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RECYCLE OF NITRIC ACID
AND ALUMINUM NITRATE

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INTRODUCTION

The Oak Ridge Y-12 Plant is located in Oak Ridge, Tennessee, and is owned by the U.S. Department of Energy (USDOE) and is managed by Martin Marietta Energy Systems, Inc. The plant's major responsibilities are (a) producing components for weapons systems and supporting DOE's design laboratories, (b) processing special materials (c) supporting other Energy Systems installations, and (d) supporting other DOE plants and other agencies of the U.S. government. One of the plant's programs is purification and recycle of non-irradiated enriched uranium. A unit operation in the uranium recycle process is solvent extraction of the removal of impurities. For this operation, some of the recycle uranium is dissolved in nitric acid which is then solvent extracted. Two types of acidic nitrate wastes are generated from the extraction process; one of which is a dilute nitric acid waste, called condensate, which is produced from several evaporation operations. This waste may contain up to 10 wt. % nitric acid with trace amounts of impurities such as organic carbon, chlorides and fluorides. The acid must be concentrated to 30 or more wt. % and the impurities require removal before the nitric acid can be reused in the plant. A second waste, called raffinate, is also produced from the extraction processes. The raffinate waste contains high levels (more than 10 wt. %) of aluminum nitrate which is used in the extraction process. It also contains most of the impurities extracted from the uranium. Most of the impurities must leave the extraction process in either the condensate or raffinate stream.

In the early 1950's, four unlined waste ponds were constructed to receive the acidic wastes from the extraction process. Other acidic wastes from non-enriched uranium processes, such as pickling baths, were also discharged into the ponds. The four ponds are located near the headwaters of two small streams: Bear Creek and the East Fork of Poplar Creek. The ponds had no surface drainage but relied on underground seepage to prevent direct overflow to the creeks. The ponds were located close to the beginning points of both creeks where the surface flow rate in either creek is usually low

to non-existent. Even at a distance of several hundred yards from the ponds, the water flow in Bear Creek is zero for much of the year. About two miles from the ponds at the exit point of the plant, the East Fork of Poplar Creek has a typical daily flow rate of seven million gallons of water per day (18,400 l/min) but most of the water is due to discharge from cooling systems within the plant.

In the early 1970's, a decision was made to develop and install processes, to recover as much acid and aluminum nitrate as practical from the wastes generated by the solvent extraction processes (Figure 2). The remaining wastes which could not be recycled would be treated to remove the environmentally objectionable compounds. A later decision in the early 1980's was made to develop and install in-situ treatment processes for the liquid wastes in the four ponds and for similar wastes that might be generated in the future.

Recycle of Condensates - One of the waste streams was a dilute nitric acid stream (condensate) which contained trace amounts of impurities. In order to recycle the acid, it had to be concentrated to at least 30 wt. % and most of the impurities had to be removed. The increase of the impurities (i.e., chlorides and fluorides) during the evaporation and concentration of the dilute nitric acid caused major problems related to the materials of construction of the distillation equipment. Also, the trace quantities of certain organic impurities created potential safety problems due to their reactivity with the hot, concentrated nitric acid product.

The condensate waste contains up to 10 wt. % nitric acid plus minor levels of fluoride and chloride (Figure 3). The selection of the materials of construction for the process equipment required a development program. In the distillation process (Figure 4 photo 174437) the minor levels of chloride and fluoride impurities are also concentrated to high levels at some location in the distillation column. A metal column, such as stainless steel, corrodes at a rapid rate at the location where these

impurities are concentrated. It is, therefore, required that these ions be removed before the distillation process.

A molten salt trap was developed to remove most of the fluoride ions from the incoming condensate wastes and also served as a heater for vaporization of the incoming feed to the column. Tests showed that the fluoride ions, as well as most of the metallic ions, could be removed by passing this stream through a hot bath of aluminum-calcium nitrate.

The salt trap (Figure 5) contained 23.8 wt. % aluminum nitrate nanohydrate, 52.9 wt. % calcium nitrate tetrahydrate and 23.3 wt. % water. The incoming dilute condensate is passed through the molten salt bath which is heated to a nominal temperature of 110°C and the fluoride ions react with aluminum ions until a 5 to 1 aluminum to fluoride weight ratio is obtained (i.e., 4800 mg/L fluoride ion concentration is usually a maximum in the trap). The fluoride salt bath also traps all metallic ions in the condensate wastes. Enriched uranium in the condensate is typically at a concentration of 0.1 to 0.5 mg/L uranium. The bath removes all of the enriched uranium and the uranium is later recovered and recycled back to the plant. The fluoride salt bath is constructed of 304L stainless steel and equipment corrosion is not a problem because the fluoride ions react with the aluminum ions in the salt bath.

The vapors from the salt bath are fed to a 50 ft. long glass distillation column which has a steam heater encased in tantalum in the re-boiler. Originally the acid was to be concentrated to 50 wt. % but trace amounts of organics from the solvent extraction process were found to be present. To prevent an organic-hot nitric acid reaction in the column, a decision was made to produce a 30 wt. % nitric product. No organic reaction is known to occur under these conditions. Most of the chloride ions remain in the 30 wt. % nitric acid product and an ozonation step was added to remove

this impurity from the product. Ozone gas at 60 to 70°C is passed through a 250 gallon batch of 30% acid until all of the chloride ions are removed in the overhead vapors which are cooled and released to the environment. The ozonation step also removes all of the organic impurities which may be present in the product. Since 1976, most of the nitric acid condensate from the main process facility has been recovered. The amounts of nitric acid purchased by the plant decreased approximately 30% because of this recycle stream. The overhead vapors from the distillation column is cooled and the water is discharged through a permitted discharge point.

The recycled acid has practically no impurities (Figure 6). Chlorides are less than 10 mg/L, fluorides less than 1 mg/L and uranium plus other metallic ions are essentially 0 mg/L. The amount of recovered acid from the condensate wastes exceeds 97.4%.

Recycle of Aluminum Nitrate - Typical chemical analysis of raffinate wastes are shown in Figure 7. As noted in Figure 8 (photo 174439), the raffinate waste is evaporated under vacuum at 60°C to concentrate the aluminum nitrate and to remove the free nitric acid in the overhead condensate. The acid is returned to the distillation process and is recycled to the plant. The density of the raffinate in the evaporator is controlled at 1.46 g/mL when measured at 60°C. A stream from the vacuum evaporator is fed to a cooler (30°C) and then into a crystallizer where the aluminum nitrate nanohydrate crystal increase in size. The solution is then put into a batch type centrifuge to remove most of the crystals. The crystal free solution is returned to the evaporator. Part of the solution is taken off as a waste stream to a holding tank where it is diluted with water to about 30 wt. % nitrate ion level and becomes the feed solution for the biological reactor. This waste solution, called mother liquor, contains the impurities from the uranium extraction process.

Approximately 40 to 60 wt. % of the aluminum nitrate and all of the free nitric acid is recycled (Figure 9). The purity of the product is equal to or better than aluminum nitrate nanohydride prepared in the normal procedure (i.e., dissolving aluminum oxide trihydrate in 50 wt. % nitric acid). Recycle of the aluminum nitrate has reduced the purchases of nitric acid and aluminum oxide by about 30%. Future plans are being made to increase the amount of recycled aluminum nitrate and 20% more aluminum nitrate crystals are expected to be recycled without a significant increase in the impurities occurring in the product.

Stirred Tank Bio-reactor process - A nitrate waste stream is generated from the crystallization process. This stream is called "mother liquor" and contains impurities which were retracted from the recycle of uranium. Laboratory and pilot plans tests developed a process using a stirred tank to biologically decompose the mother liquor wastes (Figure 10). Analysis of mother liquor is shown in Figure 11. The liquor is diluted with water to a nominal 30 wt. % nitrate in order to prevent solidification when cooled to room temperature. Biological denitrification is nitrate reduction with the nitrate ions serving as the final electron (hydrogen) acceptor in the oxidation of an organic substrate by facultative bacteria. The chemical reaction noted in Figure 12 is believed to represent the process. Many organic compounds can be used as a food source for the biodenitrification process but laboratory studies showed that calcium acetate was an effective organic food for the bacteria and calcium carbonate was one of the waste products from the reaction. The calcium carbonate in the wastes could be recycled to neutralize incoming acid wastes.

Laboratory Data - Laboratory tests operated four continuous flow stirred tank reactors for a total of 453 days. A maximum denitrification rate of 11.2 g nitrate/day/liter of reactor solution is obtained. The denitrification rate is expressed in units related to the volume of the reactor and not to the number of bacteria because of the difficulties experienced in determining the bacteria count. Because

of the high solids contents of the reactor solution (up to 9 wt. % solids), the number of bacteria could not be accurately determined using dry cell weight measurements.

The laboratory reactors operated 94.4% of the time even though many experiments were conducted to define operational parameters. The volume of gas evolved and its chemical composition were measured. In the calcium rich solutions, the evolved gases were typically 52 wt. % carbon dioxide and 48 wt. % nitrogen. The total volume of gases was 0.45 - 0.48 liters of gas/gram of nitrate destroyed. The carbon dioxide concentration and the total amount of evolved gases are indicators of reactor performance and are used in the production reactors to determine operational problems. No nitrogen oxides were found in the evolved gases except during periods when the reactor was in an upset condition. For information purposes, when neutralized raffinate (calcium nitrate) was fed to the reactor, the carbon dioxide concentration was a nominal 38 wt. % because this feed did not require neutralization. When sodium nitrate was used as a feed to the laboratory studies, a carbon dioxide level of 4% was typical in the evolved gases because most of the carbon dioxide gas remained in the effluent liquid stream as sodium bicarbonate or carbonate. The laboratory studies were concentrated on a stirred tank design but tests using a column as a denitrifier were conducted. Feeds containing sodium in place of calcium were used for these tests to prevent plugging of the column with calcium carbonate.

Production Reactors - Two 25,000 gallon sized reactors (Figure 13, photo 174438) were installed in 1976 and have been successfully operated since that time. Each of these reactors normally decompose 4 grams of nitrate/day/liter of reactor solution (840 lbs NO₃/day/reactor) but rates as high as 6 grams/day/liter of reactor solution have been obtained. Occasional operational problems have been experienced with the feed pumps and PH control. However, the reactors operate more than 95% of the time and all of the down time is normally related to the lack of waste liquor.

Solids Produced in the Reactor - During laboratory tests, the amounts of solids removed from the stirred tank reactors were measured. When a total daily feed rate of 2242 gms of raffinate and calcium acetate were fed to an operating reactor, 275 grams of dried solids were produced. The chemical composition of the dried solids were typically 13.685% calcium, 10.14% aluminum, 0.51% magnesium, and 0.59% iron. The solids contained 6.23% organic carbon and calcium carbonate; the carbon dioxide equivalent in the carbonates was 21.14%. Trace quantities (less than 0.01%) of several other metals were identified in the solids.

Process Equipment - The mother liquor waste to the biodenitrifiers is collected in a 304L stainless steel holding tank. A second holding tank serves as a feed tank for calcium acetate (17 wt. % solution). These liquids are injected at a controlled rate into a mild steel 100,000 liter (25,000 gallon) stirred tank reactor. Any free acid is neutralized in the reactor by excess calcium carbonate which is always present in the reactor. Trace quantities of phosphate ions are also added to the reactor and cooling coils are located inside the reactor to prevent the liquor from heating above 45°C since the neutralization and biological reactions are exothermic. The reactor design is based on a design used for softening water except that it has a closed top to maintain anoxic conditions. The off-gases (nitrogen and carbon dioxide) are vented through a monitoring instrument which measures the amount released to the environment. All liquids and solids which are injected or formed in the reactor are taken out once per day. These solids were then placed into four acid waste ponds to begin neutralization of the waste materials. After closure of the ponds was complete, the liquid and solids are being placed into large reactors (500,000 gallon size) where other nitrate wastes are biologically reduced.

Denitrification Process Parameters - Laboratory studies investigated columnar systems as well as stirred tanks. Because of the high amount of insoluble compounds (aluminum hydroxide and calcium

carbonate), frequent plugs developed in the column and the stirred tanks design was chosen for production operations. Some of the important control parameters are discussed below.

Control of pH - In order for the process to function at high denitrification rates, several parameters must be controlled. The pH of the reactor solution must be above 6.8 with an optimum pH range of 7.3 to 7.5. The mother liquor stream was not neutralized before injecting it into the biodenitrification reactor. Neutralization of the free acid in the liquor was accomplished by its reaction with excess calcium carbonate ions which are formed in the denitrification process. Part of the reactor is used as a neutralization zone and part is used as a biological reactor. Careful control of the reactor pH is required to maintain the biological reaction because it is sensitive to low pH conditions.

Agitation - Agitation of the solution must be present to prevent settling of the bacteria but the rate of agitation does not appear to be important.

Phosphates - Trace amounts of phosphates are required for successful operation of the reactor. Because of the high calcium levels in the reactor, the soluble phosphates are usually 50 mg/L or less. Organic phosphates (trimethyl or triethyl phosphates) as well as several inorganic phosphates have been successfully used.

