged from 200 to 500 mg/l CaO, averaging about 390 mg/l. From mid-September until the end of November, the lime dose was about

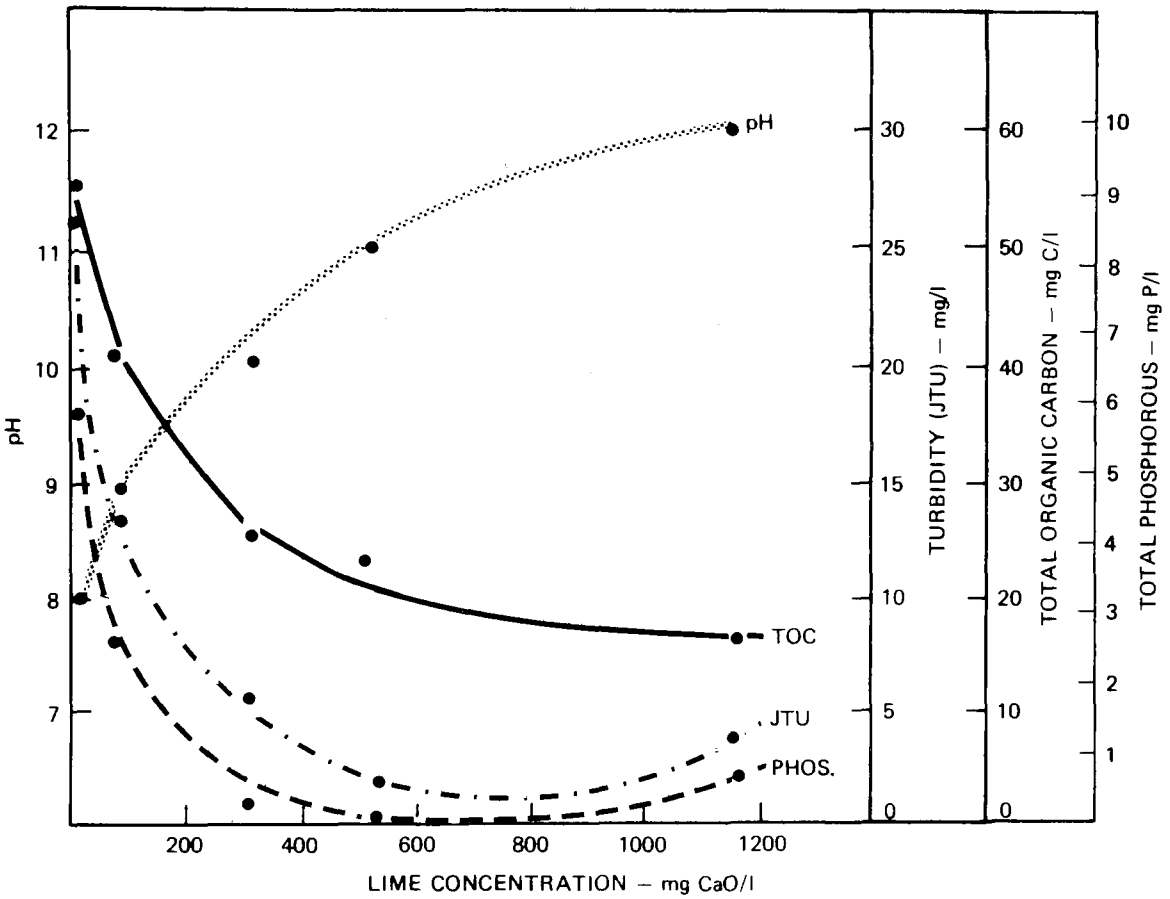

Figure 4-10. Lime Requirements for Chemical Treatment

450 mg/l, resulting in an operating pH of about 11.0. Initial studies indicated little difference in the quality of the treated effluent between flocculation times of 30 and 20 minutes, so the shorter flocculation time was adopted throughout the study. After recarbonation and second stage flocculation and sedimentation, the pH of the lime treated effluent was about 9.5. It was observed that the dispersed nature of the calcium carbonate precipitate formed after recarbonation made it difficult to separate by gravity settling. The addition of ferric chloride to coagulate the dispersed material resulted in better separation, but light flocculant materials

were observed in the effluent. By returning a portion of the calcium carbonate sludge to the second-stage flocculator, the effluent quality was improved, most likely as a result of improved particle growth. However, recycling of lime sludge to the first-stage flocculator did not appear to significantly improve the performance of that stage.

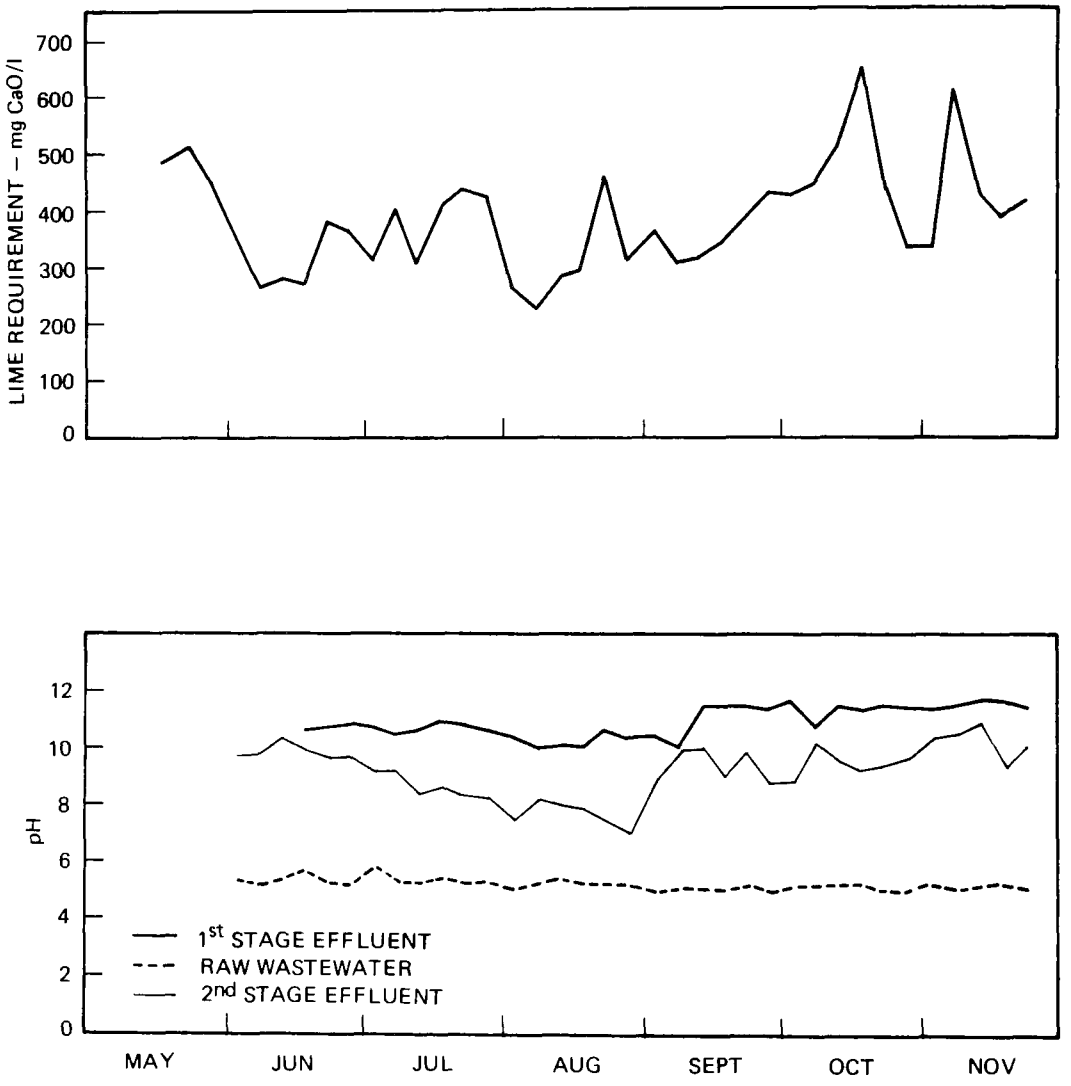

Figure 4-11. Chemical Treatment Plant Lime Requirements and pH Values

Hardness and Alkalinity

The hardness and alkalinity data for the chemical treatment system are shown in Figure 4-12. Average influent and effluent data are summarized in Table 4-8. Generally, the alkalinity of the second stage effluent was less than in the raw wastewater, its decrease averaged 18 mg/l as $CaCO_3$ or 9.1 percent. The total hardness of the treated effluent was not significantly changed. An average increase in hardness of 6 mg/l as $CaCO_3$ or 3.3 percent was observed.

Table 4-8

CHEMICAL TREATMENT AVERAGE HARDNESS AND ALKALINITY DATA

	Alkalinity	Hardness
First Stage Influent, mg/l	198	152
Second Stage Effluent, mg/l	179	158
Concentration Change, mg/l	18	+ 6
Concentration Change (%)	9.1 (Decrease)	3.3 (Increase)

Concentrations expressed as $CaCO_3$

The increase in hardness most likely resulted from incomplete precipitation of calcium carbonate following the recarbonation step. The typical chemical reactions associated with water softening were not directly applicable because of the likely interference of organic materials in the raw wastewater with the calcium. Improved performance of the chemical treatment system may have been obtained if sludge recycling was effectively used.

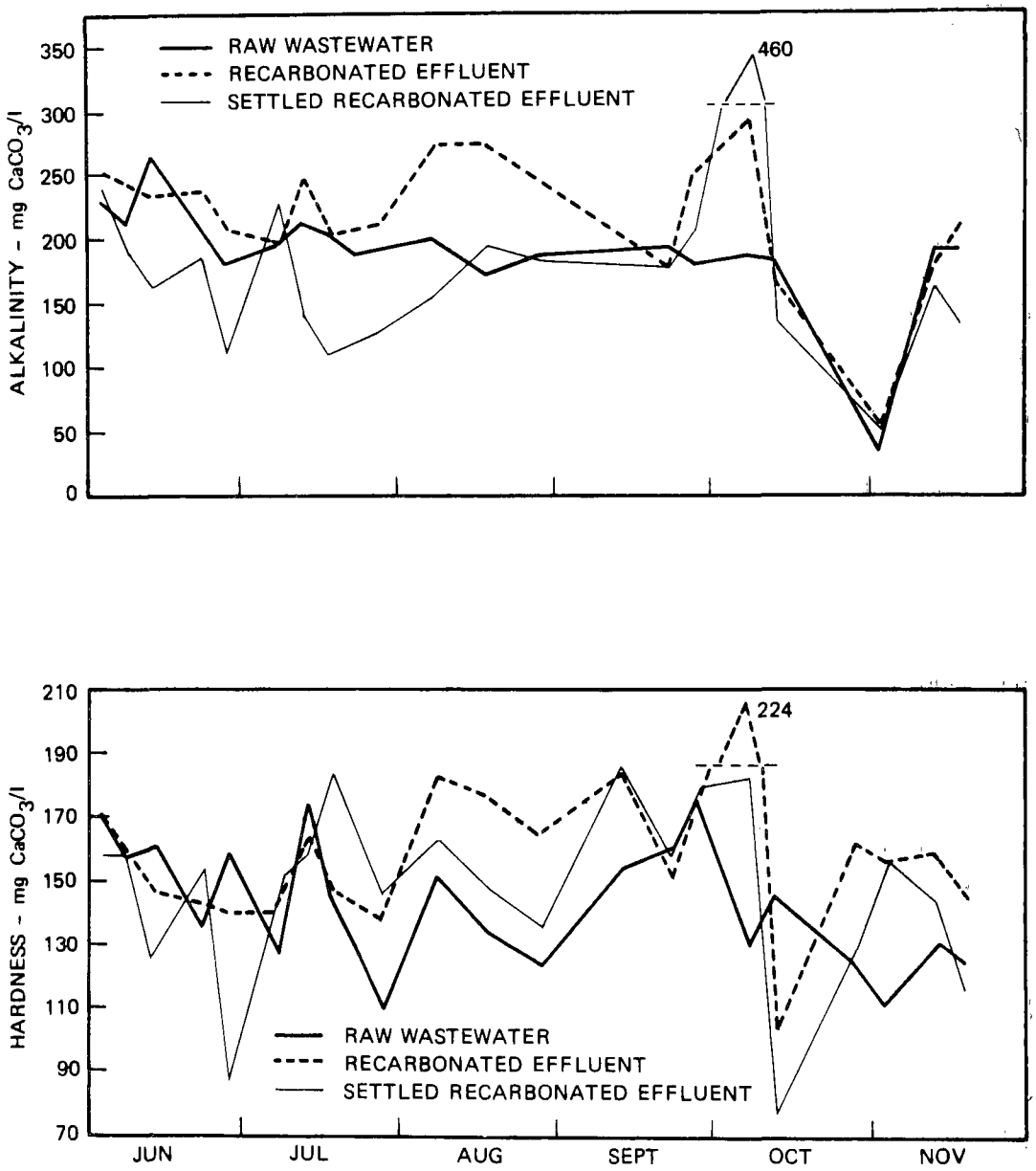

Figure 4-12. Hardness and Alkalinity of Raw Wastewater and First-Stage and Second-Stage Chemical Treatment Effluents

Suspended Solids

The suspended solids concentrations for the various stages in the chemical treatment plant are shown in the upper portion of Figure 4-13. Although the suspended solids concentration in the influent to the first-stage flocculation basin varied from about 100 mg/l to as high as 350 mg/l, the suspended solids content of the effluent from the second stage of the process was nearly constant. The variability in the suspended solids content of the raw wastewater was largely removed in the first stage of the chemical treatment process. During the 7 months of operation of the chemical treatment plant, the suspended solids removal averaged 87 percent. The chemically treated effluent suspended solids ranged from 5 to 40 mg/l, with an average concentration of 19 mg/l.

Phosphorus

The total phosphorus contents of the influent, first-stage effluent, and second-stage effluent of the chemical treatment plant are shown in the lower portion of Figure 4-13. The total phosphorus content of the influent water was generally about 7.4 mg P/l, whereas after the first stage of lime treatment, the total phosphorus was reduced to an average value of about 1.6 mg/l. This reduction represented about 80-percent removal of the total phosphorus content of the raw wastewater. The total phosphorus concentration in the effluent from the first-stage flocculation basin was higher during August due to the decrease in operating pH from 10.7 to 10.2 (Figure 4-11).