Temperature - Cooling coils are present in the continuous flow stirred tanks reactor to maintain the temperature below 45°C. The upper temperature limit of the reactor is not known but 45°C appears to be an effective upper limit. At 10°C or below, biological activity is reduced to very low levels and practically no nitrates are destroyed. However, reactor solutions can be cooled to below 10°C and

kept at this temperature for several months without impairing their biological reactivity. When the temperature is raised to above 10°C, biological activity begins within a very short period of time.

Organic Carbon - In laboratory and pilot plant tests, several types of organic compounds were investigated to obtain high denitrification rates. Ethanol, methanol, and calcium acetate solutions were found to produce high denitrification rates. Storage of concentrated ethanol and methanol feeds presented a potential fire hazard problem to the plant. Also calcium ions are required for neutralization of the acid feed. Based on the results of the pilot plant tests, a decision was made to use a 17 wt. % calcium acetate solution as the organic feed solution. A carbon to nitrogen weight ratio of 1.03 to 1.08 must be maintained when calcium acetate is used. In one series of laboratory tests, a reactor was operated several weeks using calcium acetate as the organic source. The reactor feed was changed to methanol and biological activity stopped for several weeks. Calcium acetate was again added to the reactor and the biological activity was immediately restored. These tests confirmed that bacteria, once acclimated to an organic compound, must become reacclimated to a second organic food source. A time delay of several weeks or longer for this reclamation period may be considered to be normal under the operational conditions utilized.

Type of Bacteria - This type of bacteria used in the first laboratory tests was Pseudomonas stutzeri which were obtained from a commercial source. A large batch of these bacteria was cultivated and stored near -20°C for use in the pilot plant tests and for the start-up of the two production sized reactors. After 18 months of continuous operations, one of the production reactors was samples and the bacteria were identified (Figure 14). In a sample, 1.2×10^9 colony-forming units per millimeter of reactor solution were found. There were 16 different isolates that appears to predominate (Figure 15 photo 161519). Fourteen of the 16 isolates were capable of reducing nitrates to nitrogen gas. Fifteen of the 16 isolates were nonfermenting, gram-negative rods characteristic of the family

Pseudomonadacea. Five of the 15 seemed to best belong to the fluorescent members of the Group 1 pseudomonads; eight appeared to belong to the stutzeri subgroup of nonfluorescent Group 1 pseudomonads and one isolate could only be identified as a Pseudomonas species. The remaining bacterium could not be identified to the genus level. Each year, one or more samples are taken from the continuous flow reactors and similar analysis are obtained. In all cases, a mixed population of bacteria has been found and more than 100 species have been identified at various times during these studies.

Dissolved Oxygen - The dissolved oxygen level in the reactor must be less than 2 mg/L for destruction of the nitrate ions. In laboratory and pilot plan tests, a closed reactor design was used to provide an anaerobic condition. During start-up of the reactor, a dissolved oxygen level of 8 mg/L is common. However, the dissolved oxygen is quickly depleted from the reactor solution by the bacteria and then the oxygen is stripped from the nitrates. Later, tests completed in 1983 showed that denitrification in open ponds could be accomplished and the solution in the ponds could be maintained at dissolved oxygen levels of less than 2 mg/L. It is apparent that the presence of trace amounts of oxygen does not materially affect the biodenitrification process.

State of Development - The recycle facilities and the stirred tanks denitrification reactors have operated successfully since 1976.

S-3 Ponds - Four unlined waste ponds (Figure 16 and 17) were used since the mid 1950's to collect nitrate and other liquid wastes from the plant. Figures 18 and 19 (photographs 142559 and 215000) are pictures of the original ponds and Figure 20 lists chemical analyses of the ponds. In 1983, an in-situ treatment process for the ponds was developed (Figure 21) and the wastes were biologically decomposed into nitrogen and carbon dioxide gases. The equipment used consisted of large

recirculating pumps which served as a mixer for the addition of calcium carbonate and hydroxide plus acetic acid. When denitrification was complete, the water was pumped through a precipitation and flocculation process to remove trace amounts of solids and uranium. This flocculation process involved the reduction of the pH to two to remove carbonate ions, the addition of ferric sulfate and the raising of the pH to 10 to induce co-precipitation of ferric hydroxide and other metallic hydroxides or oxides. Solids were returned to the ponds and the purified water was released to the environment. The ponds were subsequently filled with rocks to stabilize the bottom and covered with an approved multilayer cap. A process to treat future nitrate wastes was installed in place of the ponds which included 500,000 gallon sized bio-reactors. After denitrification, the liquids and solids from the bio-reactors are flocculated to remove trace amounts of solids and uranium. The solids are presently stored but plans to delist these solids are being made. A process change will also be made in the future to separate the solids before denitrification. When this change is made, the remaining solids can then be recycled (calcium carbonate) to neutralize incoming wastes.

- Treatment of Waste Pond Liquid - As previously stated, four unlined waste ponds having a combined volume of 10 million gallons were used to receive nitrate wastes from several processes for the southwest pond. Typical analysis obtained in 1983 are shown in Figure 22. The primary source of the nitrates were those emitted from the uranium purification processes which have previously been discussed. In 1983, a decision was made to eliminate the ponds and an in-situ treatment process was developed. Laboratory tests were conducted to develop a process and some data are noted in Figure 23. The southwest pond was then used as a pilot plant test to verify the laboratory tests. The other three ponds were treated using the same process. The developed process consisted of a) neutralization of the ponds, b) settling of the solids, c) denitrification, d) bio-oxidation to remove any excess organics, e) flocculation to remove suspended solids, f) filtration and release through a permitted point. Any solids generated during these operations were turned to the empty ponds.

The first step in the development program was to conduct laboratory tests using open top 55-gallon drums to determine if the bacteria would denitrify in open containers. The laboratory tests were successful and the southwest pond was used as a pilot test for the operations.

Pilot Test on Southwest Pond - The pond had an initial pH of 1.0 to 2.7 and a nominal 8000 mg/L of nitrates. A mixing system was installed and consisted of five valved intakes distributed along the north wall of the pond and the intakes were submerged about 5 ft. The flow from this manifold was pumped by two 40,00 L/min centrifugal pumps to a discharged manifold composed of five valved outlets located on the west wall. Data indicate that the pond was mixed within 24 h. using this system. On the west side in an access road to the pond, a 0.5-m drain originally was used for liquid addition to the pond. For the pilot test, the drain was used for the introduction of chemical reagents into the pond. A slurry tank used for the CaCO_3 addition was composed of a steel funnel fitted inside a 1.7-m cement culvert, positioned over the drain. Flow pumped from the discharge of the pumps was continuously introduced into the drain and intermittent loads of 150 to 200 kg of CaCO_3 were dumped from a front loader into the slurry tank (Figures 24, 25, 26, 27, 28, and 29, photographs 216742, 214445, 214450, 214447, 214444, and 216749). Acetic acid, sodium hydroxide, and the denitrification bacteria were added directly into the drain by tank truck. The phosphoric acid was dumped into the pond before neutralization.

Finely-ground and high-purity but damp calcium carbonate (CaCO_3) was used for the primary neutralization. The limestone was damp to reduce dust problems during handling. The water content was estimated at about 10-20 wt. %. Food-grade acetic acid and phosphoric acid were also added. The final pH adjustments used 50% sodium hydroxide solution and calcium hydroxide. Biodenitrification sludge from the operating production reactor was added as bacteria seed. During neutralization, the first part of the calcium carbonate addition was very efficient, and a pH of 5.0 was

quickly achieved. Several days later, acetic acid was added to the pond. The pH dropped to about 4.0 and a considerable amount of precipitated aluminum hydroxide redissolved. Sodium hydroxide was added as a 50% solution to finish the neutralization. When the biological parameters were met (Figure 30), the bacteria began to denitrify and, over the next 12 days, the pond pH ranged from 6.8 to 7.1. The total required amount of acetic acid was not initially added because of shipment delays and when it was added, the pH dropped to 5.9 because of the addition of large amounts of acetic acid. Biological activity stopped, but in one week the pH rose to a steady value of 6.4. Calcium hydroxide was added over the next several days, until the pH rose to 7.0.

When the pH was adjusted to 7.0, the bacteria count increased to 10^4 bacteria/mL in two days, and gas could be seen evolving throughout the pond. Unlike beaker experiments performed in the laboratory, the bacteria in the pond were acclimated almost immediately, showing significant denitrification (rates of 500 mg of nitrate destroyed per liter per day) within days after neutralization. As can be seen from Figure 31, the denitrification rate proceeded rapidly. During this time, the bacteria reached a concentration of 10^7 bacteria/mL. After five days, the final acetic acid needed for complete denitrification were added. The pH dropped to less than 6.0 and denitrification essentially stopped. During the first week, after adding the final amount of acetic acid, the pond was allowed to sit unperturbed to see if bacterial action could adjust the pH to 7.0. This was slowly occurring to some extent--the pH increased to 6.4 before appearing to taper off. During this time, the dissolved oxygen level increased to about 2 mg/L and the bacteria could started to decrease. During the second week, sodium hydroxide solutions were added over a period of four to five days to increase the pH. In a few days the dissolved oxygen began to decrease and the temperature began to increase as denitrification restarted. The denitrification rate over the final 18 days was a nominal 300 mg of nitrate decomposed per liter per day until the nitrate level decreased to a value of 26 mg/L. At a nitrate level of 2500 mg/L, the pH increased from 7.2 to about 8.7 in one day. The dissolved oxygen

remained steady at 0.5 mg/L and the temperature, which had been on a gradual increase, appeared to peak and gradually began falling. During the pH rise, a layer of white foam (Figure 32 photograph 226063) appeared on the top of the pond. The foam, which was probably composed of heavy metal carbonates and hydroxides saturated with bacteria, lasted for six days--until the steady state pH of 9.3 was reached. At this point, the nitrates were less than 500 mg/L and a trace sulfide like odor could be detected. The solution color appeared to change from the usual green-brown to gray-brown. Within about two days, the nitrates decreased to 26 mg/L and 510 mg/L of total organic carbon remained in the water.

The southwest pond contained a nominal 167,000 pounds of nitrates before treatment. All of the nitrates were biologically destroyed in about 20 days when all biological parameters were met.

The circulation pumps continued to operate in an attempt to bio-oxidize the remaining organic carbon. The first 17 days showed little carbon decrease. The ponds did not obtain sufficient oxygen until an aerator was installed. The organic carbon was then consumed at a nominal rate of 37 mg/L of organic carbon per day for the next seven days until a final concentration of 250 mg/L total organic carbon was obtained. At this point, the acetate carbon portion of the 250 mg/L of organic carbon was less than 50 mg/L. During the aeration period, the dissolved oxygen remained less than 1 mg/L and only began to increase when the acetate ion concentration fell below 50 mg/L. During the final days of aeration, the adjacent northwest pond was neutralized. Within a week, the total organic carbon and nitrate concentrations in the treated southwest pond began to increase due to inleakage from the northwest pond. Shortly bacterial action began again in the southwest point and these nitrates were also decomposed.

Quality of Water After Denitrification - Samples were taken of the southwest pond and analyzed before and after the in-situ denitrification treatment; the results are shown in Figure 33. These data were taken before filtration and the 45 mg/L of aluminum and 3 mg/L of boron were subsequently removed by filtration of the water before discharge to the environment.

Northwest Pond - In September 1983, the treatment of the northwest pond was begun. The mixing system was moved to this point and the procedure for introducing chemicals was the same as that of the southwest pond. The pH was less than 2, and the nitrates were over 40,000 mg/L nitrates (Figure 34). The treatment was begun when seasonal temperatures were on the decrease. Denitrification decreased the nitrates from a nominal 40,000 mg/L to a nominal 28,000 mg/L in the first six weeks and then stopped. At this time, the ambient temperature had dropped to below 10°C. There was no denitrification during the winter months (November 1983 to February 1984) until ambient temperatures began to increase). In May, the pH of the pond decreased below 6.7 and the reaction stopped until the pH was increased. After the pH adjustment, all of the nitrates were destroyed at a rate of 300 mg NO₃ per day.

This pond originally contained a nominal 837,000 pounds of nitrates. When favorable biological conditions were maintained (about 215 days), all of the nitrates were destroyed.

Northeast and Southeast Ponds - In September 1983, piping and pumps were installed in these ponds so that the waters in these two ponds could be mixed to form a common water in the two ponds. Neutralization of the two ponds was completed using the same procedure as used for the northwest pond. The amount of chemicals added (calcium hydroxide and acetic acid) was similar to the northwest pond. As noted in Figure 35, the results are very similar to the northwest pond.

During denitrification, the color of the pond waters changed and Figures 36 (photo 217965) and 37 (photo 224500) show evidence of these color changes. The pond with the boat in the photo was a clear blue and had been completely denitrified with all solids settled to the bottom. The pond without a foam was a light brown color, indicating bioactivity was almost complete. The other two ponds with foam were also a brownish color indicating considerable biological activity was present. The brown ponds cleared in color when all nitrates were destroyed.

Solids Deposited in the Ponds - The neutralization step caused most of the metallic ions to form hydroxides or oxides that settled in the ponds. Excess calcium carbonate or hydroxide was used during neutralization and these also precipitated. The biodenitrification process produced carbon dioxide gas and some of it reacted with calcium ions in solution to form calcium carbonate, which also settled to the bottom of the ponds. After the pond treatment was completed, the pond bottoms were sampled. As noted in Figure 38, the collected solids contained about 80% liquid and 20% solids. The liquid phase contained nitrate ions which was expected since these could have been trapped during the neutralization step. The solids in the ponds were stirred and the liquid in the ponds dissolved most of the nitrate ions from the solids. The nitrate ions in the liquid phase were then redenitrified when an organic carbon (acetic acid) was added.

The solids contained 3 to 5% calcium, which is believed to be present as calcium carbonate and 1 to 2% aluminum which is expected to be present as aluminum hydroxide.

Removal of excess organic carbon - As previously stated, aeration of the pond waters was used to remove the excess organics (Figure 39). The excess organic carbon was added to the ponds to insure that the nitrates would be totally destroyed. Aeration was required to remove the carbon because an oxygen source is required to form carbon dioxide from the biological decomposition of organics.

As noted in Figure 40, the southwest pond had a typical 500 mg/L of total organic carbon (TOC). Aeration reduced the TOC to less than 200 mg/L in 34 days. Figures 41 (photo 224081) and 42 (photo 216750) are photographs of the ponds while aeration was in progress.

Washing of the solids in the ponds - The precipitated solids in the ponds contained high levels of nitrates that were trapped when the solids were precipitated. During aeration, the solids were stirred and nitrates were re-dissolved into the ponds. As this occurred, excess organic carbon was again added and the ponds were allowed to denitrify again. The solids were resuspended and rewashed until no additional nitrates were obtained from the solids.

Final Closure of the Ponds - The biologically treated water was then passed through a waste water treatment facility which consisted of a reactor clarifier, filters, and carbon absorption. Figure 43 lists the operational steps used to treat the water after the biological steps were complete. The primary purpose of this treatment was to remove most of the suspended solids which may occur while pumping the water from the pond. Absorption of residual amounts of organics was also accomplished by the addition of activated carbon. The water was discharged via a National Pollution Discharge Elimination System (NPDES) permit. It met all chemical parameters required by the permit plus meeting two (2) different biological tests. The biological tests were growth and survival of fathead minnows (Pimephales promelas) and survival and reproduction of a seven day testing using Ceriodaphnia dubia. The biological quality showed no problems in the 3 to 5 wt. % range; therefore, the water was discharged at a controlled rate and the receiving stream passed both tests at 100 wt. % which means no degradation of the receiving water occurred at this discharge rate using these two species as the reference species.

Figures 44 (photo 231946) and 45 (photo 231490) are photographs taken of the ponds and chemical treatment process. Typical chemical analysis of the treated water is shown in Figure 46. The solids removed during chemical treatment were returned to one of the ponds.

Solids Deposited in the Ponds - Typical chemical analysis of the dried solids in the bottom of the empty ponds are shown in Figures 47 and 48. Photographs of the pond during different stages of dryness are shown in Figures 49 (photo 236987), 50 (photo 236563), 51 (photo 241762), and 52 (photo 242130). As noted the air dried pond bottoms were nearly free of all liquids.

Filling Ponds - The next steps in closing of the ponds are noted in Figure 53. Six inch diameter or larger limestone rocks were added to stabilize the bottom of the ponds (Figures 54 and 55 photographs 258129 and 258133). A 24-inch clay cap (Figure 56 photo 258539) was packed to have a permeability of less than 1×10^{-7} cm/sec. A flexible plastic liner and a geotextile fabric was placed on top of the compacted clay. Eighteen inches of compacted clay (Figure 57 photo 258720) was placed on top of the fabric followed by three inches of crushed stone and three inches of asphaltic concrete. Several monitoring wells are located adjacent to the cap and if data from these wells show degradation of the groundwater quality, the water will be removed and subjected to the appropriate treatment. The last layer was an asphalt cap and the ponds are now used as a parking lot (Figure 58 photo 272658).

Present Waste Treatment - The Y-12 Plant continues to generate nitrate wastes. Several large tanks (500,000 gallon) have been installed and serve as biological reactors using the same process as used on the ponds. When biologically treated, the water is passed through a settling tank to remove most of the solids and then through a waste water treatment process to remove the rest of the solids.

These solids are currently being stored in large steel tanks until sufficient data is collected to delist them from the hazardous wastes category.

Summary - The ponds have now been closed (Figure 59) and a new permitted treatment facility for new wastes consisting of large tanks have been operational since 1986.

Closure of Other Ponds - Other ponds (Figure 60) have been in-situ closed by the Y-12 Plant. These did not contain nitrate wastes and were not bio-denitrified. The closure details for New Hope Pond consisted of draining the water from the pond and installing a multi-layer cap similar to the one used at the S-3 Ponds. The final layer of asphalt was not installed on New Hope Pond. Closure of the 2 small waste oil ponds consisted of draining the water to a permitted treatment process. The water was filtered to remove solids, aerated to remove volatile organics, filtered through carbon to remove other organics and re-filtered to remove any residual solids. The water quality had to meet discharge parameters which included chemical analysis as well as the fathead minnow and ceriodaphnia tests.

The bottom solids from the 2 oil ponds were removed and stored above ground in a building since the solids contained low levels of polychlorinated biphenyls and uranium. The final disposal method for these solids have not yet been established.

The empty oil ponds were filled with a multi-layer cap similar to the S-3 ponds but no asphalt final layer was installed. A drainage system was installed along 1 side of the closed oil ponds to intercept any incoming contaminated liquids which may seep out of closed trenches filled with wastes generated in the late 1950's through the 1970's. All liquid seepage will be collected and treated in a new permitted treatment facility.

FIGURE 1

RECYCLE OF NITRIC ACID AND ALUMINUM NITRATE

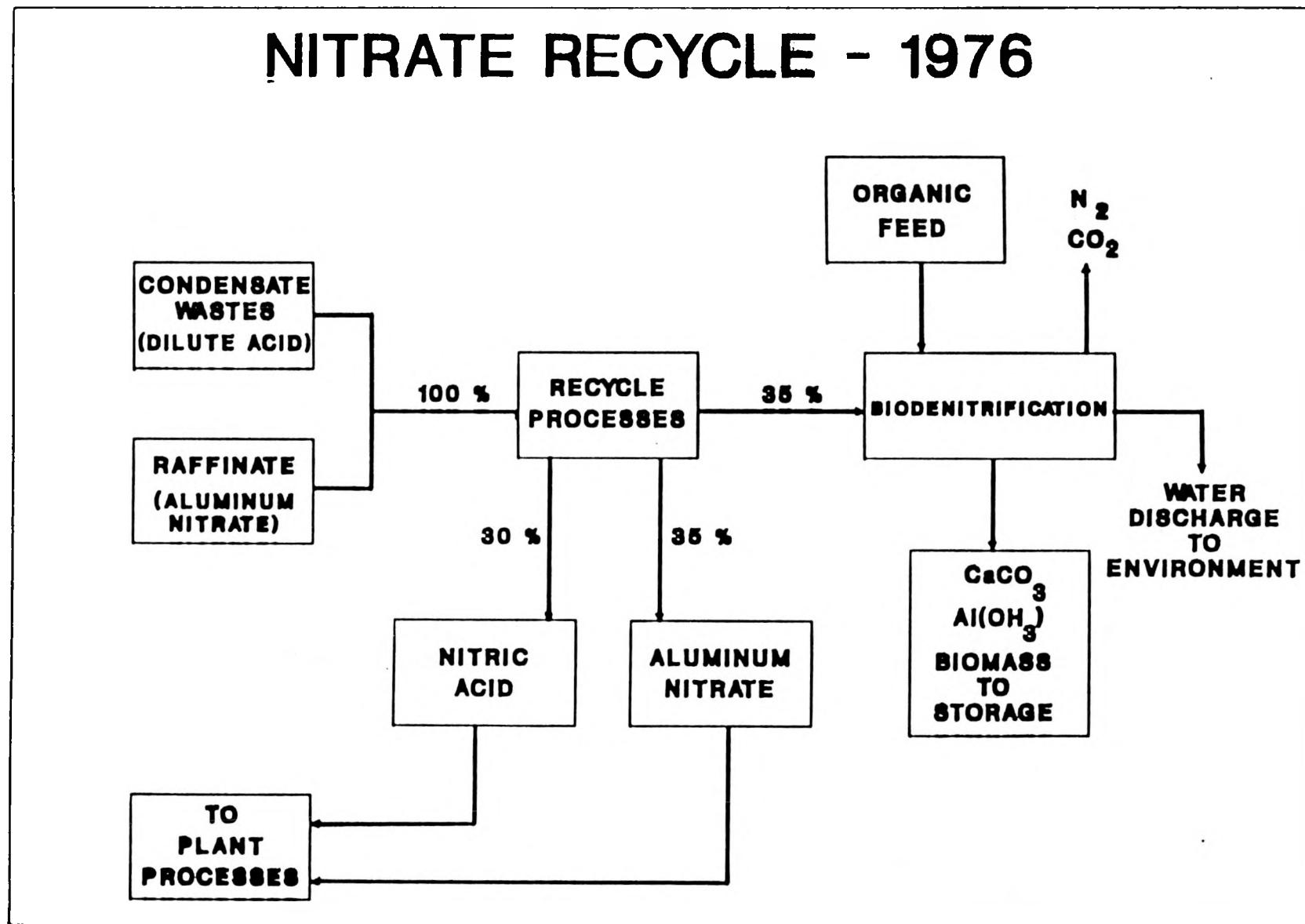
JOHN NAPIER

WASTE MINIMIZATION MEETING
Y-12 PLANT, OAK RIDGE, TENNESSEE
JULY 8, 1991

MARTIN MARIETTA

Oak Ridge Y-12 Plant
Managed by Martin Marietta Energy Systems, Inc.
for the U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400

• FIGURE 2



REC-9 MAR 1991

TYPICAL CONDENSATE ANALYSIS

NITRIC ACID	0.5 TO 9 WT.%
WATER	99.5 TO 91 WT.%
CHLORIDE	14 MG/L
FLUORIDE	9 MG/L
URANIUM	0.8 MG/L

FIGURE 4

NITRIC ACID RECYCLE.