The phosphorus concentrations in the first-stage effluent were further reduced in the second stage of the chemical treatment process. Effluent from the second stage contained approximately 0.4 mg/l of total phosphorus, making the overall phosphorus reduction in the chemical treatment

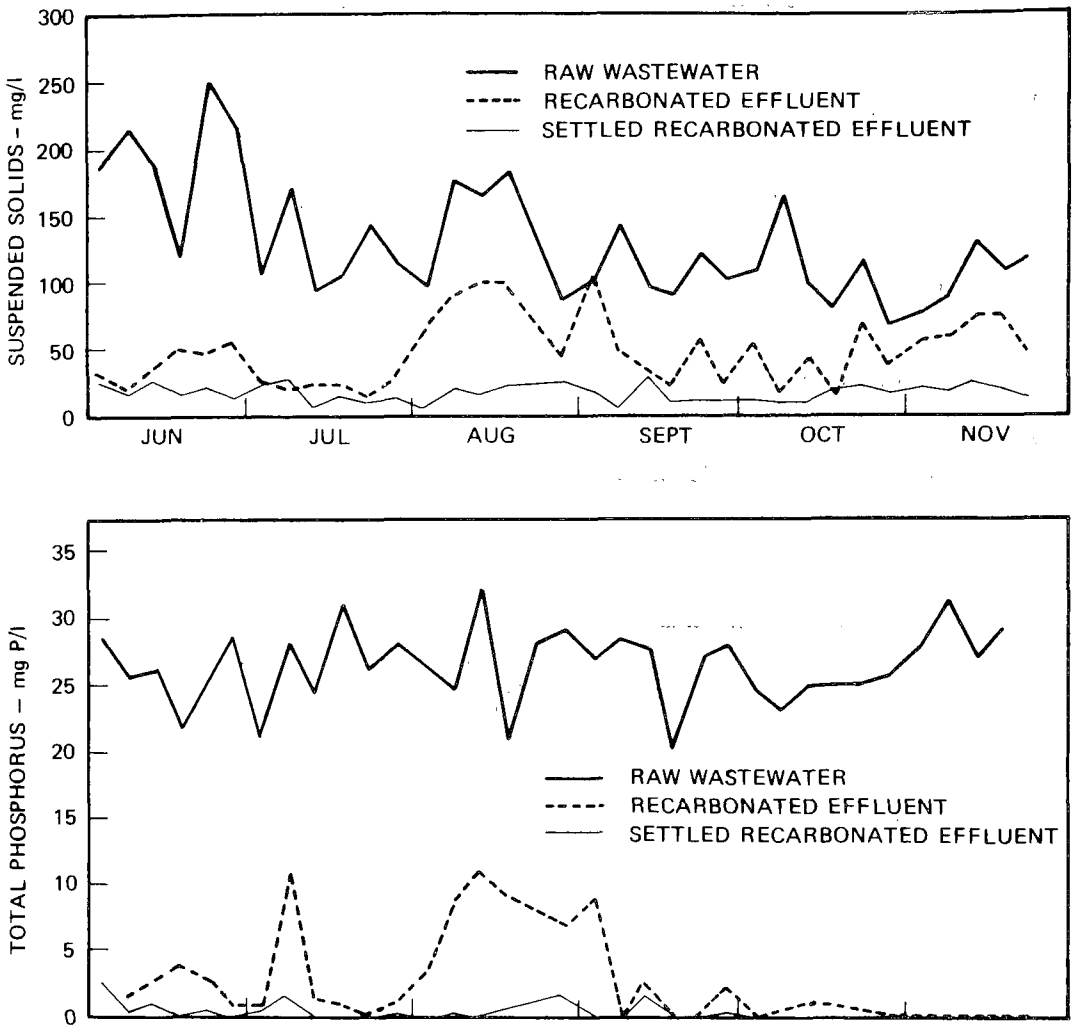

Figure 4-13. Orthophosphate and Chemical Suspended Solids Removal in the Two-Stage Chemical Treatment Process

process approximately 95 percent. During October and November, when the pH of the first stage was between 11 and 11.5, the total phosphorus concentration of the effluent was generally about 0.16 mg/l, indicating a 98-percent removal.

Organic Carbon

Figure 4-14 illustrates the TOC contents of the influent wastewater, which had been settled for about 10 minutes in the CCCSD primary sedimentation

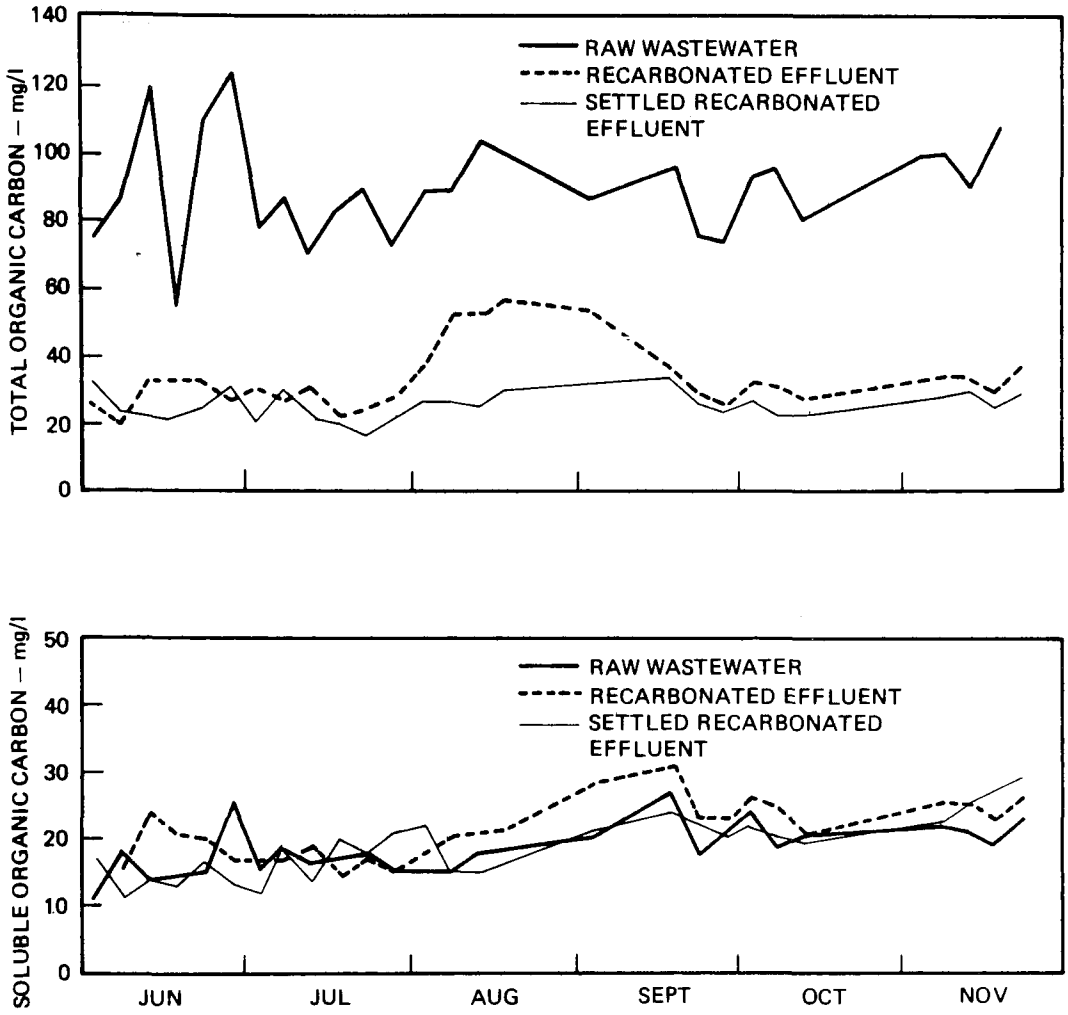

Figure 4-14. Total and Soluble Organic Carbon Removals in the Two-Stage Chemical Treatment Process

basin to remove large suspended materials and rags, the first-stage effluent, and the second-stage effluent of the chemical treatment plant. The influent TOC varied from a low of 60 mg/l to a maximum value of 125 mg/l. The average influent TOC was approximately 90 mg/l during this study, whereas the concentration of TOC in the recarbonated effluent was usually between 25 and 30 mg/l, indicating that nearly 70 percent of the TOC was removed in the chemical treatment process.

There was very little change in the SOC concentration as a result of the chemical treatment, as is apparent from the data plotted in the lower half of Figure 4-14. The SOC concentration in the effluent from the first-stage flocculation basin was usually greater than that in the influent water. Apparently, the high pH of the first stage was causing the TOC to undergo alkaline hydrolysis, converting some particulate organic carbon to soluble organic material. Some of the SOC was removed during the second-stage flocculation reaction so there was little or no net increase in SOC, and at times the SOC showed a moderate decrease through the system. Overall, SOC removals averaged about 5.0 percent for the 7-month operation of the two-stage system.

Filtration

Figure 4-15 summarizes the turbidity data from the influent and effluent of the filtration process. The settled effluent from the second stage of the chemical treatment plant was filtered at a rate of 2.5 gpm/sq ft, and the filters were backwashed at a rate of 20 gpm/sq ft.

The effluent turbidity from the chemical treatment process was generally about 5 JTU (Figure 4-15), but there were periods when much higher turbidity resulted. Filtration reduced the average turbidity by about 50 percent, producing a filtered effluent having a turbidity of 2 to 3 JTU. However, when the turbidity of the filter influent increased substantially above 5 JTU, the filters usually were able to control the turbidity, indicating that for the most part the chemical floc was readily filterable. The TOC data shown in Figure 4-15 from the filter influent and effluent indicate that there was very little removal of TOC in the filters. This was not surprising since about 70 percent of the TOC was in the soluble form (SOC). Filter No. 1 appeared to show the best performance during these tests.

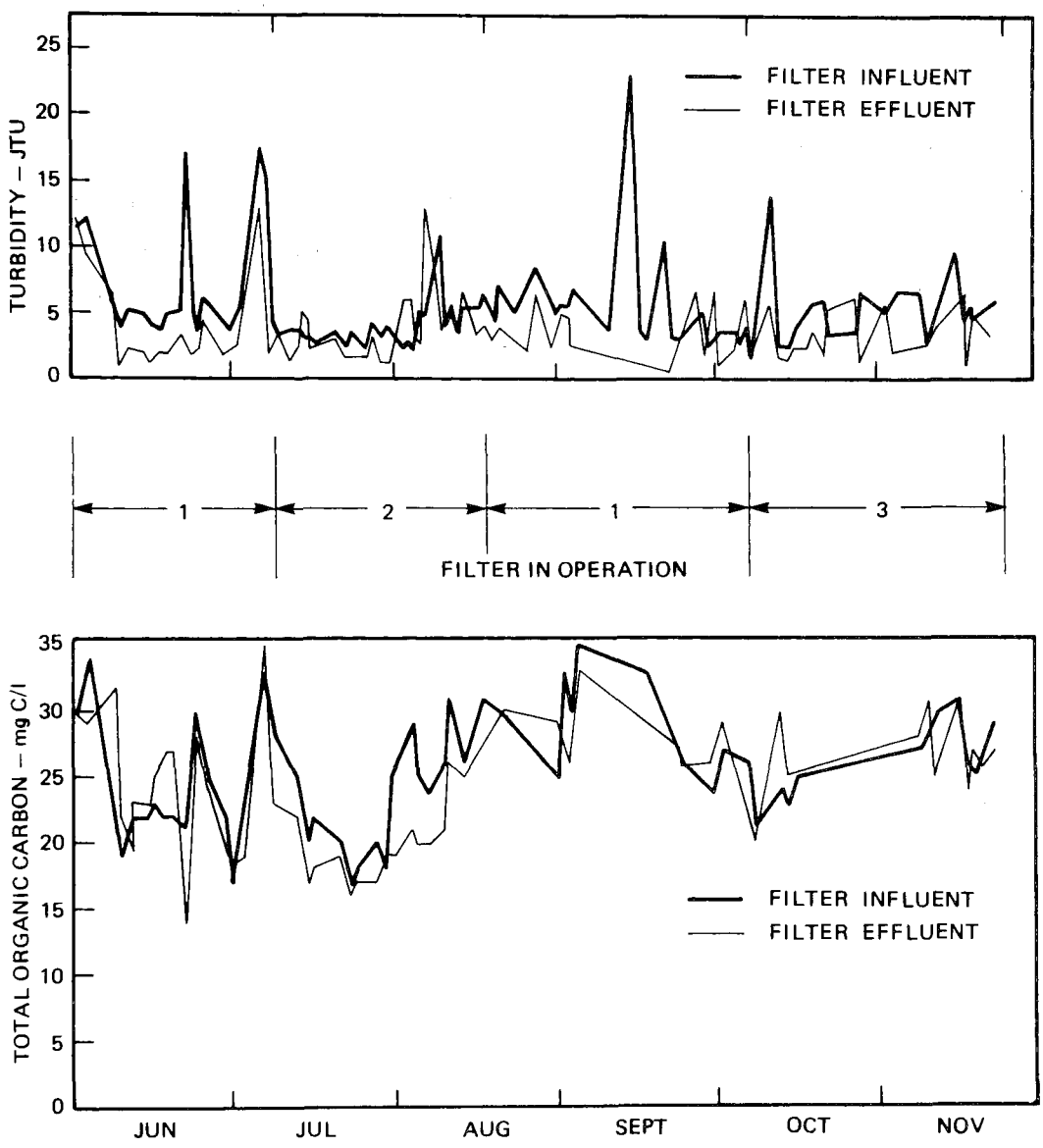

Figure 4-15. Filtration Data Using Chemical Treatment Effluent

Activated Carbon

The results of the activated carbon adsorption studies are shown in Figure 4-16. For the two stages of carbon treatment, the average empty bed contact time was about 25 minutes per stage with an average flow of 0.76 gpm/sq ft. Influent TOC concentrations averaged about 24 mg/l, whereas the first-stage effluent averaged about 13 mg/l and the second-stage about 11 mg/l. The second stage of carbon treatment appeared to have little influence on the organic carbon removal, indicating that these materials could not be readily adsorbed with activated carbon.

In early September, the effluent TOC and SOC from the carbon adsorption system began to approach the influent concentration, signifying that the columns were approaching exhaustion. Surprisingly, it was the second stage which had the greatest effluent concentration at this time, even though it had only received a very light loading of organic carbon compared to the first stage. This apparent inefficiency of the second stage may have been due to anaerobic conditions interfering with the organic carbon adsorption process. On September 9, the two carbon columns were interchanged, and the former first-stage carbon column was repacked and replaced as the second stage. After this interchange, the TOC and SOC of the effluent generally remained below 10 and 6 mg/l, respectively.