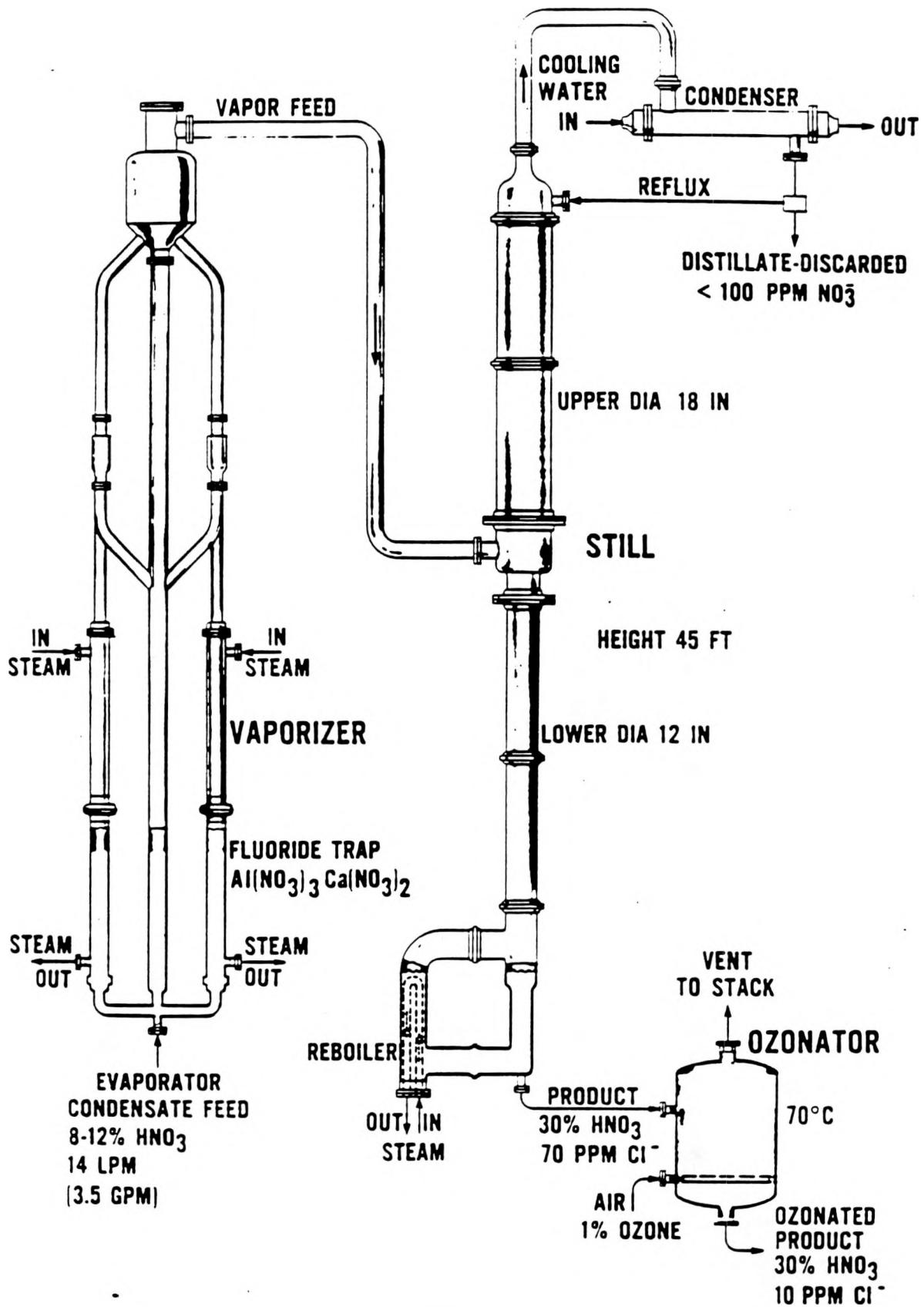


FIGURE 5

FLUORIDE AND URANIUM TRAP

23.8 WT% ALUMINUM NITRATE MONOHYDRATE

52.9 WT% CALCIUM NITRATE TETRAHYDRATE

23.3 WT% WATER

26

NORMAL OPERATING PARAMETERS

TRAP SOLUTION REPLACED WHEN A 5 : 1

ALUMINUM-TO-FLUORIDE WEIGHT RATIO IS

OBTAINED

REC-2 MAR 1991

FIGURE 6

NITRIC ACID RECYCLE

FEED

NITRIC ACID	9 WT.%
CHLORIDE	14 MG/L
FLUORIDE	9.5 MG/L
URANIUM	0.7 MG/L

PRODUCT

NITRIC ACID	35 WT.%
CHLORIDE	< 10 MG/L
FLUORIDE	< 1 MG/L
URANIUM	< 0.01 MG/L

% RECOVERED EQUALS > 97.4%

TYPICAL RAFFINATE ANALYSIS

NITRIC ACID	6.0 WT.%
TOTAL NITRATES	31.1 WT.%
ALUMINUM	3.0 WT.%
CHLORIDE	67 MG/L
FLUORIDE	3000 MG/L
URANIUM	1 MG/L
WATER	60 WT.%

FIGURE 8

ALUMINUM NITRATE RECYCLE.

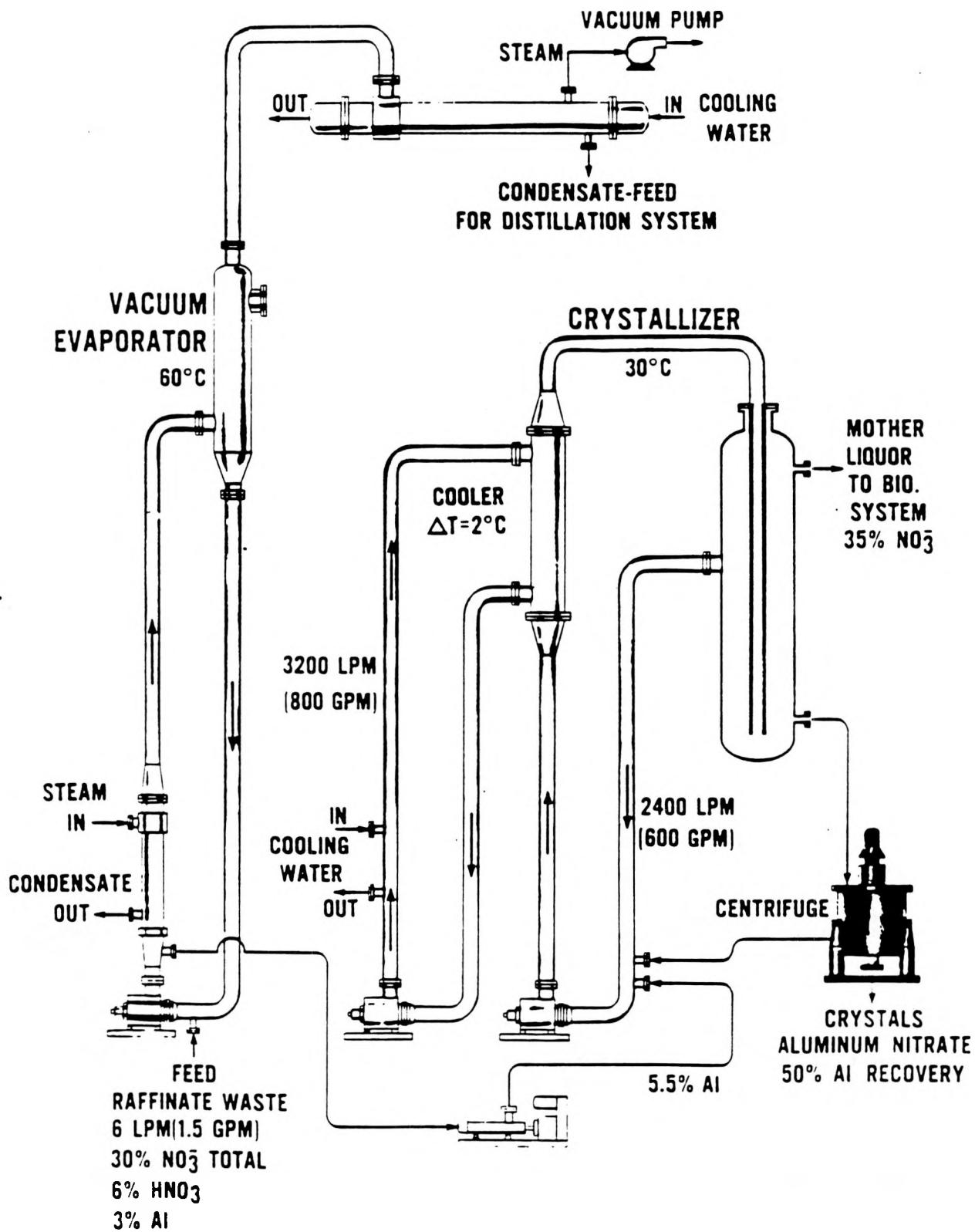
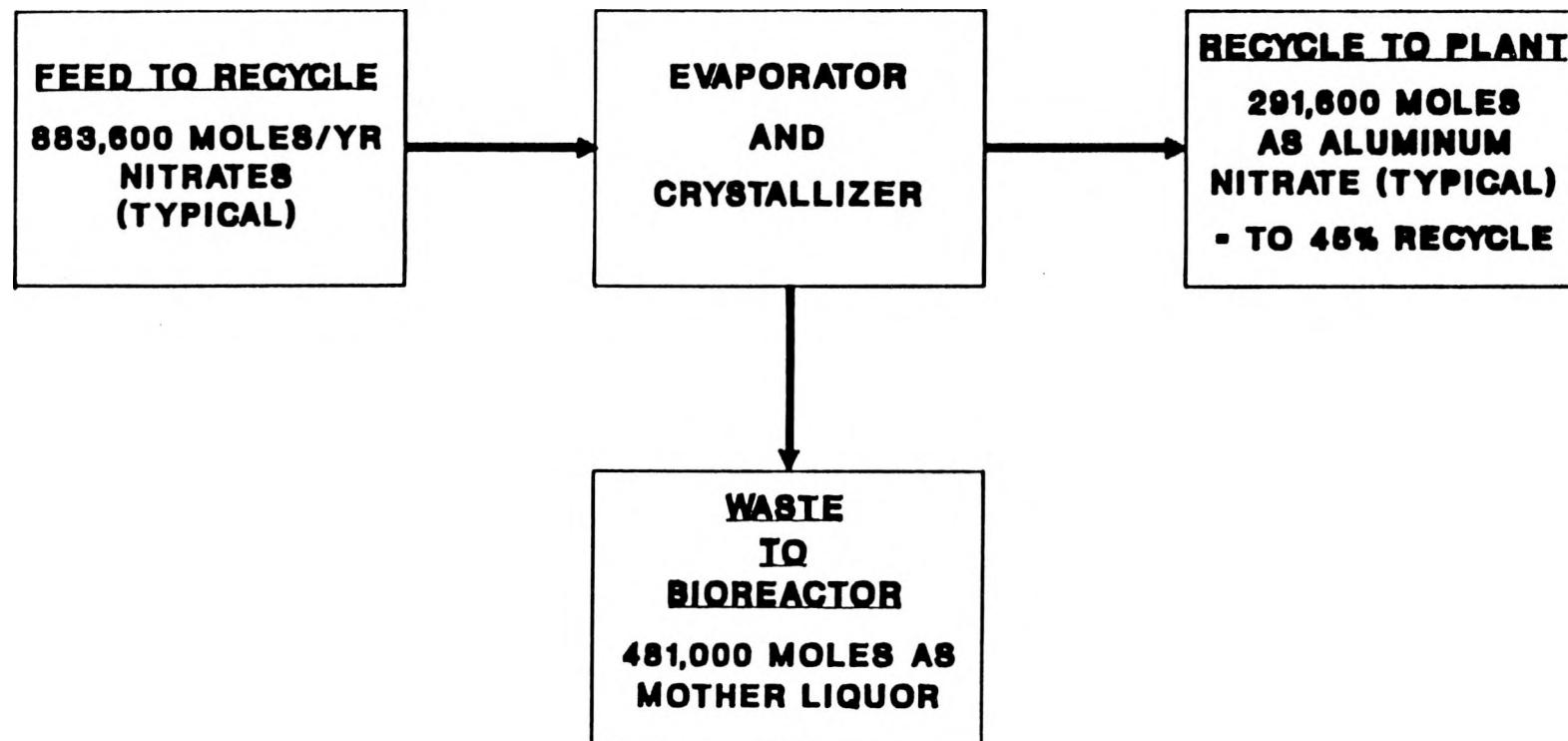


FIGURE 9

ALUMINUM NITRATE RECYCLE



STIRRED TANK BIODENITRIFICATION

- * INSTALLED IN 1976 FOR SOME WASTES
- * REACTOR VOLUME = 25,000 GALLONS
- * NITRATE DECOMPOSED PER DAY:
MAXIMUM = 2800 LBS
DESIGN RATE = 1400 LBS/DAY OF NO₃

FIGURE 11

TYPICAL MOTHER LIQUOR ANALYSIS

NITRIC ACID	6.0 WT.%
TOTAL NITRATES	50.0 WT.%
ALUMINUM	4.4 WT.%
CHLORIDE	81 MG/L
FLUORIDE	6500 MG/L
URANIUM	2 MG/L
WATER	39.6 WT.%

FIGURE 12

BIODENITRIFICATION

START-UP PHASE (estimated):