The overall performance of the activated carbon columns is summarized in Table 4-9, showing approximately 52 and 60 percent removal of TOC and SOC for the two-stage system. At the time the first stage became exhausted, 0.14 lbs of organic carbon had been removed per pound of activated carbon, and this occurred after a volume of 640,000 gallons had been treated. Based on the observed loading rates for the first stage,

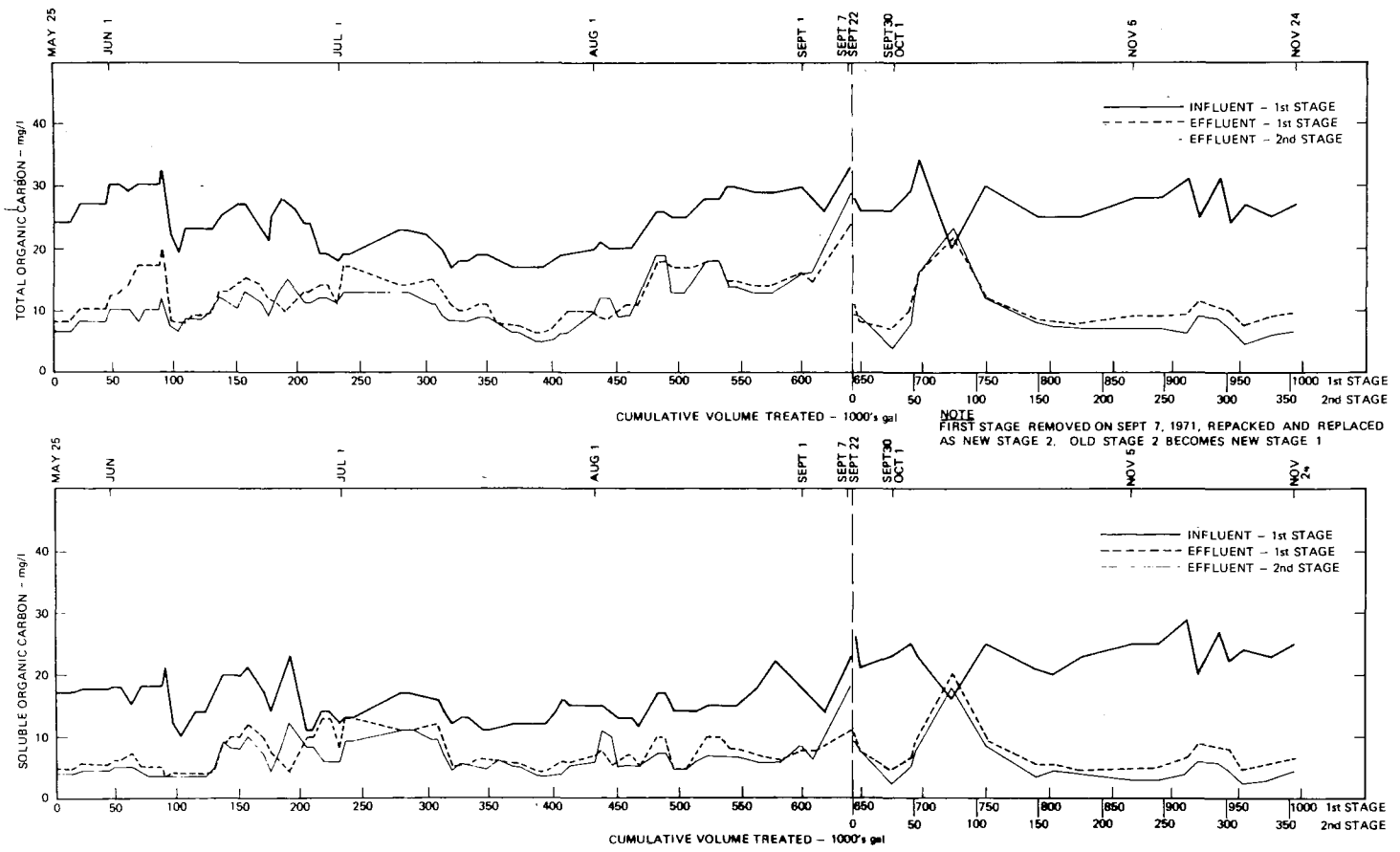

Figure 4-16. Performance Data of Activated Carbon on Chemical Treatment Process Effluent

it would require about 690 lb of activated carbon to treat one million gallons of chemically treated wastewater for the removal of about 50 percent of the influent TOC at a concentration of 24 mg/l.

Table 4-9

AVERAGE PERFORMANCE DATA OF ACTIVATED CARBON CHEMICAL TREATMENT PROCESS EFFLUENT

Item		First Stage Organic Carbon		Second Stage Organic Carbon		Overall Organic Carbon	
		Total	Soluble	Total	Soluble	Total	Soluble
Influent Carbon	mg/l	23.8	15.2	12.6[a]	7.2		
Effluent Carbon	mg/l	12.6	7.2	11.0	6.1		
Total Applied	lbs	131	82	69	40	131	82
Total Removed	lbs	62	43	8	6	70	49
Removal	$\frac{\text{lb TOC removed}}{\text{lb act. carbon}}$	0.14	0.10	0.019	0.014		
Average Removal	percent	47	52	12	15	52	60
Average Flow	gpm/sq ft	0.76	.76	0.76	0.76		
Total Throughput Volume	gallons	0.64×10^6		0.64×10^6			
Average Empty Bed Contact Time	minutes	24 5		24.5			

One major difficulty that arose during the activated carbon processing of the chemically treated wastewaters was the noxious odors that were generated in the carbon columns. Since little or no aeration occurred during chemical treatment, the influent to the carbon columns was devoid of oxygen. As the amount of adsorbed organics increased, bacteria began to attack these materials, producing odors through putrefaction (production of hydrogen sulfide). Injection of oxygen and chlorine ahead of the filters was not sufficient to overcome the anerobic conditions that had developed in the filters and the carbon columns. It is possible that if aeration and chlorination had been practiced from the start of the experimental runs, the anerobic conditions might never have developed. The use of diffused air aeration ahead of activated carbon treatment would most likely be impracticable; it would undoubtedly result in considerable foaming due to the high concentrations of MBAS (Methylene Blue Active Substances, surfactants) that had not been removed by chemical treatment alone. Additional studies are required to develop a suitable means of eliminating these problems.

EVALUATION OF TREATMENT PROCESSES

The average physical and chemical quality parameters of the treated effluents from the various pilot plant studies are summarized in Table 4-10. Where applicable, average removal efficiencies are also presented. In this section, these parameters are discussed along with a comparison of the results from the filtration and activated carbon adsorption studies. Also, results from separate virus and trace metal removal investigations are presented.

Organic Carbon

The average TOC and SOC of the various effluents differed only moderately during the study. Nitrification reduced the TOC and SOC of the

Table 4-10

PILOT PLANT AVERAGE EFFLUENT QUALITY
COMPARED TO THE RAW WASTEWATER

Parameter	Activated Sludge (1 Stage)	Nitrified Effluent (2 Stage)	Denitrified Effluent (3 Stage)	Chemical Effluent (2 Stage)
TOC				
mg/l	26	15	20	26
% removal	82	89	85	70
SOC				
mg/l	9	6.0	7.2	18
% removal	61	83	79	5
Suspended Solids				
mg/l	33	23	42	19
% removal	86	90	82	87
pH				
mg/l	7.4	7.1	7.0	9.2
Conductivity				
µmhos/cm	982	846	767	827
Ammonia-N				
mg/l	7.4	2.9	2.1	16
% removal	70	88	—	20
Nitrate-N				
mg/l	16.5	19.3	3.5	0.1
% removal	—	—	82	—
Ortho-Phosphate				
mg/l P	7.7	9.3	7.3	0.2
% removal	—	—	—	98
Total Phosphate				
mg/l P	8.6	9.7	7.4	0.4
% removal	—	—	—	95

activated sludge effluent by 11 mg/l and 3 mg/l, respectively. Following denitrification, both the TOC and the SOC increased, the former due primarily to the carryover of suspended solids from the settling basin and the latter probably from excess methanol in the treated effluent.

The average TOC and SOC of the chemically treated effluent (without activated carbon treatment) was somewhat greater than that resulting from biological treatment, primarily due to the poor removal of soluble organic material in the lime treatment operation. Alkaline hydrolysis of particulate organic carbon apparently released as much as new soluble organic material as was adsorbed by the insoluble calcium floc formed during the chemical treatment process, leading to an average SOC reduction of less than 5 percent. Although the quality of the chemically treated effluent was poorer than that of the biological process, chemical treatment demonstrated less variability than was observed with the biological processes.

Suspended Solids

The average suspended solids concentrations for the biologically treated settled effluents in the pilot plant decreased in proceeding from activated sludge through nitrification and then increased through the denitrification process. The activated sludge effluent observed in the pilot plant, with a suspended solids concentration of 33 mg/l, was significantly higher than observed in the CCCSD 1-mgd plant, which had an average concentration of 8.7 mg/l. This difference can be attributed to (1) poorer efficiencies normally associated with small scale sedimentation basins and (2) the relatively low overflow rates in the CCCSD plant. Also, problems were encountered in the activated sludge pilot plant with floating sludge that significantly increased the effluent suspended solids concentration. The effluent suspended solids from the nitrification process at 23 mg/l were generally lower than observed with the activated sludge pilot plant but were higher than observed with the CCCSD 1-mgd activated sludge plant.

The nitrified effluent quality further degenerated after denitrification, primarily due to the buoying of settled solids by nitrogen gas bubbles. Thus, the combined treatment of activated sludge, nitrification, and denitrification removed only about 82 percent of the suspended solids with respect to the raw wastewater. As noted earlier, recent information illustrates that significant improvement in denitrification MLSS separation can be achieved by aerating these solids for 30 to 60 minutes prior to final sedimentation.

Chemical treatment of the partially settled raw wastewater removed 87 percent of the suspended material, producing an effluent having 19 mg/l. Since the chemical treatment process resulted in the precipitation of soluble materials during the flocculation reaction, a significant portion of the 19 mg/l suspended solids in the effluent was calcium carbonate rather than suspended organic materials. Contrary to the biological processes, chemical treatment consistently produced a moderately low suspended solids concentration in the effluent.

Inorganic Nitrogen

The data listed in Table 4-10 indicate that the activated sludge process oxidized 70 percent of the ammonia -N to nitrate. However, this did not represent the conversion efficiency desired, particularly since only about 50 percent of the ammonia could be consistently oxidized in this single-stage system. By using a two-stage biological treatment system, the average ammonia concentration was reduced to 2.9 mg/l for an overall ammonia conversion of 88 percent. This higher efficiency was attained despite the difficulties of maintaining an adequate MLSS in the second-stage nitrification basin. Although the average effluent ammonia -N was 2.9 mg/l, much of the time concentrations of 1 mg/l or less of ammonia -N were observed. The data indicate that by maintaining an adequate

MLSS concentration in the nitrification reactor very low effluent ammonia concentrations can be consistently obtained.

On the average, 82 percent of the influent nitrate -N was reduced to nitrogen gas in the denitrification reaction, resulting in an average effluent concentration during the study period of 3.5 mg/l nitrate -N (Table 4-10) As with the nitrification process, the major difficulty encountered during the operation of the denitrification process was in maintaining the MLSS at a desired level.

A small amount of ammonia was lost during chemical treatment, reducing the ammonia -N content by approximately 20 percent. These losses probably resulted from the high surface-area-to-volume ratio in the pilot plant in conjunction with the high pH. It is doubtful that the ammonia removal would be this great in a full-scale plant.

Phosphorus Removal

Between 95 and 98 percent of the phosphorus was removed in the chemical treatment system, resulting in effluent concentrations of about 0.4 mg/l of phosphorus. While this is enough phosphorus to stimulate algal growths, potential problems in industrial cooling towers could be controlled by periodic shock doses of chlorine.

Filtration

Results of the filtration studies indicated that the average turbidity of the effluents from the activated sludge, nitrification, and chemical treatment processes could be maintained at 5 JTU or less. Apparently, the floc formed during these operations had a high shear strength and was readily removed during filtration.

Floc from the denitrification basin did not exhibit the same filterability characteristics. Solids frequently broke through the filters during operation with this effluent. Thus, the denitrification process floc was either finer than floc from the other processes or had a lower shear strength. In either case, filtration of denitrification process effluent most likely will require the use of filter aids (polyelectrolyte) in order to produce a desirable effluent quality. Aeration of the denitrification MLSS prior to settling may also be expected to improve the solids filterability.

Since the filters were not operated in parallel on the same effluents, it is very difficult to compare their performance. There was no significant difference in the effluent quality from the three filters during all operations except that of denitrification, where there appeared to be a reduction in the variability of the effluent turbidity when the mixed media filter was used instead of the dual media. Further evaluations of filterability would be necessary before the optimum media could be selected.

During the operation of the filtration process, biological growths developed in the filter media. To prevent this problem from occurring, a suitable means of disinfection should be provided ahead of the filters. In general, filtration significantly improved the effluent quality and process reliability of all the pilot plant systems investigated.

Activated Carbon

The results for the first stage of carbon adsorption of filtered activated sludge, denitrification, and chemical effluents are summarized in Table 4-11. The carbon used with the activated-sludge-treated wastewater had

a slightly greater adsorption capacity than that determined with the other two effluents. Carbon adsorption of the denitrified effluent produced the lowest organic carbon concentrations in the treated effluent, despite the fact that the influent TOC was greater than that from the activated sludge unit. These lower concentrations in the denitrified effluent were probably due to the increased detention time in the carbon column which was necessitated by the need for a lower hydraulic loading in the denitrification reactor and settling basin. The chemically-treated wastewater, which had the highest influent TOC and SOC, also had the highest effluent organic carbon concentration. Removal efficiencies in the first-stage treatment of the chemical effluent were nearly equivalent to those in the denitrified effluent and somewhat better than those for activated sludge, although increased contact time for the activated sludge effluent would be expected to improve the removal efficiency.

Table 4-11

COMPARISON OF SINGLE-STAGE ACTIVATED CARBON PERFORMANCE FOR VARIOUS PILOT PLANT EFFLUENTS

Parameter	Activated Sludge	Denitrified Effluent	Chemical Effluent
Effluent			
TOC mg/l	8.3	7.2	12.6
SOC mg/l	5.8	3.4	7.2
lbs TOC Removed / lb Activated Carbon	0.17	0.13	0.14
Average Removal			
TOC %	40	50	47
SOC %	33	60	52
Contact Time (min)	11	21	24.5
lbs Carbon required to treat 1 mg	410	—	690

Because of the higher influent TOC and SOC concentrations to the activated carbon columns in the chemical treatment plant effluent, more activated carbon per unit volume treated was required to adsorb the organic carbon. Thus, it would require 690 pounds of activated carbon to treat 1 mg chemically treated effluent, about 1.7 times the amount required to treat the activated sludge effleunt.

While having two activated carbon columns in series increased the reliability of the adsorption system during the pilot plant studies, the second carbon stage generally removed no more than 2 to 4 mg/l of organic carbon. It is possible that the activated carbon treatment could be optimized either by decreasing the carbon contact time in each stage or by having only a single-stage treatment system to provide sufficient contact time. The type of system which would actually be employed would depend on the water quality requirements for each specific reuse application and the relative cost of the two-stage versus single-stage system.

Virus Removal

The viral content of the treated wastewater is important from a public health point of view, especially if the water is to be reused or discharged to a waterway which may be used for body contact recreation. Because of the potential hazards of virus in the treated waters, a separate study was sponsored by CCCSD/CCCWD and Bechtel Corporation to determine virus removals in the pilot plant processes. A brief summary of the results of the study conducted by Cooper et al. (Reference 5) is presented here.

Since the viral content of treated wastewater was anticipated to be at or near the detection limit of available analytical procedures, it was deemed necessary to add a known amount of attenuated virus (poliovirus type 1, strain LSc) to the wastewater and then measure the concentration of

virus after various steps in the treatment process. Tracer studies through the various processes were made so that effluent concentrations with respect to time could be predicted and compared with actual measurements.

Results of experiments in the biological treatment systems indicated that the viral concentration was reduced from one-one hundreth to one-one thousandth of its initial concentration in the treatment units containing high MLSS concentrations. When the pH of the chemical treatment system was maintained at 11 or above, there was no virus found in the effluent. The results indicated a very rapid reduction in the viral concentration in the lime reactor so that after just three minutes, the viral concentration was reduced to about one-five hundredth of its original concentration. Limited removal of virus occurred in the filtration process, while the activated carbon reduced the influent viral concentrations by about 75 percent.