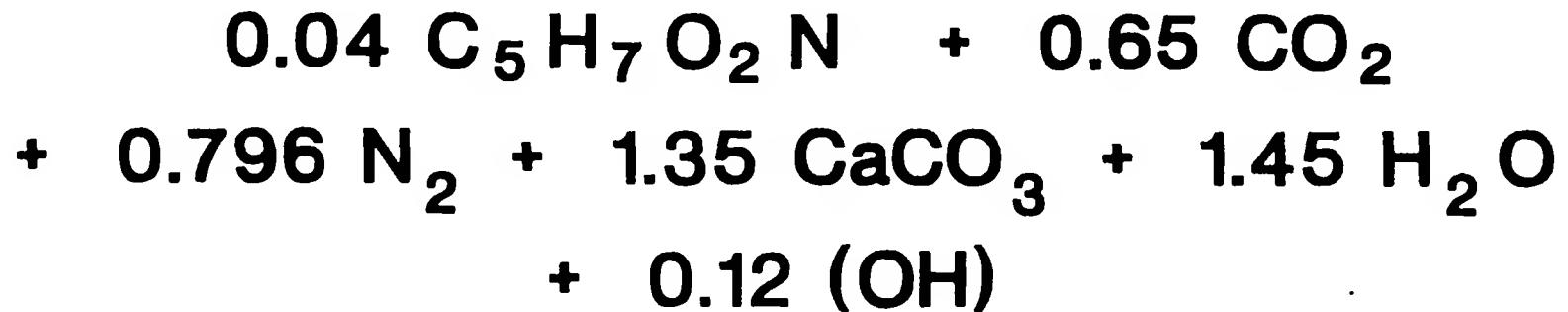
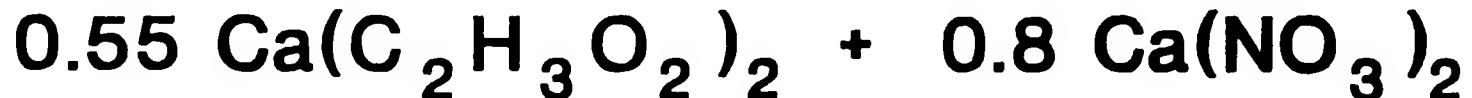
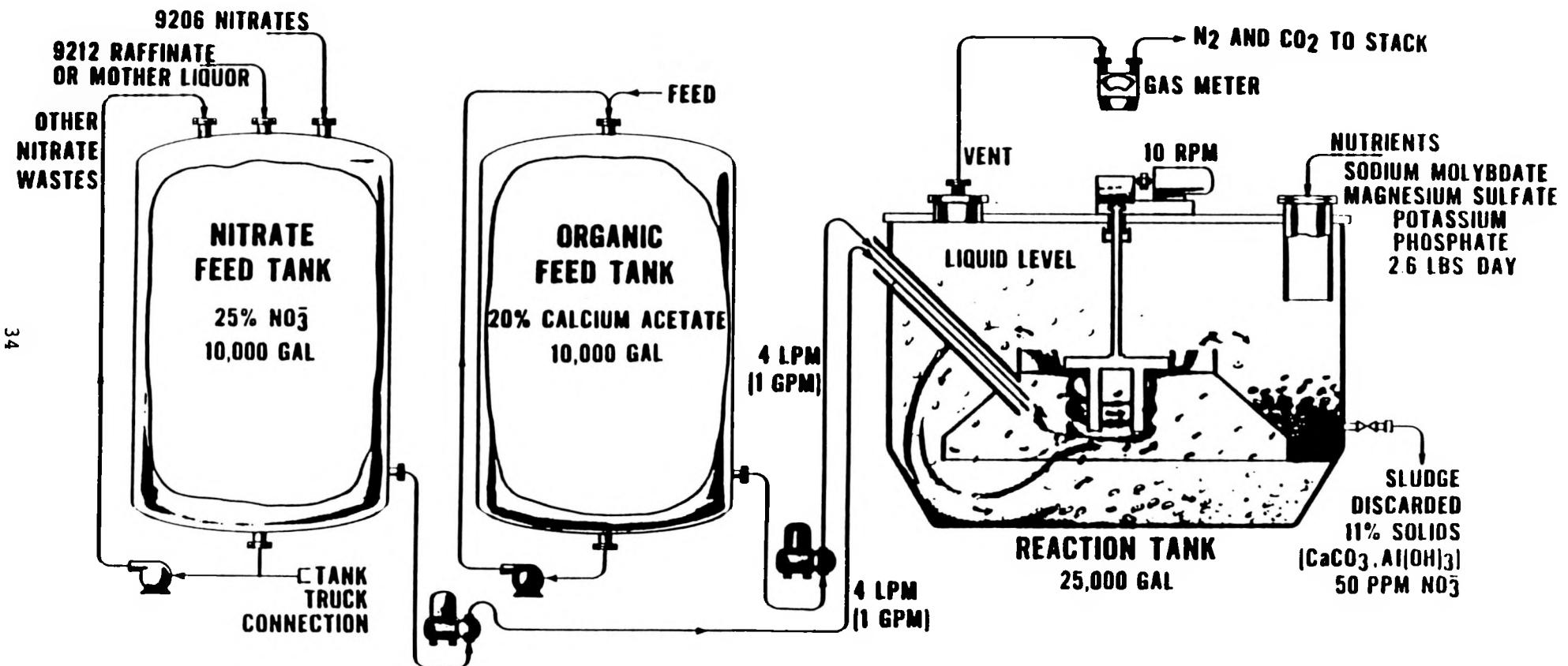


FIGURE 13

STIRRED TANK BIODENTRIFICATION.



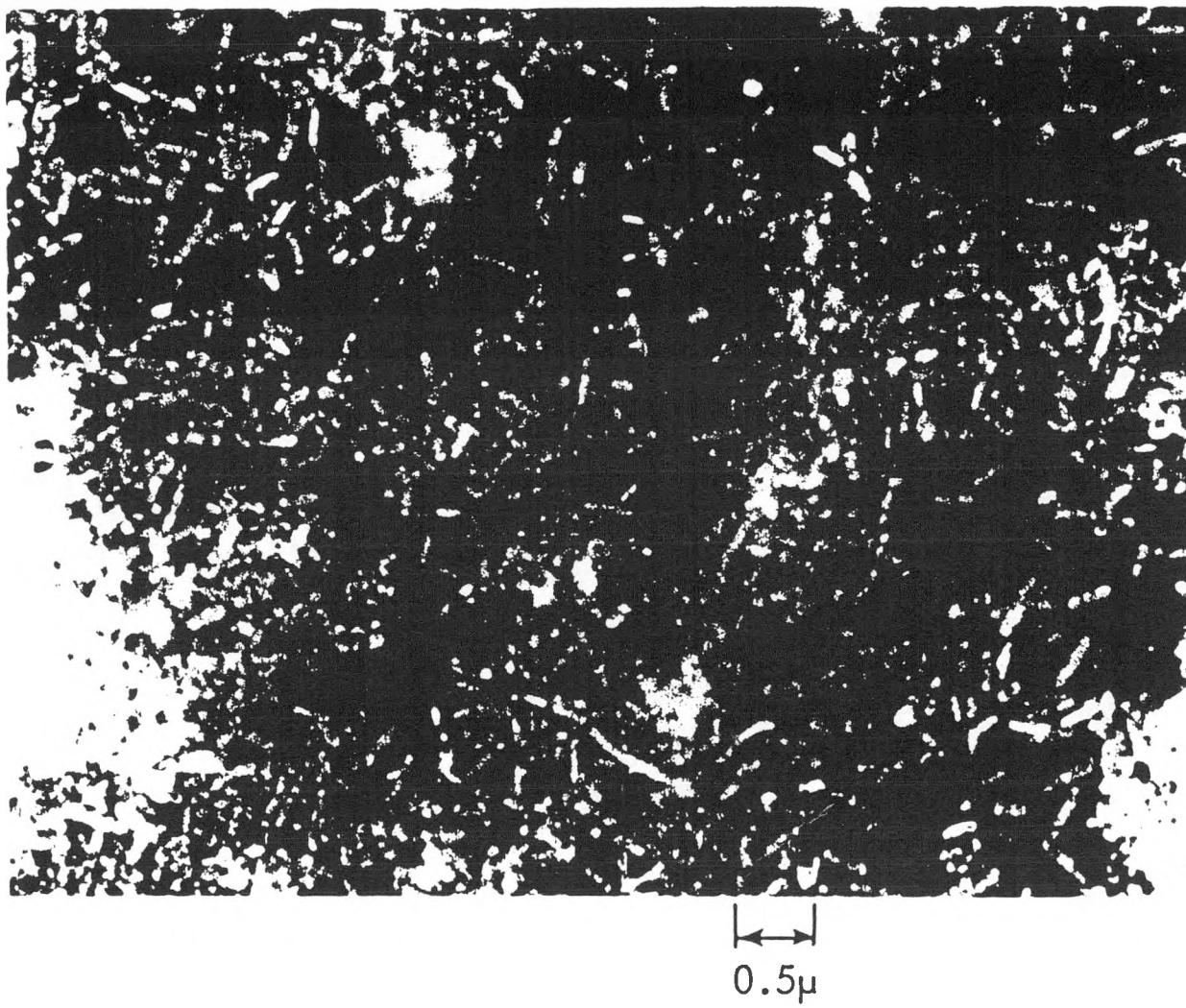
BACTERIA

- * **PSEUDONOMAS STUTZERI WAS THE ORIGINAL BACTERIA**
- * **A CONTINUOUS FLOW REACTOR CONTAINED 16 DIFFERENT ISOLATES:**
 - 14 REDUCED NO₃ TO N₂**
 - 15 WERE GRAM-NEGATIVE RODS**
- * **MORE THAN 100 SPECIES IDENTIFIED IN CONTINUOUS FLOW REACTORS**

FIGURE 15

DENITRIFICATION BACTERIA

DENITRIFYING BACTERIA ARE A MIXED CULTURE WITH A LARGE NUMBER OF ROD-SHAPED CELLS (*PSEUDOMONAS STUTZERI*).



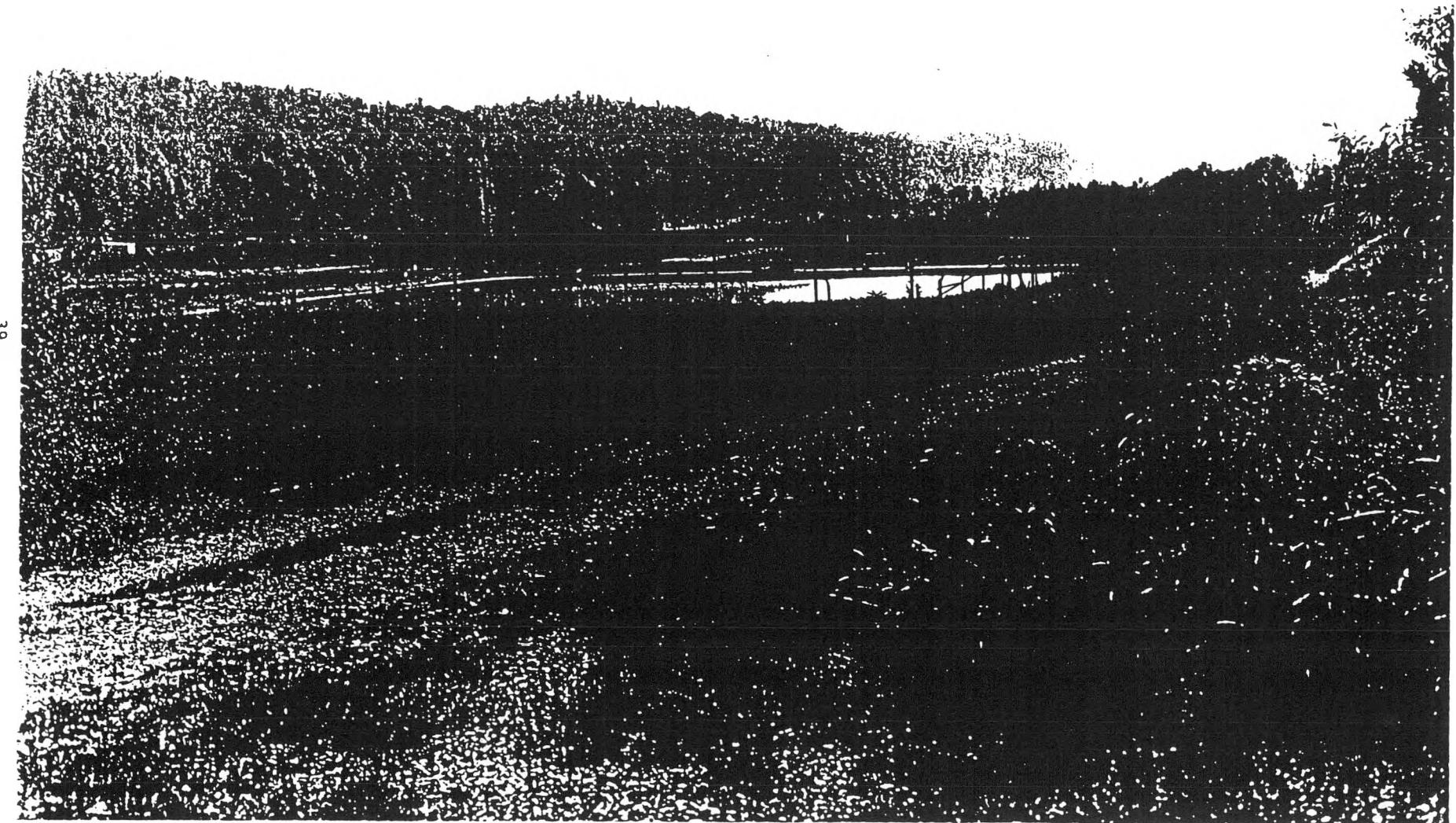
BACKGROUND INFORMATION

- Y-12 PLANT LOCATED IN OAK RIDGE, TENNESSEE
- OWNED BY DOE AND MANAGED BY MARTIN MARIETTA ENERGY SYSTEMS
- SPECIAL MATERIALS ARE MADE TO SUPPORT DOE'S PROGRAM AND OTHER GOVERNMENT AGENCIES
- NITRATE WASTES EMITTED FROM URANIUM RECYCLE OPERATIONS AND OTHER PLANT WASTE STREAMS

S-3 POND INFORMATION

- 4 PONDS INSTALLED IN EARLY 1950'S
- ACIDIC WASTES PUT INTO PONDS
- PONDS WERE SEEPAGE BASINS
- WASTES ENTERING PONDS:
 - ACIDIC NITRATES FROM URANIUM RECYCLE
 - PLATING AND PICKLE BATH WASTES
 - MOP WATERS FROM OPERATING AREAS
 - AQUEOUS MACHINE COOLANTS
- EACH POND WAS 200 FT WIDE, 200 FT LONG,
15 TO 20 FT DEEP
- IN 1976, A PROCESS WAS INSTALLED TO
RECYCLE SOME OF THE NITRATE WASTES