HEAVY METALS

Table 4-12 illustrates the removal of heavy metals in the activated sludge, filtration, activated carbon, and chemical treatment processes. Atomic absorption spectroscopy and emission spectrography analyses were made on 24-hour composites of the influent and effluent streams. Removal efficiencies were generally better in the chemical treatment process than they were in the activated sludge process, except for copper and nickel. However, since the influent concentrations were generally quite low, analytical variance makes it difficult to compare the results directly.

Appendix B contains a summary of data from a series of experimental runs using the chemical treatment plant in which a solution of soluble metal salts was added to the influent stream. Removals ranged between

85 and 99 percent for all metals except hexavalent chromium, which was reduced by an average of only about 20 percent. Filtration generally removed between 60 and 99 percent of the metals, indicating that most of the metals were associated with the particulate material in the waste streams. Similar results were achieved using activated carbon, where adsorption of organometallic compounds as well as straining of particulate metallic materials were undoubtedly taking place.

Table 4-12

REMOVAL OF HEAVY METALS

	Biological-Physical Treatment												Chemical Treatment		
	Activated Sludge														
	Pilot Plant			CCCSD 1 mgd			Filtration			Activated Carbon					
	In mg/l	Out mg/l	% rem	In mg/l	Out mg/l	% rem	In mg/l	Out mg/l	% rem	In mg/l	Out mg/l	% rem	In mg/l	Out mg/l	% Rem
Barium	Nil	Nil		0.50	Nil	99+	Nil	Nil	-	Nil	Nil	-	0.43	Nil	99+
Chromium	0.12	0.06	50	0.05	0.01	80	0.42	0.01	98	Nil	Nil	-	0.17	0.07	59
Copper	0.12	0.03	73	0.8	0.19	76	0.033	0.023	30	0.023	0.015	35	0.14	0.05	64
Lead	0.09	0.02	78	0.115	0.008	93	Nil	Nil	-	0.040	0.001	98	0.13	Nil	99+
Manganese	0.27	0.24	11	0.39	0.32	18	0.017	0.015	12	0.060	0.001	99	0.33	0.02	94
Nickel	0.07	0.02	71	0.085	0.021	75	0.01	0.006	60	0.010	0.005	50	0.015	0.004	67
Silver	0.012	0.006	50	0.007	0.001	85	Nil	Nil	-	Nil	Nil	-	0.013	0.002	85
Titanium	0.035	0.016	54	0.27	0.01	98	0.01	Nil	99+	0.34	0.01	98	Nil	Nil	-
Zinc	Nil	Nil		0.33	0.10	70	Nil	Nil		0.020	0.001	98	Nil	Nil	-

Section V

RESULTS AND DISCUSSION OF INDUSTRIAL TEST LOOP STUDIES

Three industrial test loops and a test boiler received Contra Costa Canal water and various grades of renovated water during the pilot-demonstration project to evaluate the feasibility of using renovated wastewater for industrial purposes in relationship to the present water source. Data on the scaling potential, corrosion rate, algal growth potential, and toxicity of the circulating water in the test loops are presented and discussed in this section.

HEAT EXCHANGER FOULING DATA

During the normal operation of heat exchangers, there may be scale formed on the walls of the heat exchanger tubes. This scale may reduce corrosion, but, if it becomes excessive, a significant loss in heat transfer occurs. During this study, reduction in the rates of heat transfer and the related fouling factors were used as one means of evaluating the various renovated waters and canal water. Knowing the temperature of the water and steam at the inlet and outlet of the heat exchanger, the flow (contact time), surface area, and type of heat exchanger tubes, it was possible to calculate the heat transfer coefficient. Any reduction from the original value indicated that fouling was occurring and a fouling factor could be calculated.

Heat exchanger tube fouling may result from precipitation of calcium phosphate or carbonate, corrosion products (rust), dirt, and other foreign materials which accumulate on the heat exchanger surfaces. Table 5

Table 5-1

FOULING FACTORS AND SCALE ANALYSES

Makeup Source	Contra Costa Canal				Nitrification Effluent		Activated Carbon/ Nitrification Effluent		Denitrification Effluent		Filtered Denitrification Effluent		Filtered Chemical Treatment Effluent	Activated Carbon Chemical Treatment Effluent	
Exchanger Tube Exposure Period	5/18-6/8	6/9-7/1	7/6-7/30	8/1-8/31	5/18-6/8	6/9-7/1	5/18-6/8	6/9-7/1	7/6-7/30	8/1-8/31	7/6-7/30	8/1-8/31	10/29-11/24	10/29-11/24	10/29-11/24
Corrosion Inhibitor	No	No	No	Yes	No	No	No	No	No	No	No	No	No	No	Yes
Phosphorus Removal	No	No	No	No	No	No	No	No	No	Yes	No	Yes	Yes	Yes	Yes
Fouling Factor (hr-°F-sq ft/Btu)	0.001	0.003	0.0005	0.002	0.0005	0.0005	0.0005	0.003	0.002	0.001	0.004	0.002	0.001	0.002	0.003
pH	8.5	8.0	8.5	7.5	8.4	7.8	8.0	7.8	8.5	7.9	8.4	7.4	8.6	8.1	8.4
Cycles of Concentration	9.8	7.0	8.9	6.5	5.6	6.7	4.4	6.2	8.0	8.4	5.7	5.4	10.4	5.4	7.4
Temperature (Heat Exchange Outlet) (°F)	125	140	108	134	100	115	100	108	102	134	106	134	126	117	120
Spectrographic Analysis (%)															
Aluminum	0.08	0.6	0.3	0.6	0.03	0.02	0.02	0.01	0.02	0.04	0.6	0.03	0.15	0.2	0.2
Boron	Nil	Nil	Nil	Nil	Nil	0.03	Nil	0.03	0.05	0.02	0.01	0.009	0.002	0.004	0.002
Calcium	0.01	0.3	0.06	0.2	16	30	15	35	20	4.2	23	5.3	0.08	0.5	0.30
Chromium	0.01	0.01	0.59	1.5	0.01	0.005	0.002	0.008	0.01	0.024	0.015	0.02	0.66	0.05	0.009
Cobalt	0.002	0.003	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	0.002
Copper	0.08	0.10	0.09	0.009	0.01	0.03	0.01	0.04	Nil	0.09	0.006	0.09	0.08	0.06	0.05
Iron	65	60	62	54	4.3	0.4	2.4	0.7	0.36	48	15	29	20	17	19
Lead	Nil	0.01	0.03	0.2	0.06	0.005	0.05	0.005	0.1	0.05	0.06	0.1	0.02	0.06	0.05
Magnesium	0.3	0.7	0.2	1.2	6.1	2.0	4.3	1.5	3.9	1.4	2.5	1.0	0.6	0.6	0.4
Manganese	0.2	0.2	0.1	0.20	0.5	0.2	0.4	0.08	0.4	0.3	0.4	0.2	0.08	0.10	0.08
Molybdenum	0.01	0.01	0.09	0.2	Nil	Nil	Nil	Nil	Nil	0.02	Nil	0.01	0.04	0.007	0.006
Nickel	0.002	0.007	Nil	0.02	0.006	0.003	0.001	0.003	0.006	0.01	0.008	0.009	0.005	0.005	0.007
Phosphorous	Nil	Nil	Nil	Nil	22	15	26	15	25	2.8	6.1	11	Nil	1.6	1.0
Silicon	Nil	5	2.4	3.6	Nil	0.05	Nil	0.04	0.7	2.7	0.06	0.7	0.40	0.3	0.3
Silver	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	0.0007	0.0006	0.0009	0.0003	Nil	Nil	Nil
Strontium	0.002	0.01	Nil	Nil	0.50	0.1	0.30	0.1	0.1	0.04	0.2	0.04	0.01	0.02	0.02
Tin	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	0.011	Nil	Nil	Nil	Nil	Nil	Nil
Titanium	0.002	Nil	0.006	0.01	0.01	Nil	Nil	Nil	Nil	Nil	Nil	Nil	0.003	Nil	Nil
Vanadium	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Zinc	3.0	6.0	2.0	7.5	6.2	5.0	6.9	6.0	1.7	6.3	3.1	16	2.5	5.2	5.0
Other Metals	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil

summarizes the data for the chemical composition of the scale that formed during the various test periods of this study. Fouling factors (see Reference 6 for calculation procedure) and operating data for each test period are included in the table. Analyses of the scale formed in the heat exchanger tubes where canal water was used in the test loops indicated that it consisted primarily of corrosion products (iron) with some deposition of silica. There was no significant calcium or phosphorous scale formed during use of the canal water. The addition of a corrosion inhibitor (NALCO 370) to the canal water caused a moderate increase in the amount of zinc-chromate in the scale but had little effect on the magnitude of the fouling factor. The relatively high levels of zinc in the scale formed by all of the samples listed in Table 5-1 most likely resulted from the dissolution of the galvanized coating on the bottom pans of the cooling towers, since analysis of the influent waters indicated relatively low zinc levels.

In contrast to the canal water, the renovated waters that had received biological-physical treatment generally resulted in scale formation composed largely of calcium and phosphate. When ferric chloride was added to the denitrification basin to reduce the phosphorous concentrations in late July and August, the iron content of the scale formed in using this water increased sharply, whereas the phosphate was appreciably reduced. This reduction in phosphorous scale was observed even though only about 50 percent of the phosphorous had been removed from the makeup water.

The significance of this scale is further illustrated by the results obtained with the effluents from the chemical treatment process. With these effluents, the phosphorous and calcium scale formed in the heat exchanger tubes was greatly reduced, resulting in a scale composition nearly the same as found with the canal water. Also, the silicon in the scale was generally less when using the renovated waters than when using the canal

water. Based on these observations, it must be concluded that a high degree of phosphorous removal is desirable for the use of renovated water for industrial cooling purposes.

The fouling factors, shown in Table 5-1, ranged from 0.0005 to 0.004 hr-^{o}F-sq ft/Btu for the canal water and the renovated waters. These factors represent the values observed after 18 days from the time new heat exchanger tubes were placed in operation for each exposure period.

The nitrified effluents consistently resulted in the lowest fouling factors observed (i.e., 0.0005 hr-^{o}F-sq ft/Btu). However, nitrified effluent that was filtered and treated with activated carbon resulted in fouling factors ranging from 0.0005 to 0.003 hr-^{o}F-sq ft/Btu. There is no apparent reason for the lower fouling factors observed with the nitrified effluent.

The fouling factor observed for the filtered chemically treated effluent was 0.001 hr-^{o}F-sq ft/Btu, whereas this effluent that had activated carbon treatment resulted in a fouling factor of 0.002 hr-^{o}F-sq ft/Btu. The addition of corrosion inhibitor to the carbon-treated effluent further increased the fouling factor to 0.003 hr-^{o}F-sq ft/Btu.

An interesting observation from the tests conducted is that the renovated waters that were filtered or treated with activated carbon generally had somewhat higher fouling factors than the same waters without these additional treatment steps. The scale formed with the filtered and carbon-treated waters appeared to be considerably harder and adhered to the heat exchanger surface more tenaciously. It is possible that when organic and particulate materials were present (i.e., no filtration or activated carbon treatment), the scale formed on the heat exchanger tubes was partially removed because of scouring action of the circulating water.

Visual observations made during operation of the test loops indicated that the rather turbid canal waters and the unfiltered renovated waters deposited a significantly greater amount of solids in the bottoms of the cooling towers than the filtered effluents. Although the solids deposition did not adversely affect the operation of the test loop equipment, such deposition in heat exchange components used by industry could result in significant operational problems. These difficulties would be expected to offset any benefits derived from the softer scale formed without filtration.

Figure 5-1 summarizes the fouling factor data described above. The data plotted for the canal water and various process waters represent average values. The data indicate that the fouling potential of the renovated waters is nearly the same as observed with the canal waters. Filtered, chemically treated effluent and nitrified effluent had the lowest fouling potentials.

Control of the pH in the pilot plant treatment processes and the industrial test loops was limited so that on occasion pH values in the makeup and circulating waters were higher than desired. During these periods, increased precipitation of calcium salts most likely occurred, resulting in an accelerated decrease in the heat transfer rate over a very short time period. Although the process difficulties were generally corrected within a day, the precipitated materials probably remained, thus keeping the heat transfer at a low rate. Because of this, it is felt that the data presented for the fouling factors represent conservative (high) values for fouling. In a full-scale treatment plant, operating conditions would be more closely controlled, thereby eliminating some of the problems resulting from abnormally high pH values.

Representative fouling factors for various types of waters used in industrial applications are listed in Table 5-2. The fouling factors (corrected