FIGURE 18
S-3 PONDS



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Photo #142559

FIGURE 19
PHOTOGRAPH OF S-3 PONDS

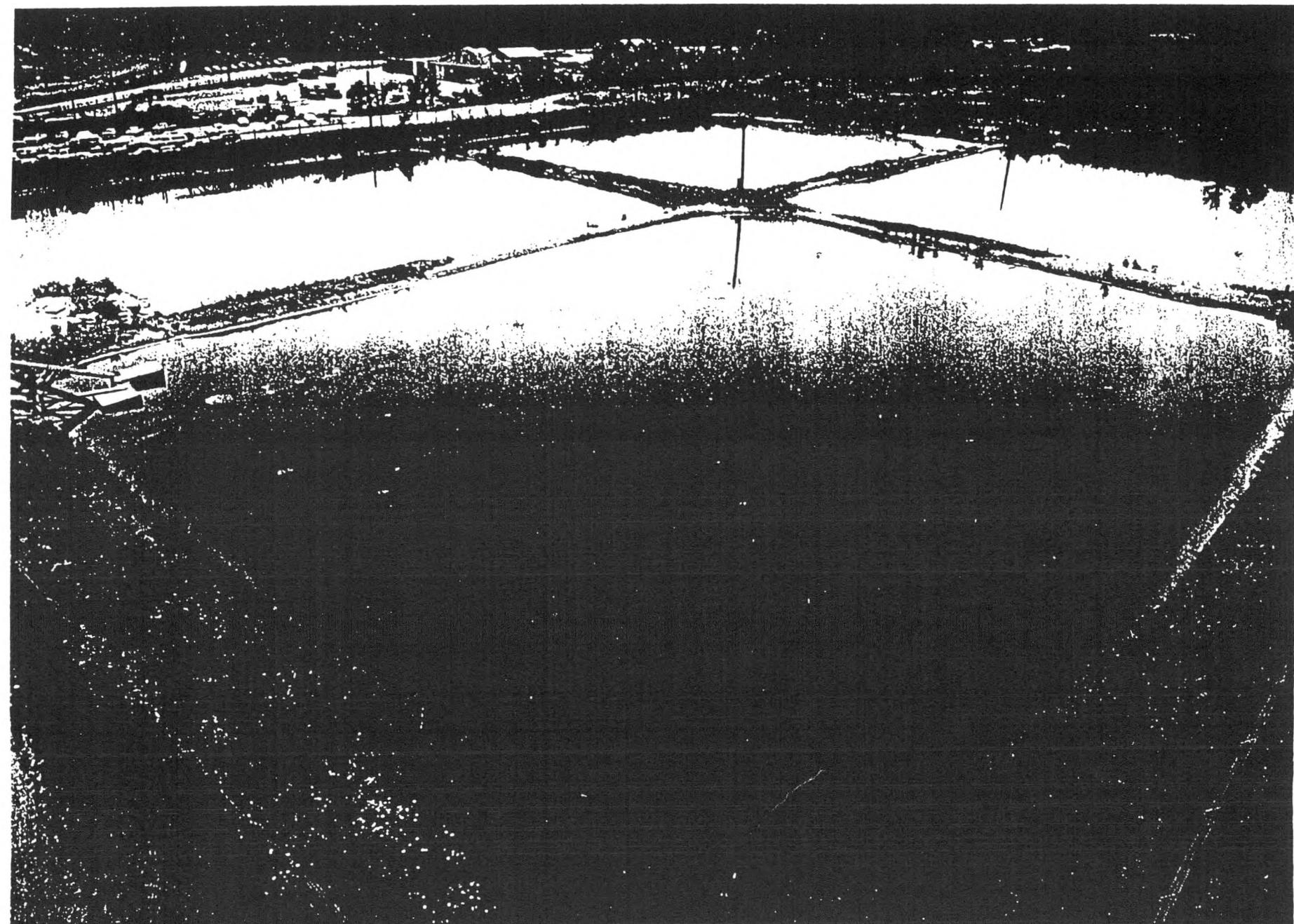


Photo #215000

FIGURE 20

S-3 PONDS

TYPICAL CHEMICAL ANALYSIS IN 1978

(NOTE: CHEMICALS VARIED WITH TIME)

<u>ION</u>	<u>MG/L</u>	*
NO ₃	45,000	
Al	2300	Ponds varied from 9,000
B	25	to 45,000 mg/l of NO ₃ ,
Ca	450	dependent on pond. The
Cd	80	listed impurities are
Cr	50	maximum amounts found
Cu	28	in several samples.
Fe	470	
Mg	410	
Na	3000	
Ni	90	
Th	65	
U	230	
Ph	1.0	

POND TREATMENT PLANS

- NEUTRALIZATION WITH CaCO_3 OR Ca(OH)_2
- ADDITION OF ACETIC ACID (ORGANIC CARBON SOURCE)
- BACTERIA FOR DENITRIFICATION
- AERATION TO DECOMPOSE EXCESS ORGANICS
- TREATMENT OF WATER:
 - SETTLE SOLIDS IN PONDS
 - FLOCCULATE WATER WITH FERRIC SULFATE
 - FILTER SOLIDS
 - STORAGE OF SOLIDS
- RELEASE OF WATER
- CAP PONDS
- INSTALL PARKING LOT

FIGURE 22

TYPICAL ANALYSIS OF THE SOUTHWEST POND
(1983 DATA)

	MG/L
NO_3	7900 to 8300
Al	583
Be	0.06
Cd	0.23
Cn	4.8
Fe	26
Ni	31
Pb	2
Zn	4
U	35
TOTAL ORGANIC CARBON	260
pH	1.0 to 2.7

FIGURE 23

LABORATORY TESTS USING S-3 POND WATERS

ION	MG/L		
	BEFORE TREATMENT	AS-NEUTRALIZED AND FILTERED	AFTER DENITRIFICATION
NO ₃	17,000	17,000	< 50
U	-	5.0	2.3
TOC	-	-	140
Al	740	0.3	0.2
B	7.7	2.9	3.8
Be	0.25	0.3	0.005
Ca	930	3166	-
Cd	0.27	0.02	-
Cr	4.3	0.005	0.002
Cu	6.2	0.019	< 0.03
Fe	19.4	< 0.007	0.18
Mn	4.8	1.0	< 0.001
NI	41.6	0.15	0.10
Pb	1.1	< 0.1	< 0.015
Th	0.39	< 0.04	< 0.04
Zn	4.3	< 0.002	< 0.011
Ph	2.7	7.5	7.5