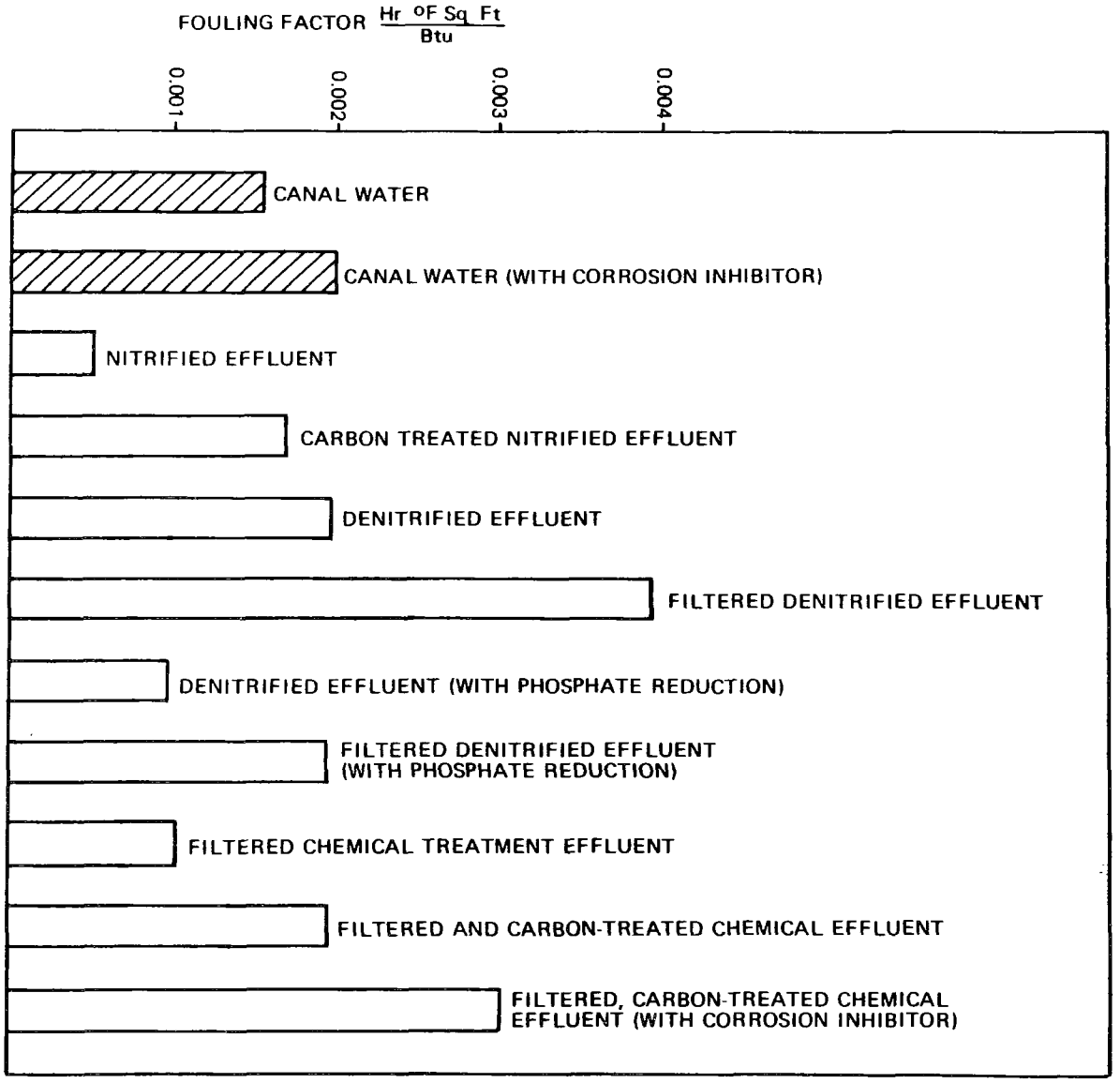

Figure 5-1. Average Fouling Factor Data

Table 5-2

TYPICAL FOULING FACTORS*

Temperature of Heating Medium	Up to 240°F		240°F to 400°F	
Temperature of Water	125°F or Less		Over 125°F	
Types of Water	Water Velocity		Water Velocity	
	3 ft/sec and Less	Over 3 ft/sec	3 ft/sec and Less	Over 3 ft/sec
Sea Water	.0005	.0005	.001	.001
Brackish Water	.002	.001	.003	.002
Cooling Tower and Artificial Spray Pond:				
Treated Makeup	.001	.001	.002	.002
Untreated	.003	.003	.005	.004
City or Well Water (Such as Great Lakes)	.001	.001	.002	.002
Great Lakes	.001	.001	.002	.002
River Water:				
Minimum	.002	.001	.003	.002
Mississippi	.003	.002	.004	.003
Delaware, Schuylkill	.003	.002	.004	.003
East River & New York Bay	.003	.002	.004	.003
Chicago Sanitary Canal	.008	.006	.010	.008
Muddy or Silty	.003	.002	.004	.003
Hard (Over 15 grains/gal)	.003	.003	.005	.005
Engine Jacket	.001	.001	.001	.001
Distilled	.0005	.0005	.0005	.0005
Treated Boiler Feedwater	.001	.0005	.001	.001
Boiler Blowdown	.002	.002	.002	.002

*Reference 6

to a velocity of 2 ft/sec), observed during this study for various renovated waters compare favorably with the published data. This information supports the results obtained and indicates that on the basis of fouling, renovated waters are comparable to many other sources of industrial cooling water being used throughout the United States.

CORROSION RATES

Corrosion rates associated with the use of Contra Costa Canal water and various grades of renovated water in industrial applications were determined using two procedures. The first procedure involved the use of a portable corrosion meter (Magna Corporation) in conjunction with a corrosion probe inserted in the circulating water of each industrial test loop. Corrosion rates using this procedure could be determined immediately. The second procedure utilized standard corrosion coupons made from carbon steel. Four of these coupons were inserted in a circulating water bypass loop during each testing period. Coupon weight loss was determined after each testing period using ASTM procedures, and a corrosion rate was calculated. The corrosion probes and the corrosion coupons were located on the discharge side of the heat exchangers.

Table 5-3 summarizes the corrosion rates obtained with the corrosion probes and meter for the various renovated waters and canal water in the industrial test loops. Chemical characteristics of the circulating waters are also shown in Table 5-3.

The observed corrosion rates, as determined with the corrosion probes and meter, for the untreated canal water ranged from 23 to 106 mils/yr while the corrosion rates for the renovated waters that received biological treatment ranged from 6.3 to 14 mils/yr. A corrosion rate of 3 mils/yr was observed for the filtered chemical treatment effluent. However, a

Table 5-3

CORROSION RATES AND CIRCULATING WATER QUALITY

Source of Water		Contra Costa Canal					Act Sludge Eff	Filtered Act Sludge Eff	Nitrification Eff		Act Carbon Nitrification Eff		Dentrification Eff		Filtered Dentrification Eff		Filtered Chem Treatment	Act Carbon Chemical Treatment Eff	
Exchanger Tube Exposure Period		3/5-5/4	5/18-6/8	6/9-7/1	7/6-7/30	8/1-8/31	3/5-5/4	3/5-5/4	5/18-6/8	6/9-7/1	5/18-6/8	6/9-7/1	7/6-7/30	8/1-8/31	7/6-7/30	8/1-8/31	10/29-11/24	10/29-11/24	10/29-11/24
Corrosion Inhibitor		No	No	No	No	Yes	No	No	No	No	No	No	No	No	No	No	No	No	Yes
Phosphorous Removal		No	No	No	No	No	No	No	No	No	No	No	No	Yes	No	Yes	Yes	Yes	Yes
Corrosion Rate*	mils/yr	106	68	41	23	5.4	10	6.3	14	7.8	10.7	6.3	12	9.2	7.9	11	3.0	72	26
Cycles of Concentration		4.7	9.8	7.0	8.9	6.5	4.0	4.7	5.6	6.7	4.4	6.2	8.0	8.4	5.7	5.4	10.4	6.1	7.4
Langelier Index		0.27	1.42	0.83	1.74	3.05	1.31	0.98	1.47	0.82	0.68	0.77	1.75	0.89	1.53	0.27	2.7	0.5	0.93
Calcium	mg/l $CaCO_3$	168	267	215	316	239	424	496	395	471	289	426	366	542	338	496	580	260	396
Alkalinity	mg/l $CaCO_3$	69	88	70	230	52	112	87	111	64	59	52	258	74	207	49	230	40	60
pH		8.0	8.5	8.0	8.5	7.5	8.0	7.9	8.4	7.8	8.0	7.8	8.5	7.9	8.4	7.4	8.6	8.1	8.3
Total Phosphate	mg/l PO_4	0.08	0.60	0.70	1.5	1.8	18	15	10	24	9	19	13	13	12	20	1.2	1.5	1.9
Conductivity	µmhos/cm	1263	2143	1415	1780	1323	3063	4740	5180	5855	3944	5406	5780	5242	5100	4929	7610	4400	5420
Exit Temperature	°F	104	119	114	102	123	110	107	121	118	122	128	105	127	104	130	130	130	119
Suspended Solids	mg/l	15.2	18	14	48	9.0	6.3	35	1[illegible]	3.6	4.7	2.4	8.3	13	5.8	11	7.5	4.0	3.7

Data obtained from corrosion probes

Note: Act Activated
Eff Effluent

rate of 72 mils/yr was determined for the chemical treatment effluent that was also treated with activated carbon. These latter values suggest that the 3 mils/yr corrosion rate observed with the filtered chemical treatment effluent may be erroneously low. The addition of a corrosion inhibitor to the canal water decreased the corrosion rate from an average of 60 mils/yr to 5.4 mils/yr. A decrease in the corrosion rate from 72 mils/yr to 26 mils/yr was observed when corrosion inhibitor was added to renovated water having chemical and activated carbon treatment.

Shown in Table 5-4 are the corrosion rates obtained from the corrosion coupons inserted in the industrial test loop circulating waters. Also shown in this table are the corresponding corrosion rates observed with the corrosion probes and meter. In all cases the corrosion coupons indicated lower corrosion rates than determined with the corrosion probes and meter. Biologically treated renovated waters resulted in corrosion rates ranging from 2.0 to 6.6 mils/yr as determined with the corrosion coupons, while untreated canal water had corrosion rates of 12.5 to 37.2 mils/yr. The canal water corrosion rate was reduced to 2.7 mils/yr by the addition of a corrosion inhibitor. The renovated water receiving chemical and activated carbon treatment resulted in a corrosion rate, as determined with the coupons, of 31.0 mils/yr; this rate was reduced to 11.9 mils/yr with the use of a corrosion inhibitor.

The data generally indicated that the renovated waters receiving treatment for the reduction of phosphorous had higher corrosion rates than when phosphorous was not removed. This observation suggests that either the phosphorous in the water inhibited corrosion or the phosphate scale that was formed on the heat exchanger tubes provided a protective coating. Visual observation of the heat exchanger tubes suggested that the scale formation most likely was the primary mechanism associated with the reduction in the corrosion rate. Also, solubility coefficients for various calcium and phosphorous compounds indicate that scale formation could continue to the point that heat exchanger tubes would be severely restricted.

Table 5-4

COMPARISON OF CORROSION RATES

Type of Water	Date	Corrosion Rate mils/yr	
		Coupons*	Probe**
Canal Water	6/9 7/1	37.2	41.0
Canal Water	7/6 7/30	12.5	23.0
Canal Water with Corrosion Inhibition	8/1 8/31	2.7	5.4
Nitrified Effluent	6/9 7/1	2.9	7.8
Denitrified Effluent	7/6 - 7/30	2.0	12.0
Denitrified Effluent with Phosphorous Reduction	8/1 - 8/31	6.6	9.2
Filtered Denitrified Effluent	7/6 7/30	2.2	7.9
Filtered Denitrified Effluent with Phosphorous Reduction	8/1 8/31	4.9	11.0
Chemical and Activated Carbon Treated Effluent	10/29 11/24	31.0	72.0
Chemical and Activated Carbon Treated Effluent with Corrosion Inhibitor	10/29 - 11/24	11.9	26.0

*Data obtained with corrosion coupons
**Data obtained with corrosion probes

For the canal water and the renovated waters, the Langelier Index did not provide an accurate assessment of the potential for scaling or corrosion. The chemical characteristics of these waters must have been responsible for these inaccuracies.

ALGAL GROWTH POTENTIAL

Since many of the problems which arise from the discharge of nutrients into the environment are associated with the response of algae to the increased nutrient levels, a means of assessing this potential growth would be useful in evaluating the efficacy of various treatment procedures. The Provisional Algae Assay Procedure (PAAP) was used for this purpose during this investigation in accordance with the methods developed by the University of California Sanitary Engineering Research Laboratory. This procedure utilized algae to measure the growth potential of a given sample of water in much the same way that BOD is used to assess the oxygen consuming capacity of a given waste by bacteria. Growth of the test species of algae Selenastrum capricornutum was assessed using a standard solution which had all of the nutrients required for the growth of the algae. The growth of the algae in this standard solution was then compared to the growth in the sample solutions.

Table 5-5 indicates the phosphorous and inorganic nitrogen concentrations in the sample waters which were used for the Provisional Algae Assay Procedure. The denitrified effluent had a high poshphorous content but relatively low concentrations of nitrate and ammonia, whereas the activated sludge effluent had both high phosphorous and high inorganic nitrogen concentrations. The chemically treated effluent had a very low phosphorous concentration and almost all of the nitrogen present was in the ammonia form. When these process waters were used as the algal growth

Table 5-5

SAMPLE ANALYSIS FOR ALGAE GROWTH POTENTIAL TESTS

Measured Parameter		Process Water		
		Denitrified Effluent	Activated Sludge Effluent	Lime-Treated Effluent
pH		7.4	7.5	7.8
Conductivity	μ mho	630	610	895
Orthophosphate as P	mg/l	8.5	9.1	Nil
Nitrate, as N	mg/l	0.3	9.0	Nil
Ammonia, as N	mg/l	0.6	9.0	17.0

medium, the response curves shown in Figure 5-2 were obtained. The algae grew rapidly in the standard test medium and, even after six days, were still in the log growth phase in this medium. There was a lag of about three to five days in the growth of the algae in the denitrified medium and the activated sludge medium. The log growth phase in the denitrified effluent lasted for approximately three days, and after about nine days it appeared as if nitrogen limited further growth of the algae in this medium. Growth in the activated sludge medium continued for the duration of the experiment after the initial lag period. Apparently, there was some material in the activated sludge effluent which inhibited the growth of the algae initially, but, once the algae became acclimated to the activated sludge effluent, they began to grow rapidly since they were not nutrient limited. The chemically treated effluent apparently had a low enough phosphorous concentration so as to limit the growth of algae in this medium. The data plotted in Figure 5-2 indicated that lime treatment produced an effluent which permitted little or no growth of the algae, whereas denitrification permitted a limited

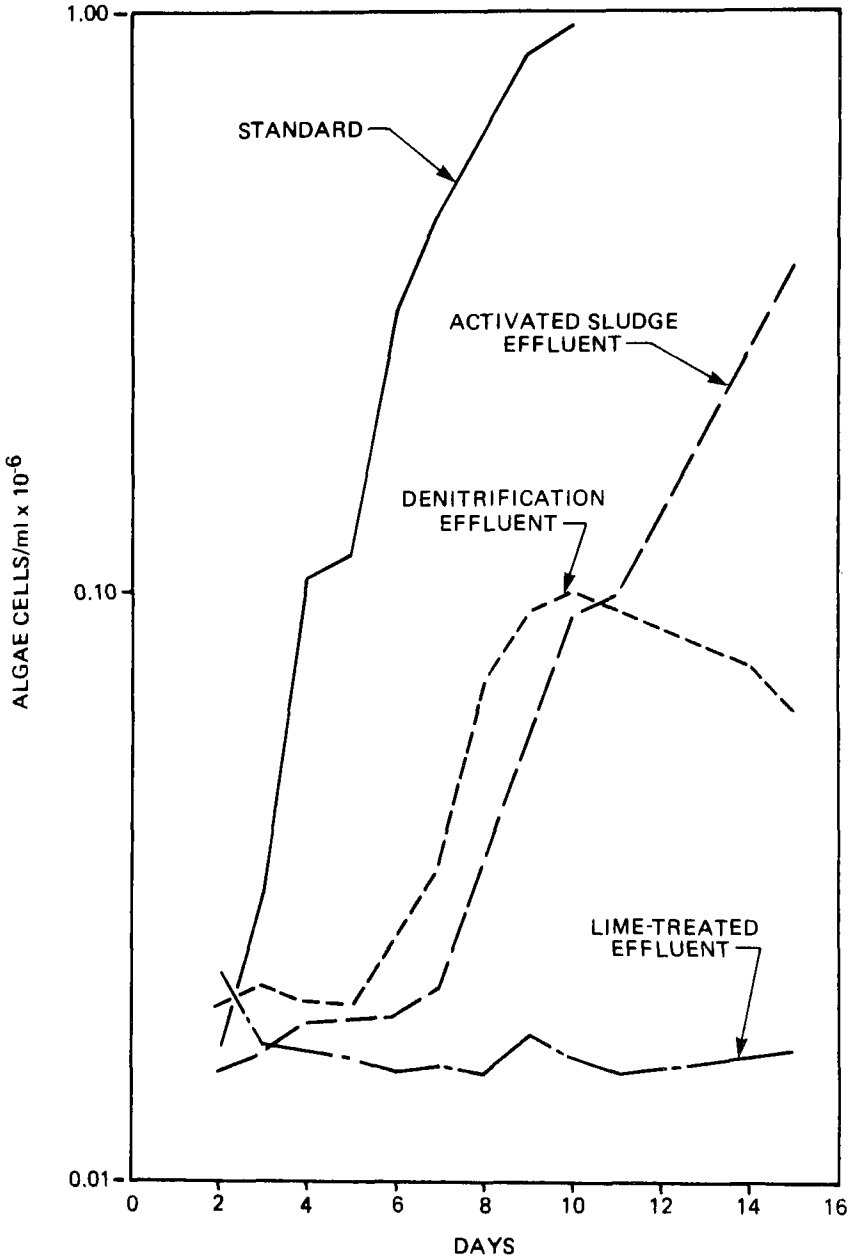

Figure 5-2. Algae Growth in Renovated Waters

growth of the algae. The high nutrient concentrations in the activated sludge effluent permitted a rapid and continuous growth of algae for at least a 15-day period.

With nutrient removals in excess of 95 percent, there will be sufficient nutrients remaining to support some growth of algae. However, it is anticipated that such growth can be readily controlled in cooling towers using established techniques.

TOXICITY ANALYSIS

The toxicities of effluent streams receiving primary and activated sludge treatment and primary with lime treatment are summarized in Table 5-6.* The results indicated that primary treatment produced an effluent which, when diluted to an average value of 47.5 percent of its initial concentration with dechlorinated tap water, killed 50 percent of the test species (stickleback) in 96 hours (96 hr LC-50% = 47.5). Undiluted effluent from the activated sludge plant killed less than 50 percent of the test species in 96 hours. Lime precipitation, on the other hand, produced an effluent which was only slightly less toxic than that resulting from primary treatment alone, so that diluting to 61 and 81 percent of the initial concentration produced a 50-percent kill after 96 hours in the chemical effluent. By providing activated carbon adsorption after chemical treatment, the toxicity of the effluent was reduced to the point that less than half of the fish were killed in the undiluted waste after 96 hours. Since the ammonia concentration did not change appreciably due to the carbon treatment, something other than just ammonia must have been contributing to the toxicity. When the ammonia was removed from the chemical treatment effluent by ion exchange (clinoptilolite), the survival was again greater than 50 percent after 96 hours in the undiluted waste.

*Toxicity analyses performed by Sanitary Engineering Research Laboratory, College of Engineering and School of Public Health, University of California, Berkeley.

Table 5-6

SUMMARY OF TOXICITY RESULTS*
(Central Contra Costa Sanitary District)

Constituent	Biological Treatment		Physical-Chemical Treatment			
	Primary Effluent	Activated Sludge Effluent	Partially Settled Sewage	Chemical Treatment Effluent	Chemical Treatment Effluent After	
					Activated Carbon	Ammonia Removal
Week 1 (April 23-27)						
COD, mg/l	208	86	260	83	43	69
BOD, mg/l	112	42	112	29	19	29
SS, mg/l	96	37	178	13	11	13
NH_3-N, mg/l	22.7	7.2	22.2	18.5	19.0	3.1
Total P, mg/l	9.3	10.5	12.8	1.6	1.5	1.5
pH	7.3	7.2	7.1	6.8	6.9	7.0
96 hour LC-50, percent	45	**	58	61	**	**
Week 2 (April 27-May 1)						
COD, mg/l	217	110	299	78	44	55
BOD, mg/l	80	30	130	20	13	14
SS, mg/l	88	32	129	15	9	8
NH_3-N, mg/l	24.8	7.8	20.9	20.9	21.0	1.9
Total P, mg/l	9.2	9.7	13.3	1.0	1.0	0.7
pH	7.4	7.3	7.4	7.2	6.7	7.6
96 hour LC-50, percent	50	**				
72 hour LC-50, percent			71	81	**	**

* Average of daily composites

** Less than 50 percent kill in 100 percent waste at 96 hours

Note: The above data were obtained by the Sanitary Engineering Research Laboratory at the University of California, Berkeley

Table 5-7 presents a comparison of the toxicity of activated sludge, nitrified, and filtered nitrified effluents from the pilot plant. After 96 hours of contact, two of the test species were dead in the undiluted activated sludge effluent, whereas there was complete survival of the test species in the nitrified effluents. All of the test species survived after 96 hours exposure to the circulating waters in the industrial test loops which had been concentrated by approximately five times. These data indicated that blowdown from cooling towers using renovated water should not result in toxicity problems if adequate treatment is provided.

Table 5-7

INDUSTRIAL TEST LOOP TOXICITY RESULTS

	Dilution	DO mg/l	pH	Number Surviving/Percent Survival				
				Start	24 hr	48 hr	72 hr	96 hr
CCSD Activated Sludge Effluent	None	8.2	7.95	10/100	10/100	10/100	8/80	8/80
Pilot Nitrified Effluent	None	8.6	7.45	10/100	10/100	10/100	10/100	10/100
Pilot Filtered Nitrified Effluent	None	9.0	7.55	10/100	10/100	10/100	10/100	10/100
Cooling Tower Circulating Water*								
Canal	None	8.6	7.15	10/100	10/100	10/100	10/100	10/100
Nitrified Effluent	None	8.0	7.70	10/100	10/100	10/100	10/100	10/100
Filtered Nitrified Effluent	None	8.8	7.60	10/100	10/100	10/100	10/100	10/100
Filtered Chemically Treated Effluent	None	7.9	8.4	10/100	10/100	10/100	10/100	10/100
Filtered Activated Carbon Chemically Treated Effluent	None	9.5	7.0	10/100	10/100	10/100	10/100	10/100

*Temperature 71° F Test Species stickleback Number of fish per test 10
*Blowdown from the cooling towers was about five times the concentration of the cooling tower feed.

EVALUATION OF INDUSTRIAL TEST LOOP RESULTS

The most significant finding during the investigation of fouling factors and corrosion rates was that renovated water used as cooling tower makeup performed at least as well as the Contra Costa Canal water. Precipitation of phosphorous in the test heat exchanger tubes was observed for the renovated waters not receiving treatment for phosphorous removal. Calcium precipitation was associated with the precipitated phosphorous. However, scale formed from the use of lime-treated renovated water with very low phosphorous concentrations had very small contents of calcium and phosphorous. Since the calcium concentrations and pH of the lime-treated waters were approximately the same as in the biologically treated effluents not receiving treatment for phosphorous removal, it appears that little precipitation of calcium carbonate or calcium sulfate would be expected when the renovated water tested in this study is used as cooling water. Thus, the chemical constituent in these wastewaters most directly influencing the scaling of heat exchanger tubes was phosphorous. Removing the phosphorous increased the corrosion rate of the renovated water, but this was satisfactorily reduced by the addition of a corrosion inhibitor.

When the above considerations are taken into account, the potential fallacy of considering only the total dissolved solids content when evaluating a water's utility for reuse becomes apparent. Water having a high TDS but a low phosphorous or calcium content generally would not be expected to result in problems associated with scaling from precipitation. Similarly, chlorides, which are important with respect to the stresses imposed on stainless steel heat exchanger tubes, may be relatively low in waters having a high TDS if these waters are derived primarily from the dissolution of calcareous sediments. Thus, TDS concentrations can be used to provide a preliminary indication of the potential utility of a given water for reuse, but should not be relied upon as the sole criterion for a given water.

The fouling factor results, summarized in Figure 5-1, indicate that nitrified effluent, denitrified effluent with phosphorous reduction, and filtered lime-treated effluent with and without carbon adsorption have fouling tendencies comparable to canal waters treated with a corrosion inhibitor. Corrosion rates for these waters were also approximately the same as canal water with a corrosion inhibitor added. The only exception to this was the filtered, activated carbon, lime-treated effluent which had a relatively high corrosion rate of 31 mils/yr. When the corrosion rate was reduced to about 12 mils/yr with a corrosion inhibitor, the fouling increased appreciably. These results indicate that a balance between corrosion and fouling can be achieved by the treatment processes of activated sludge and nitrification. Denitrification did not appear to improve the fouling or corrosion rates, so this added process would not be expected to significantly improve the industrial usability of the treated wastewater. While it is likely that adding a corrosion inhibitor to the filtered, lime-treated effluent would produce a water which would have a lower corrosion rate than the same water receiving activated carbon treatment, there is no way of knowing how this would affect the fouling factor, since experimental results for this particular test were not obtained.

Table 5-8 summarizes typical concentrations of chemical constituents in the Contra Costa Canal water and CCCSD primary influent. As is apparent from this table, there is an appreciable increase in the concentration of dissolved salts in the wastewater in relationship to the canal water. However, of major concern in the reuse of this wastewater for cooling water purposes is the calcium, carbonate, and sulfate concentrations. These constituents as well as phosphorous are important since they may enter into precipitation reactions in a cooling water system as the number of cycles of concentration and the temperature increase. Studies concerning the fouling tendencies of the renovated waters indicated that no significant precipitation of calcium salts occurred during the industrial test loop studies other than that due to precipitation with phosphorous.

Table 5-8

TYPICAL QUALITY CHARACTERISTICS
OF WASTEWATER AND CANAL WATER

Chemical Parameters	CCCSD Primary Influent (mg/l)	Canal Water (mg/l)
Na^{+}	106	57
K^{+}	12	2.4
Ca^{++}	51	26
Mg^{++}	17	15
Cl^{-}	123	78
$SO_4^{=}$	116	56
HCO_3^{-}	205	103
TDS	860	310
Alkalinity (as $CaCO_3$)	179	84
Hardness (as $CaCO_3$)	197	129

Thus, no trouble with precipitation of calcium salts is expected in using renovated water for cooling purposes at cycles of concentration of approximately five to eight, if the total phosphorous concentrations are reduced to 0.5 mg/l as P or less.

Chloride concentrations are of concern with regard to stress corrosion in the heat exchanger tubes fabricated from stainless steel. However, the chloride concentrations which cause such problems with Stainless Steel 316 are in excess of those in the renovated wastewater even at cycles of concentration above ten. Therefore, chlorides are not expected to be a major problem during the use of renovated water for cooling purposes.

In using renovated water for industrial purposes, limitations on the discharge of blowdown waters to receiving waters must be considered. Of prime concern is the toxicity of the discharge to established fish species and the tendency for the discharge to stimulate algal growths. Fish bioassay studies using the blowdown from the industrial test loops indicated that 100 percent of the test organisms survived after 96 hours of exposure to the concentrated canal water, unfiltered and filtered nitrified effluent, and filtered chemically treated effluent with and without activated carbon treatment. However, it should be noted that the bioassay studies were made on the blowdown waters after they had been aerated sufficiently to increase the DO to greater than 7 mg/l and cooled to ambient temperatures.

Zinc, which may be toxic to fish at concentrations of 1 mg/l or less, was one of the relatively high constituents of the scale formed in the industrial test loop studies. Although zinc was probably present in the blowdown water as well as the scale, there was no apparent effect of this toxic metal on the test organisms during the bioassay studies. As discussed earlier, the high zinc concentrations were probably due to leaching of zinc from the galvanized coating on the cooling tower bottom pans. This source of zinc would be greatly reduced or eliminated in a commercial-scale cooling tower.

The use of zinc chromate as a corrosion inhibitor in cooling towers, however, may cause toxicity to fish or other sensitive organisms if they are exposed to the blowdown water. This effect was not evaluated during this study but should be reviewed before such water is discharged to receiving bodies of water. In this respect, the use of renovated wastewaters is not expected to cause any greater problems than encountered in the use of canal water.

Based on the findings of the industrial test loop studies, the nitrified effluent produced the most satisfactory water for industrial reuse except with respect to algal growth potential. This growth potential was significant because neither nitrogen nor phosphorous was reduced by this treatment sequence. The nitrified effluent did have a relatively high potential for phosphorous precipitation, and the scale analysis did show appreciable amounts of calcium and phosphorous. Despite this scaling, the fouling factor was surprisingly low for the nitrified effluent. With phosphorous removal to minimize algae growth and scaling problems, biological oxidation and nitrification to remove organics and ammonia, and filtration following nitrification to reduce suspended solids and improve process reliability, a suitable renovated water can be produced for industrial cooling purposes. In the event that total nitrogen is limited to receiving bodies of water, biological denitrification may also be necessary.

Activated carbon treatment tended to increase the fouling factors but had little or no effect on decreasing the corrosion rates of the renovated waters. Since it did not appear necessary to remove organic materials to reduce toxicity, the use of activated carbon treatment for general industrial water supply does not appear to be necessary. Where specific uses call for low organic carbon in the water, such as may be required for boiler feedwater, it would probably be most economical for the industry concerned to supply the additional treatment necessary. Biological denitrification and activated carbon treatment may also be important if the renovated water is to be demineralized and used for boiler feed or process purposes.

Section VI

ESTIMATED WASTEWATER TREATMENT COSTS

This section presents cost estimates for 30-mgd plants using the treatment processes investigated during the pilot-demonstration studies. These cost estimates can serve as an aid in the comparison of alternative treatment procedures; they are based on assumed average conditions for land, easements, site preparation, and engineering. Actual costs for specific treatment facilities, such as those for the Central Contra Costa Sanitary District, will differ somewhat because of factors peculiar to the individual construction site. The costs discussed below are for treatment facilities only; they do not include the cost of administration and maintenance buildings, utility tunnels and discharge lines, or storm water treatment, since these facilities would be common to all of the treatment systems.