S3P-18 MAR 1991

FIGURE 24
UNLOADING CALCIUM CARBONATE

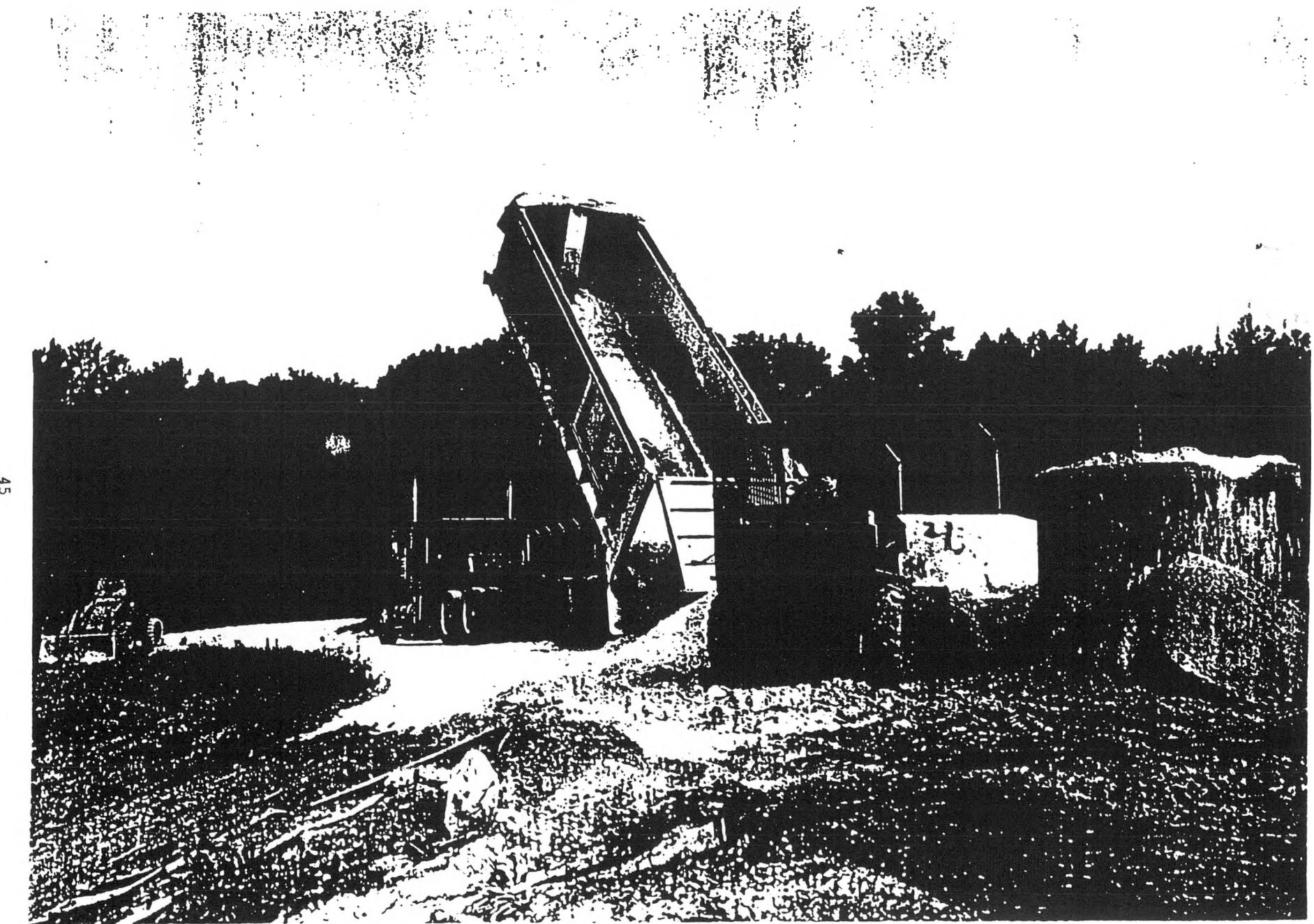


Photo #216742

FIGURE 25
ADDING CALCIUM CARBONATE TO PONDS

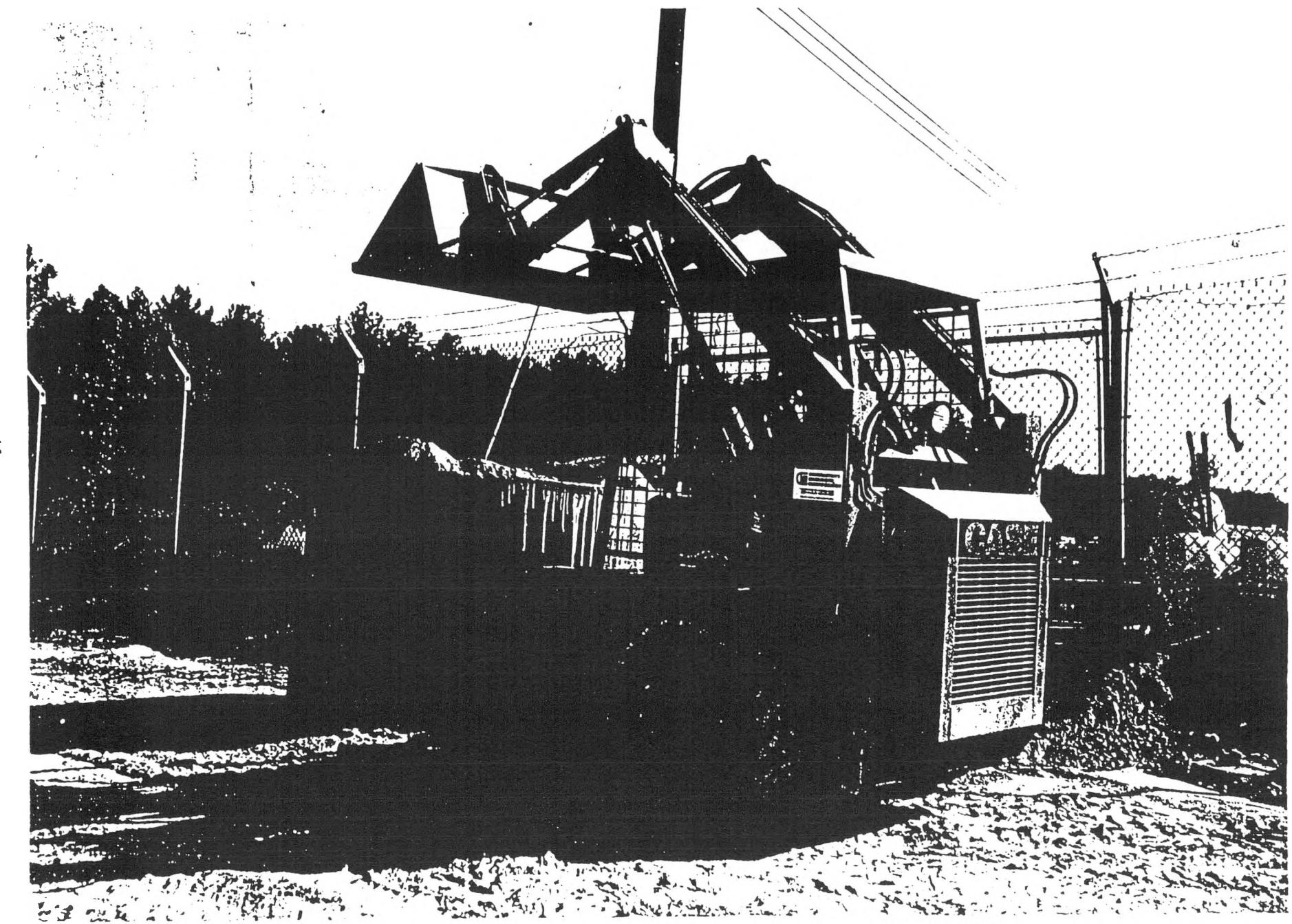


Photo #214445

FIGURE 26
NEUTRALIZATION OF PONDS



Photo #214450

FIGURE 27

S-3 NEUTRALIZATION

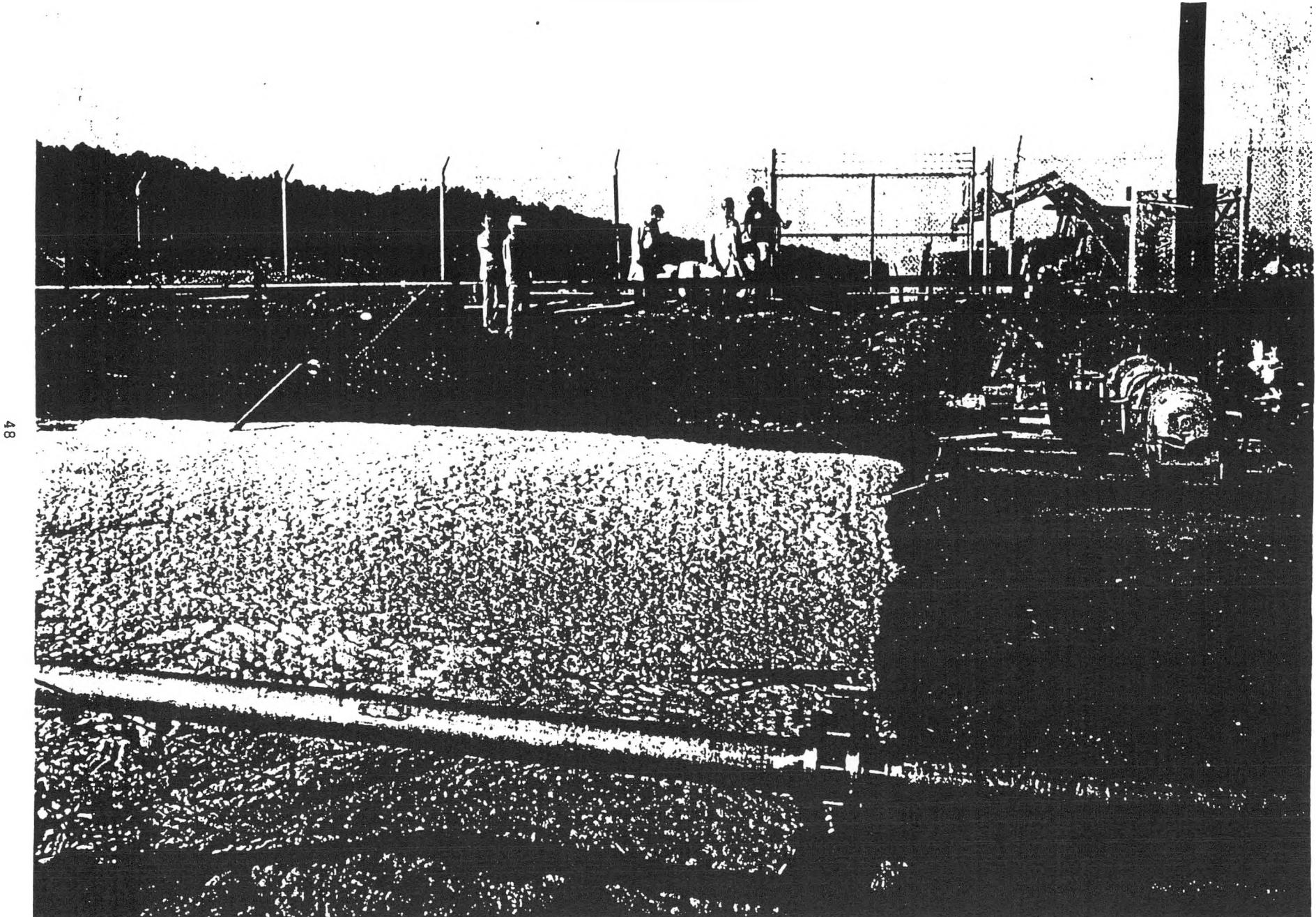


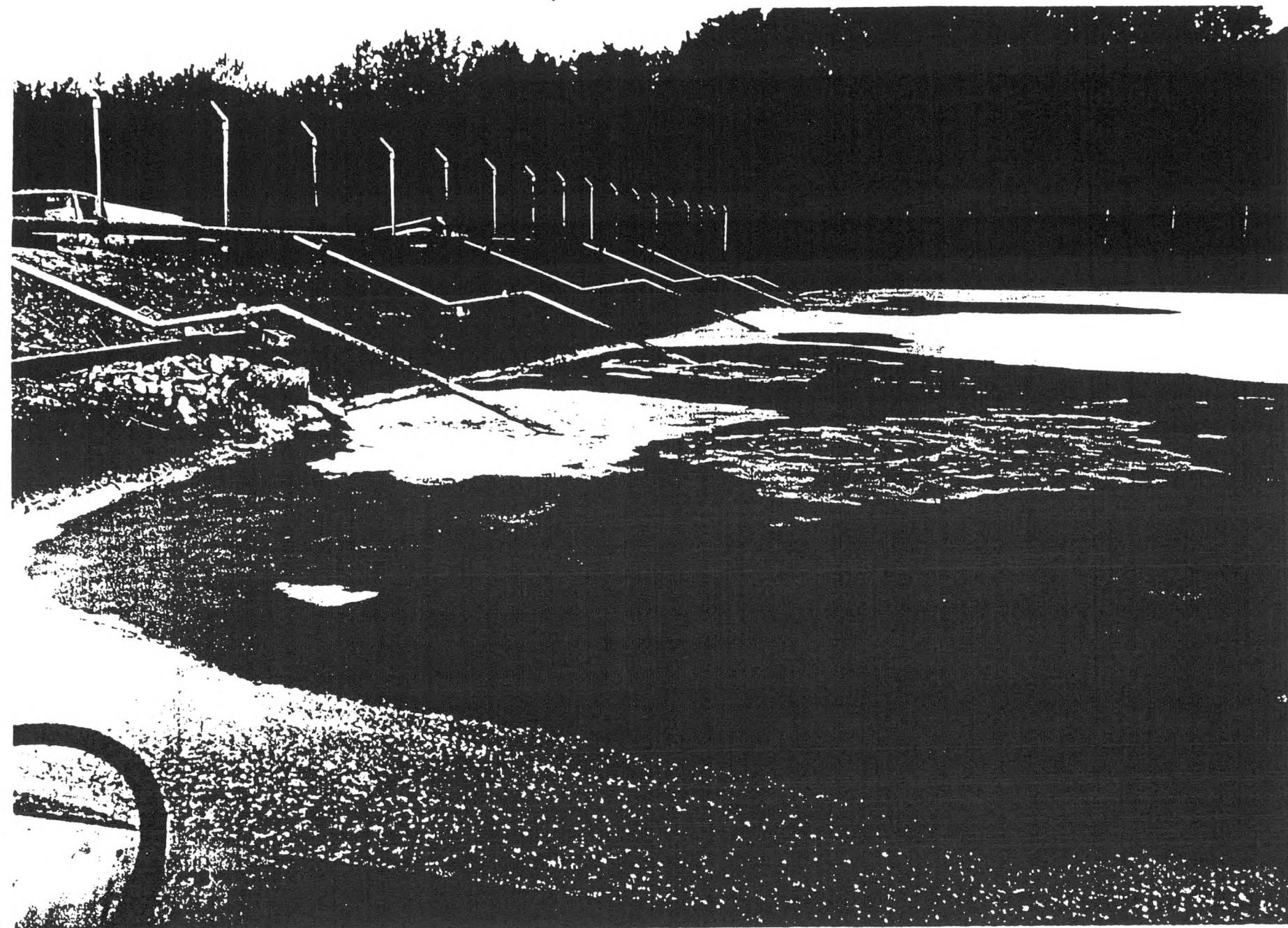
Photo #214447

FIGURE 28
NEUTRALIZATION



Photo #214444

FIGURE 29
MIXING OF PONDS



50

Photo #216749

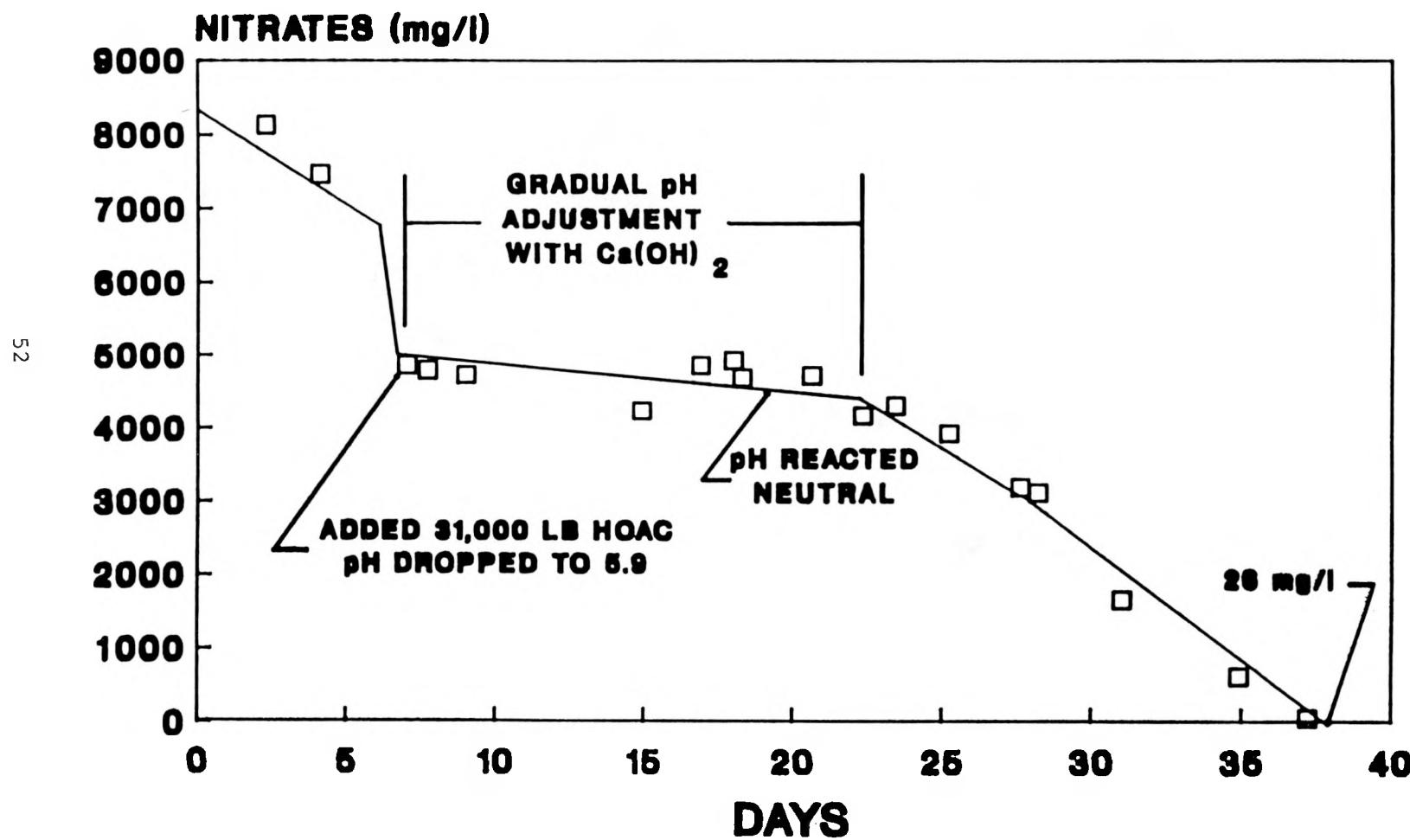
DENITRIFICATION

OPERATING PARAMETERS

- CARBON-TO-NITROGEN WT. RATIO: 1.1 TO 1.2
- AGITATION
- pH: BELOW 6.8 - NO BIOLOGICAL ACTIVITY
- TEMPERATURE: BELOW 10 DEGREES C - NO BIOLOGICAL ACTIVITY
- PHOSPHATE: 5 MG/L OR MORE
- BACTERIA: 10^7 BACTERIA/ML OR MORE
- ORGANIC CARBON: ACETATE CARBON
- DISSOLVED OXYGEN