Figure 6-1 indicates the estimated treatment costs, in 1972 dollars, for each of the three phases of the study. A cost index of 185.0, as published by the EPA for wastewater treatment plants, was used to adjust cost data presented by others (References 7, 8, 9, and 10). Amortization costs were based on capital recovery at 6-percent interest over a 25-year period. For the purpose of cost comparison, no federal or state grants were assumed. It is emphasized that the costs presented in this section may not be directly related to water renovation costs because in many cases considerable treatment is required simply to discharge the effluent to receiving bodies of water. In such cases, the costs associated

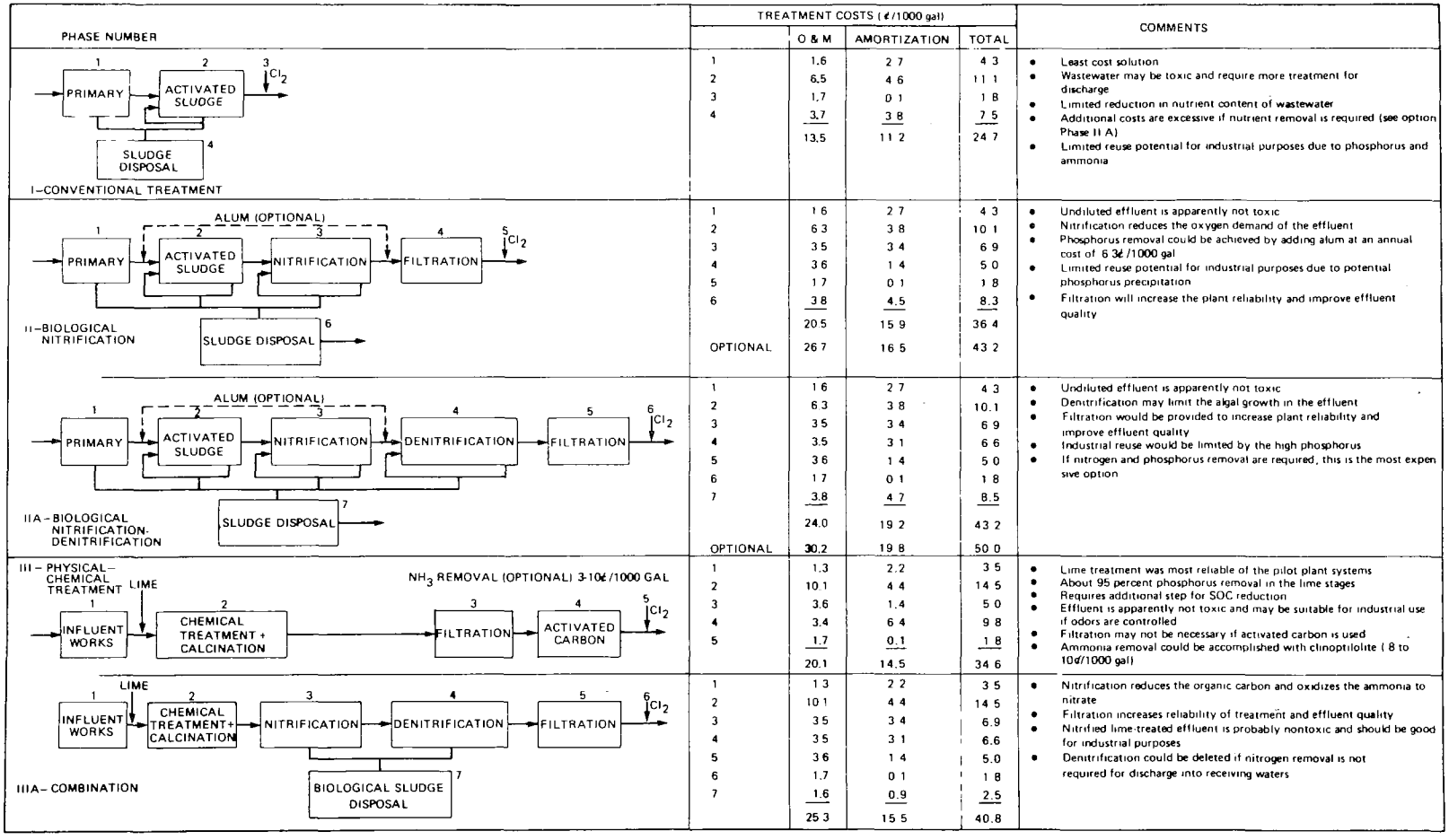

Figure 6-1. Flow Diagrams and Treatment Costs for 30-mgd Plants

with wastewater treatment should be deducted from the water renovation costs.

General process descriptions of the alternatives considered in the cost estimates are presented below. Advantages and disadvantages of the various alternatives are also discussed.

PHASE I: ACTIVATED SLUDGE

The first alternative considered was a conventional activated sludge plant consisting of primary and activated sludge treatment along with sludge dewatering and incineration. This alternative was the least expensive treatment sequence having water costs for a 30-mgd plant of approximately 24.7 cents per thousand gallons at capacity. Although this plant should remove approximately 90 percent of the suspended solids and BOD, the nutrient content of the effluent, especially ammonia and phosphorous, would support appreciable algal growth. Because of the chlorine demands associated with the ammonia, the potential for algal growth, and the likelihood of precipitation of calcium phosphate, additional treatment processes would be required to produce an effluent suitable for direct industrial cooling water use. It is possible, however, that an industrial user could provide the additional treatment necessary.

PHASE II: BIOLOGICAL NITRIFICATION

This system includes the basic plant considered in Phase I with the addition of a nitrification stage to oxidize the ammonia to nitrate, followed by

filtration to increase the overall reliability of the plant and improve the renovated water quality. The detention time in the activated sludge unit (i.e., the first stage) would be reduced so there would be a higher organic carbon concentration in the nitrification reactor influent and the treatment costs for this stage can be reduced. As with the Phase I alternative, sludge disposal would consist of dewatering and incineration. Costs for this treatment system were estimated at 36.4 cents per thousand gallons.

For phosphorous removal, it would be possible to use alum in conjunction with the biological treatment processes, as indicated in Figure 6-1, Phase II. Annual operating costs for this treatment would be approximately 6.8 cents per thousand gallons in addition to the above mentioned costs, resulting in a total cost of 43.2 cents per thousand gallons. This additional cost represents the cost for the alum feeding system as well as the additional sludge disposal costs.

With phosphorus removal, the effluent produced with this treatment system should, in most cases, be suitable for direct reuse by industry for cooling water makeup. Consideration must be given, however, to the increase in TDS, specifically sulfate, resulting from the use of alum for phosphorus removal.

PHASE II A: BIOLOGICAL NITRIFICATION-DENITRIFICATION

This option is identical to that discussed under Phase II except the additional step of denitrification has been added to remove inorganic nitrogen from the effluent. The additional cost for this option would be about 6.6 cents per thousand gallons. Included in these costs would be the denitrification reactor, an aeration basin, and a settling basin. Aeration is provided to aid in the sedimentation of the denitrification solids and to biologically oxidize any excess methanol.

Sludge disposal costs for this three-stage system were increased to account for the additional sludge produced during the denitrification reaction. The total cost for this system was estimated to be 43.2 cents per thousand gallons. As discussed for biological nitrification, alum could be added to the treatment system for phosphorous removal at an additional cost of about 6.8 cents per thousand gallons.

As with the Phase II alternative, the effluent produced with the treatment system including biological denitrification should be suitable for industrial reuse. The nitrogen removal step should reduce the algae growth potential in industrial cooling towers. However, with phosphorous removal and typical industrial cooling water treatment, the need for biological denitrification does not appear justified. This treatment step may be necessary in some cases when the effluent is discharged to a receiving body of water.

PHASE III: PHYSICAL-CHEMICAL TREATMENT

The physical-chemical treatment system considered for the Phase III alternative consisted of two-stage chemical treatment followed by filtration and activated carbon adsorption. Lime would be added to the first chemical treatment stage, the effluent recarbonated, and the precipitated calcium carbonate removed in the second stage. The sludges produced would be dewatered, classified, and recalcined or incinerated. Recalcined materials would be reused in the first stage of the chemical treatment process.

This treatment sequence has been estimated to cost 34.6 cents per thousand gallons. For an additional 8 to 10 cents per thousand gallons, clinoptilolite could be used to remove ammonia, while ammonia stripping would cost between 3 and 5 cents per thousand gallons treated. Ammonia removal would be very desirable in conjunction with the industrial reuse of the water produced.

Based on the observed limited removals of turbidity and TOC in the filtration process during the pilot plant studies, it might be possible to eliminate this process step and use the activated carbon columns to remove the small amounts of suspended material carried over from the chemical treatment process. Also, a single-stage chemical treatment process may be satisfactory for wastewater having relatively high hardness concentrations. If either the filtration process, second-stage chemical treatment process, or both could be eliminated, the treatment cost would be significantly reduced.

Noxious odors that developed in the pilot plant activated carbon columns indicated the need to develop a satisfactory odor control procedure. The costs for such a procedure would be expected to be less than 1.0 cent per thousand gallons, but the procedure must be satisfactorily developed before this alternative becomes suitable.

PHASE III A: COMBINED PHYSICAL-CHEMICAL-BIOLOGICAL TREATMENT

An alternative to the independent physical-chemical treatment sequence described for Phase III would be the use of a combined physical-chemical biological treatment sequence. Based on the results of the pilot plant and industrial test loop studies, chemical treatment, as described for the Phase III alternative, preceding biological treatment, could provide definite advantages. Since chemically treated wastewater is too high in organic carbon content (BOD) for reuse or discharge, either activated carbon or biological oxidation is required to reduce the organic carbon concentration. Biological nitrification in conjunction with oxidation of the remaining organic materials could be used. Such a system would also provide the advantage of phosphorous removal. If discharge limitations require inorganic nitrogen removal, this could be accomplished by biological denitrification, as described under Phase II A. To

assure effluent quality and increase process reliability, filtration would also be desirable. Sludge disposal consisting of dewatering, recalcination, and incineration would also be included.

The cost for the Phase III A alternative described above and shown in Figure 6-1, is approximately 40.8 cents per thousand gallons. This cost is nearly the same as that for the Phase III alternative with ammonia removal included. As discussed for the Phase III alternative, if a single-stage chemical treatment system can be used, the cost will be reduced. The effluent produced with this treatment sequence – in which phosphorous nitrogen, organics, and suspended solids are removed – should be suitable for direct reuse by industry for cooling water makeup.

Section VII

ACKNOWLEDGEMENTS

The assistance and cooperation provided by the Boards of Directors and staffs of the Central Contra Costa Sanitary District and the Contra Costa County Water District are gratefully acknowledged. The support of the Environmental Protection Agency and the help provided by Dr. Carl Brunner, Project Officer, were sincerely appreciated.

Section VIII

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Section IX

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Appendix A

ANALYTICAL PROCEDURES

This appendix includes a description of analytical procedures that differ from those of Reference 2 and a tabulation of the sampling frequencies for the various analytical procedures employed. Sample analysis generally followed the schedule outlined in this appendix. On some occasions, the schedule was modified due to process upsets or analytical backlog.

DETERMINATION OF MERCURY

The emergence of mercury as a significant environmental contaminant stimulated the need for the development of methods for its extraction or separation from a wide variety of substances, including reclaimed wastewater. In view of the fact that it is a cumulative-type poison, mercury is of particular importance when considering the use of reclaimed sewage as an eventual source of potable and irrigational waters.

It was the purpose of this analytical program to determine whether mercury was present in sewage and, if so, to what extent it was removed by conventional sewage treatment processes. Therefore, it was imperative to develop a reasonable method for the determination of mercury in sewage.

Summary of Method

Sewage samples were taken periodically, either as 24-hour composites or as hourly samples, and placed in a metal-free sulfuric-nitric acid solution

to promote dissolution of metallic compounds and to prevent sorption of the metals on the walls of the collection vessel. Glass was the preferred material for the collection vessel, and great care was taken to avoid contamination of the sample. All containers and utensils making contact with the sample were thoroughly washed, acid cleaned (metal free), and double rinsed with deionized water.

The acidified sewage sample was blended for 2 minutes or until completely homogenized. An appropriate aliquot (50 ml) of the sample was digested in a reflux condenser for at least 30 minutes. Glass beads were used to prevent bumping and excessive foaming during digestion. The condenser unit was highly efficient so as to return any mercury that had been vaporized. After digestion was completed, the condenser was rinsed thoroughly with dilute nitric acid (50 ml) to remove any mercury adhering to the walls, and the digest was filtered through a glass-fiber filter to remove any organic residue. Provision was made for any dilution with the rinse water.

Mercury was determined by a spectrophotometer procedure where the sample was wet ashed or digested with nitric acid-chromium trioxide. Potential interferences were eliminated by adding ethylene glycol monomethyl ether and EDTA. An iodide complex was formed that reacted with crystal violet and was extracted with toluene. The absorbance of the extracted crystal violet complex was determined at 605 millimicrons (mμ), and the mercury concentration was determined by comparison with standard mercury solutions. The detection limit was 0.25 micrograms of mercury.

Procedure

The following apparatus and reagents were used:

- Apparatus
  - 300-ml standard tapered (24/40) Erlenmeyer flasks
  - 250-ml glass stoppered separatory funnels
  - Reflux condensers
  - Hot plate
  - Spectrophotometer (capable of reading absorbance in the 600 mμ range)
- Reagents
  - Hydrogen peroxide, 30 percent
  - Nitric acid, 10 N containing 1-percent chromium trioxide
  - Potassium permanganate, 5 percent
  - Hydrochloric acid, 5 N
  - Toluene
  - Sodium metabisulphite – $Na_2S_2O_5$, 20 percent
  - Potassium iodide, 2.5 percent
  - EDTA – disodium salt, 5 percent
  - Crystal violet, 0.1- and 1-percent solution (Dissolve 1 g of crystal violet in 1 ml of ethylene glycol monomethyl ether. This solution diluted [10-100] with distilled water was used in the determination.)
  - Mercury stock solution (Dissolve 135 mg of $HgCl_2$ in 100 ml of distilled water, add 2 ml of concentrated HCl, and dilute to 200 ml [1 ml = 0.5 mg]. Working standard was made by diluting [1 ml to 1000] with distilled water [1ml = 0.5 g]. The working standard was made fresh daily.)

Analysis. Three ml of 10 N nitric acid containing 1-percent chromium trioxide were added to a 100-ml aliquot of the completely mixed wastewater sample. The flask was attached to a reflux condenser (or lightly stoppered with a glass stopper using a Teflon shim) and placed on a hot plate set at 250°F. After 30 minutes of refluxing, the sample was removed, 20 ml of 5-percent potassium permanganate were added, and the sample was refluxed again for 15 minutes. The flasks were removed and 30-percent hydrogen peroxide was added dropwise to react with excess permanganate and any manganese dioxide formed. One ml of excess hydrogen peroxide was added, and the digestion was continued for an additional 30 minutes. The inside walls of the condenser were rinsed with a small amount of distilled water before the flask was removed and cooled to room temperature. Five ml of 5 N HCl and 1 ml of 2.5-percent KI were added to the flasks. A few seconds were allowed for the iodide complex to form, then 20-percent sodium metabisulphite was added dropwise to reduce the excess iodine, adding five drops in excess (should have odor of SO_2). Then 2 ml of 5-percent disodium EDTA were added and thoroughly mixed with the sample. The contents of the flasks were transferred to a 250-ml separatory funnel, where 5 ml of 0.1-percent crystal violet solution was added and mixed in to form an emerald green color. If blue or very dark green colors developed, 2 to 5 ml of 5 N HCl were added. Five ml of toluene were added and the stoppered flask shaken gently 10 times, repeating the shaking after coalescence of drops. After total coalescence of droplets, the bottom (water layer) was drained out. The stem of the separatory funnel was dried, and a plug was inserted to remove suspended materials. The toluene layer was run directly into a 1-cm cell, and the absorbance was read at 605 mμ within 20 minutes.

A standard curve was prepared according to the above procedure by adding 0, 0.5, 1, 2.5, and 5 μg of mercury to 100 ml of the wastewater.

Discussion. It was found that the amount of nitric acid-chromium trioxide digestion mixture was critical. Too large an excess after digestion of organic material had a fading effect on the crystal violet-mercury color complex, with subsequent loss of sensitivity. The 3-ml addition of nitric acid outlined in the method in conjunction with the 20 ml of 5-percent permanganate and 30-percent peroxide should be ample for complete digestion of the organic material found in normal wastewater. If not, it is recommended that digestion be continued with 30-percent hydrogen peroxide.

The above procedure can be used for potable water by using 1 ml of nitric acid-chromium trioxide mixture instead of 3 ml and reducing the digestion time by one-half.

There were no significant differences observed in the partition effects when the crystal violet-Hg complex was extracted with toluene from 75 ml instead of 150 ml of total solution.

It is important that glassware used for the mercury determination be cleaned with either diluted chromic or nitric acid and rinsed with distilled water before attempting the analysis.

There were no significant mercury losses during the digestion period. Wastewater samples spiked with increasing amounts of mercury gave the results listed in Table A-1.

Table A-1

RECOVERY OF MERCURY FROM 100 ML OF WASTEWATER

Mercury Added (μg)	Mercury Found (μg)	% Recovered
0.0	0.00	
0.5	0.49	98.0
1.0	0.94	94.0
2.5	2.45	98.0
5.0	5.05	101.0

DETERMINATION OF SELENIUM IN WASTEWATER*

Summary of Method

The method used involved simultaneous evaporation and oxidation of the sample with hydrogen peroxide in an alkaline medium. The residue was further oxidized by taking up with concentrated HNO_3 and evaporated to dryness. The selenate that was formed was reduced to selenite with concentrated HCl. Reaction with diaminobenzidine produced a yellow colored compound extractable with toluene, and the absorbance was determined at 420 mμ with a spectrophotometer.

An average standard deviation of $\pm$ 0.001 mg/l was found by spiking five samples with 10 micrograms of selenium and another five samples with 30 micrograms of selenium.

Procedure (Reference 2)

To 1000 ml of the sample in a 1500-ml beaker were added 5 ml of 0.1 N NaOH, 5 ml of calcium chloride solution, and 10 ml of 30 percent H_2O_2. A few boiling chips were added, and the solution was placed on a hot plate and evaporated just to dryness. Ten ml of concentrated HNO_3 were added to oxidize any remaining organic material (evaporation to dryness should take place on a hot plate at low heat or on a steam bath). The sides of the beaker were rinsed with approximately 10 ml of distilled water, and the solution was evaporated to dryness. The residue was cooled; 5 ml of concentrated HCl were added, followed by 10 ml NH_4Cl solution; and the mixture was heated on a steam bath for 10 $\pm$ 0.5 minutes. The warm solution and precipitate, if any, were transferred to a graduated 100-ml beaker suitable for pH adjustment; the larger beaker was rinsed with 5 ml of EDTA-sulfate reagent and 5-ml of 5 N NH_4OH. The pH was adjusted

* Modified Diaminobenzidine Method A

to 1.5 ± 0.3 with NH_4OH. One ml of diaminobenzidine solution was added, and the mixture was heated on a steam bath for approximately 5 minutes and cooled and adjusted to pH 8 ± 1 with NH_4OH. The volume was then adjusted to 50 ml.

The contents were poured into a 250-ml separatory funnel with 10 ml of toluene and shaken for approximately 30 seconds. After the lower aqueous layer was drained, the stem of the separatory funnel was dried and a cotton plug inserted. The toluene was allowed to run into a 1-cm cuvette, and the absorbance was determined at 420 mμ. If there was any difficulty in obtaining two distinct layers, as much of the bottom layer as possible was drained. The remaining material was transferred to a centrifuge tube and centrifuged for approximately 3 minutes, or until a definitely clear toluene layer was obtained. (If no centrifuge is available, the upper layer could be filtered through paper containing anhydrous sodium sulfate.)

A calibration curve was made by spiking approximately 500 ml of the sample with 0, 10, 20, 30, and 40 micrograms of selenium. The stock selenium solution was made by dissolving an accurately weighed amount of reagent grade selenium in 5 ml of concentrated HNO_3. The solution was heated to dryness and diluted to 1000 ml with distilled water. Appropriate dilutions of the stock solution resulted in a standard solution such that 1 ml was equivalent to 1.0 microgram of selenium.

DETERMINATION OF TRACE METALS BY EMISSION SPECTROSCOPY

Summary of Method

Emission spectrography was used as a means of determining total trace metal concentration. Samples were treated with sulfuric acid and dried at 600°C before being weighed and arced using standard spectographic

techniques. These analyses, coupled with atomic absorption results, gave values for both soluble and insoluble metal content necessary for the investigation of treatment removal efficiencies.

Procedure (Reference 11)

Two ml of concentrated H_2SO_4 were added to a 400-ml water or wastewater sample of known total solids concentration. The sample was partially dried on a water bath. A hot plate was used to dry the sample completely by heating it under a hood at 600°C. After 30 minutes, the residue was scraped with a plastic spatula. A 10-mg residue sample was weighed and analyzed using a DC arc in accordance with ASTM methods for spectrographic determination of solids.

Table A-2

SAMPLING AND ANALYSIS SCHEDULE
ACTIVATED SLUDGE PROCESS

Analysis*	Activated Sludge			Filter Effluent	Activated Carbon Effluent	
	Influent	Mixed Liquor	Effluent		1st	2nd
Turbidity	D		D	D	D	D
pH	D		D		D	D
Conductivity	D		D	D	D	D
Total organic carbon	D		D	D	D	D
Soluble organic carbon	D		D	D	D	D
Ammonia	D		D	D		D
Nitrate	D		D	D		D
Total Kjeldahl nitrogen	W		W	W		W
Ortho-phosphate	D		D	D		D
Total phosphate	D		D	D		D
Total hardness	W		W			
Calcium hardness	W		W			
Total alkalinity	W		W			
Chloride	W		W			
Sulfate	W		W			
Silica	W		W			
Settleable solids	D	D	D			
Suspended solids	D	D	D			
Volatile suspended solids	D	D	D			
Dissolved solids						
Dissolved oxygen		D				
Coliform count			W	D		D
Chemical oxygen demand	W		W			W
Biochemical oxygen demand	W			W	W	W
Temperature		D				

D = Daily T = Three times/week W = Weekly

* All samples were hourly samples composited on a daily basis except for the mixed liquor sample, which was a daily "grab" sample.

Table A-3

SAMPLING AND ANALYSIS SCHEDULE NITRIFICATION/DENITRIFICATION PROCESSES

Analysis*	CCCSD		Nitrification			Denitrification		Filter Effluent	Activated Carbon Effluent	
	Primary Influent	Primary Effluent	Influent	Mixed Liquor	Effluent	Mixed Liquor	Effluent		1st	2nd
Turbidity			D		D		D	D	D	D
pH	T	T	T		D		D			T
Conductivity	T	T	T				T			T
Total organic carbon	T	T	T		T		T	T	T	T
Soluble organic carbon	T	T	T		T		T	T	T	T
Ammonia			D		D		D	W		W
Nitrate			D		D		D	W		W
Ortho-phosphate	T		T		T		T			T
Total phosphate	W		W		W		W	W		W
Total hardness							W			W
Calcium hardness							W			W
Total alkalinity							W			W
Chloride										W
Sulfate										W
Silica										W
Settleable solids	T	T		D		D				
Suspended solids	W	W	T	D	T	D	T	W		W
Volatile suspended solids				W		W				
Dissolved solids	W									W

D Daily T Three times/week W Weekly

All samples were hourly samples composited on a daily basis except for the mixed liquor samples, which were daily "grab" samples.

Table A-4

SAMPLING AND ANALYSIS SCHEDULE
PHYSICAL/CHEMICAL PROCESSES

Analysis*	Flocculation			Filter Effluent	Activated Carbon Effluent	
	Influent		Effluent			
	1st	2nd	2nd		1st	2nd
Turbidity		D	D	D	D	D
pH	T	D	D	T		T
Conductivity	T	D	D	T		T
Total organic carbon	T	T	T	T	T	T
Soluble organic carbon	T	T	T	T	T	T
Ammonia	W		W			W
Nitrate	W		W			W
Ortho-phosphate	T	T	T			T
Total phosphate	W	W	W	W		W
Total hardness	W	W	W			W
Calcium hardness	W	W	W			W
Total alkalinity	W	W	W			W
Chloride	W					W
Sulfate	W					W
Silica	W					W
Settleable solids						
Suspended solids	T	T	T	W		W
Volatile suspended solids	W	W				
Dissolved solids	W	W	W			

D = Daily T = Three times/week W = Weekly

* All samples were hourly samples composited on a daily basis.

Table A-5

SAMPLING AND ANALYSIS SCHEDULE
INDUSTRIAL TEST LOOPS

Analysis*	Circulating Water			Boiler Blow-down	Softener Effluent	Con-densate	Canal Water
	Tower No. 1	Tower No. 2	Tower No. 3				
Turbidity	T	T	T			W	W
pH	D	D	D	W	W	W	
Conductivity	D	D	D	W	W	W	W
Total organic carbon	W	W	W			W	W
Ammonia	W	W	W			W	W
Nitrate	W	W	W				
Ortho-phosphate	W	W	W				W
Total phosphate	W	W	W	W			
Total hardness	W	W	W		W		W
Calcium hardness	W	W	W		W		W
Total alkalinity	W	W	W	W	W		W
Chloride	W	W	W				W
Sulfate	W	W	W				W
Silica	W	W	W				W
Sulfite				W			
Suspended solids	W	W	W				W
Corrator	D	D	D				
Temperature	D	D	D				

D = Daily T = Three times/week W = Weekly

* All samples were hourly samples composited on a daily basis.

Appendix B

TRACE METALS

This appendix contains a summary of a separate study concerned with the removal of heavy metals in the various treatment stages of a physical-chemical pilot treatment facility. The treatment plant influent was dosed with a mixture of heavy metals, and their removal was monitored through the treatment process. Although this study was not within the scope of work of the project, the results are included here for informational purposes. A complete discussion of the data may be found in a paper by Yerachmiel Argaman and Clark L. Weddle (Reference 12).

Table B-1

SUMMARY OF HEAVY METALS TESTING PROGRAM

Run No.	Heavy Metals Added	Point of Addition	NTA Added
1	Zn 10 mg/l, Mn 2.0 mg/l, others* 0.5 mg/l	Ahead of lime coagulation	0
2	Zn 10 mg/l, Mn 2.0 mg/l, others - 2.0 mg/l	Ahead of lime coagulation	0
3	Zn 10 mg/l, Mn 2.0 mg/l, others 2.0 mg/l	Ahead of lime coagulation	10.0 mg/l
4	As, Cd, Cu, Hg 10.0 mg/l, Mn 2.0 mg/l	Ahead of lime coagulation	0
5	As, Cd, Cu 10 mg/l, Hg 5 mg/l, Mn 20.0 mg/l	Ahead of lime coagulation	0
6	Zn 10 mg/l, Mn 2.0 mg/l, others 2.0 mg/l	Ahead of ferric coagulation (2nd stage)	0
7	Zn - 10 mg/l, Mn 2.0 mg/l, others 2.0 mg/l	Ahead of filtration	0

*Other metals added were: Ag, As, Ba, Cd, Co, Cr, Cu, Hg, Ni, Pb

Table B-2

REMOVAL OF HEAVY METALS BY LIME COAGULATION, SETTLING, AND RECARBONATION
(In and Out Concentrations in mg/l)

Metal	Run #1			Run #2			Run #3 (NTA)			Run #4			Run #5		
	In	Out	% rem	In	Out	% rem	In	Out	% rem	In	Out	% rem	In	Out	% rem
Ag	0.04	0.01	96	1.51	0.02	99	1.20	0.06	95						
As										8.40	0.30	96	7.00	0.20	97
Ba	0.36	0.04	89	1.08	0.14	87	1.48	0.06	96						
Cd	0.54	0.01	98	1.42	0.02	99	1.48	0.19	87	4.00	0.19	95	5.78	0.13	98
Co	0.42	0.04	90	1.29	0.05	96	2.07	1.58	24						
Cr	0.45	0.30	33	1.40	1.25	11	2.95	2.53	14						
Cu	0.60	0.04	93	1.47	0.23	84	0.71	0.32	55	4.60	0.31	93	4.60	0.20	96
Hg										4.45	0.61	86	3.26	0.29	91
Mn	2.26	0.02	99	1.37	0.01	99	2.37	1.27	46						
Ni	0.75	0.11	85	1.36	0.20	85	1.48	1.27	14						
Pb	0.41	0.04	90	1.21	0.05	96	1.48	0.25	83						
Zn	9.61	0.12	99	7.34	0.18	97	10.00	1.58	84						

Table B-3

REMOVAL OF HEAVY METALS BY (SECONDARY)
FERRIC CHLORIDE COAGULATION AND SETTLING
(In and Out Concentrations in mg/l)

Metal	Run #1			Run #2			Run #3			Run #4			Run #5			Run #6		
	In	Out	% rem	In	Out	% rem	In	Out	% rem	In	Out	% rem	In	Out	% rem	In	Out	% rem
Ag	0.01	0.01		0.02	0.01	50	0.06	0.02	67							3.30	0.09	97
As										0.30	0.01	97	0.20	0.01	95			
Ba	0.04	0.03	25	0.14	0.07	50	0.06	0.09								2.30	0.23	90
Cd	0.01	0.01		0.02	0.01	50	0.19	0.01	95	0.19	0.04	79	0.13	0.03	77	3.90	0.29	92
Co	0.04	0.017	58	0.05	0.02	60	1.58	0.49	69							3.30	1.40	58
Cr	0.30	0.07	72	1.25	0.63	50	2.53	0.65	78							6.60	3.40	48
Cu	0.04	0.03	25	0.23	0.02	56	0.32	0.29	9	0.31	0.32	-	0.20	0.23	-	1.30	0.17	87
Hg										0.61	0.28	54	0.29	0.03	90			
Mn	0.02	0.02		0.01	0.01		1.27	0.19	85							3.90	2.30	41
Ni	0.11	0.09	18	0.20	0.15	25	1.27	0.66	48							4.60	1.10	76
Pb	0.04	0.04		0.05	0.023	54	0.25	0.12	52							2.30	0.23	90
Zn	0.12	0.13		0.18	0.04	78	1.58	0.53	66							9.90	1.0	90

Table B-4

REMOVAL OF HEAVY METALS BY
FILTRATION AND CARBON ADSORPTION
(In and Out Concentration in mg/l)

Metal	Dual Media Filtration						Activated Carbon Adsorption					
	Run #6			Run #7			Run #6			Run #7		
	In	Out	% rem	In	Out	% rem	In	Out	% rem	In	Out	% rem
Ag	0.09	0.02	78	1.90	0.02	99	0.02	0.01	50	0.02	0.01	50
Ba	0.23	0.16	30	1.90	0.05	97	0.16	0.02	88	0.05	0.01	80
Cd	0.29	0.01	96	2.10	0.02	99	0.01	ND		0.02	0.01	50
Co	1.40	0.16	89	1.90	0.48	75	0.16	0.014	91	0.48	0.004	99
Cr	3.40	0.83	76	2.70	1.04	61	0.83	0.01	99	1.04	0.01	99
Cu	0.17	0.24		0.54	0.28	48	0.24	0.01	96	0.28	0.01	96
Mn	2.30	0.27	88	2.70	0.29	89	0.27	0.01	96	0.29	0.28	34
Ni	1.10	0.41	63	1.60	0.51	68	0.41	0.0014	97	0.51	0.12	76
Pb	0.23	0.03	87	1.40	0.02	99	0.03	0.04		0.02	0.0002	90
Zn	0.04	0.03	25	4.30	0.05	99	0.03	0.03		0.05	0.02	60

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16. Abstract

Three pilot plant treatment sequences were operated during this study to produce various grades of effluent for subsequent testing as industrial water sources. The testing was conducted in pilot-sized test loops consisting of small cooling towers and heat exchangers. At the same time the renovated waters were tested, Contra Costa Canal water, which is presently used by industry in the study area, was also investigated in a test-loop identical to those used for the renovated water.

The study results illustrated that the wastewater investigated can be treated satisfactorily for reuse in industrial applications. Corrosion rates and fouling factors observed with renovated water were equal to or less than found with the canal water. Precipitation of phosphorous was the major source of scale formation while using renovated water for cooling purposes, thus indicating the need for phosphorous removal.

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