FIGURE 31

NITRATE CONCENTRATION SOUTHWEST POND



89P-24 MAR 1991

FIGURE 32
FOAM DURING DENITRIFICATION



53

Photo #226063

FIGURE 33

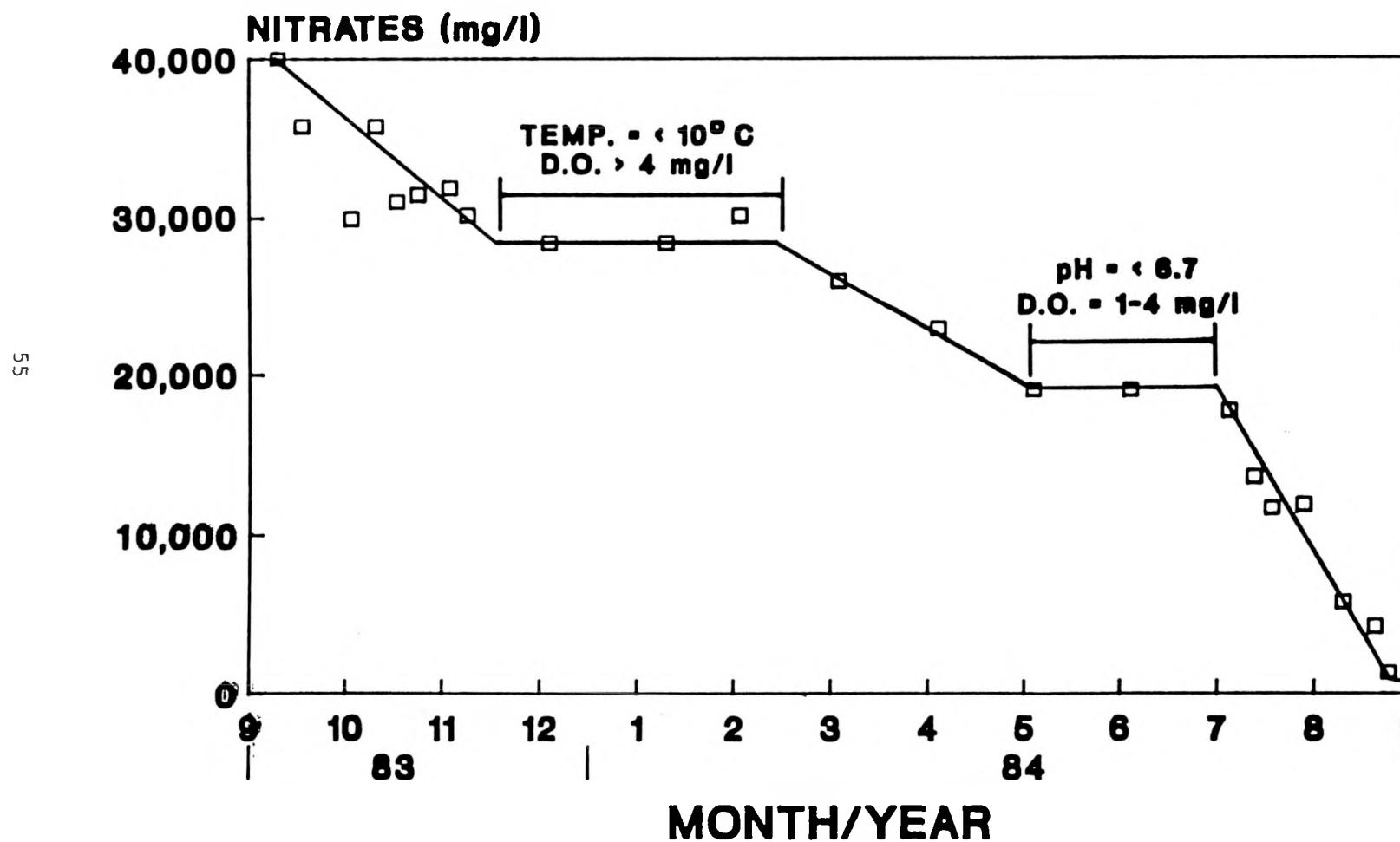
SOUTHWEST POND (1983)

BEFORE AND AFTER TREATMENT DATA

<u>ION</u>	<u>MG/L</u>	
<u>ION</u>	<u>BEFORE</u>	<u>AFTER</u>
NO ₃	7900	< 50
Al	583	45
B	8	3
Ca	845	55
Cd	0.23	< 0.007
Cr	4.8	< 0.003
Cu	5.6	< 0.08
Fe	26	0.1
Mg	119	47
Ni	31.3	0.32
Pb	2	< 0.02
Th	0.7	< 0.04
Zn	4.25	< 0.009
U	35	2.6

FIGURE 34

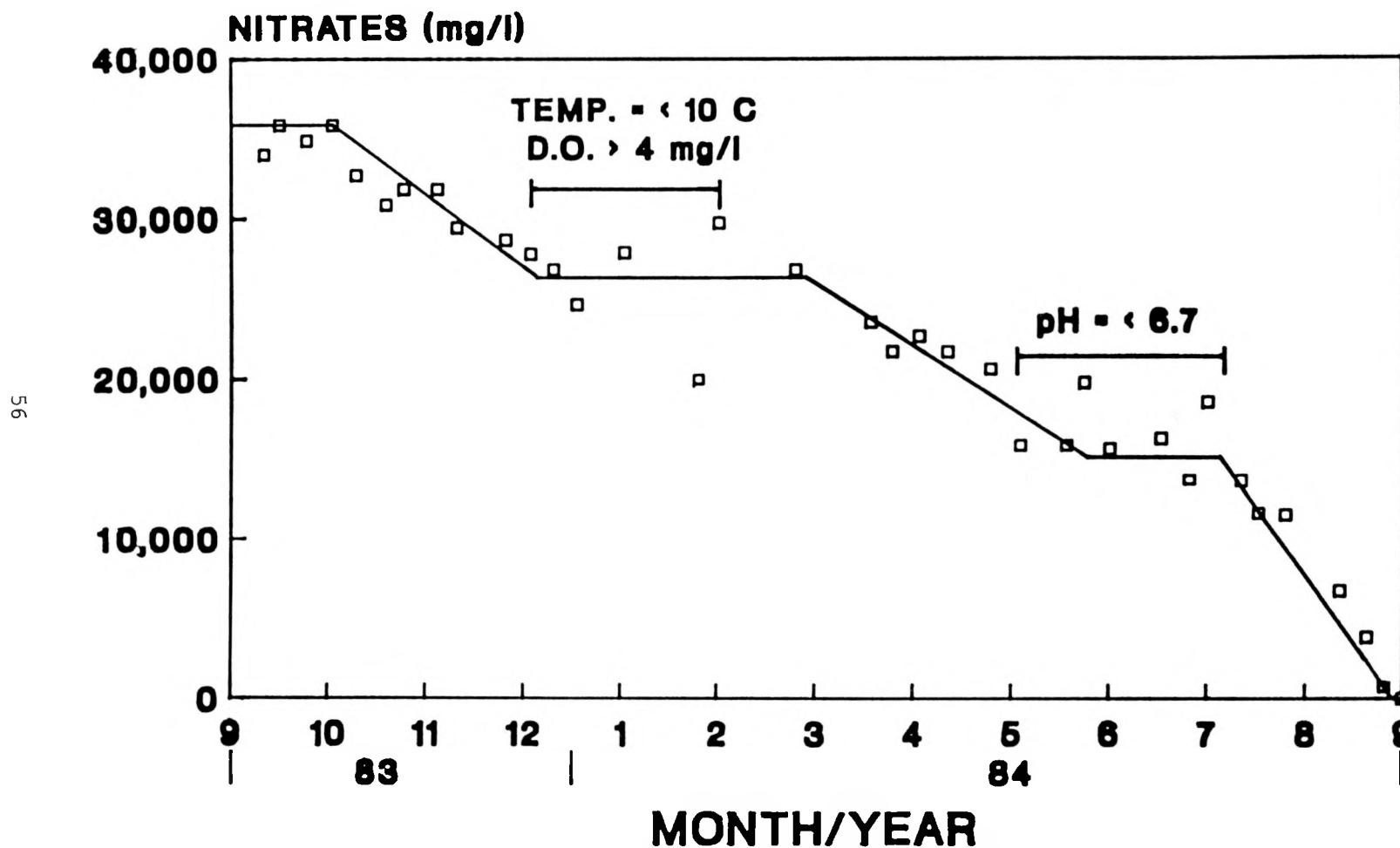
NITRATE CONCENTRATION NORTHWEST POND



83P-22 MAR 1991

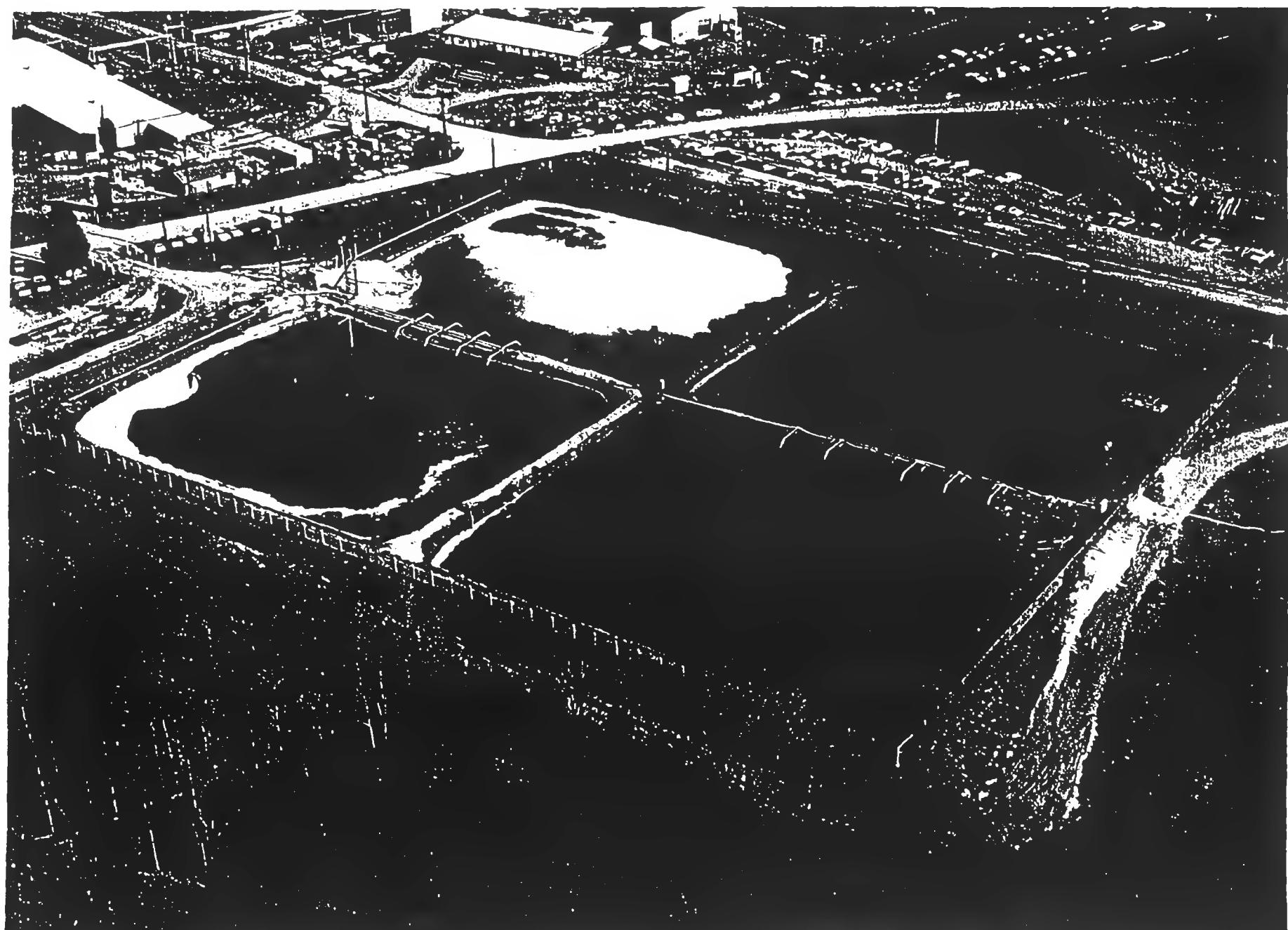
FIGURE 35

NITRATE CONCENTRATION NORTHEAST AND SOUTHEAST PONDS



83P-23 MAR 1991

FIGURE 36
PONDS DURING DENITRIFICATION



57

Photo #217965

FIGURE 37

S-3 PONDS DURING TREATMENT

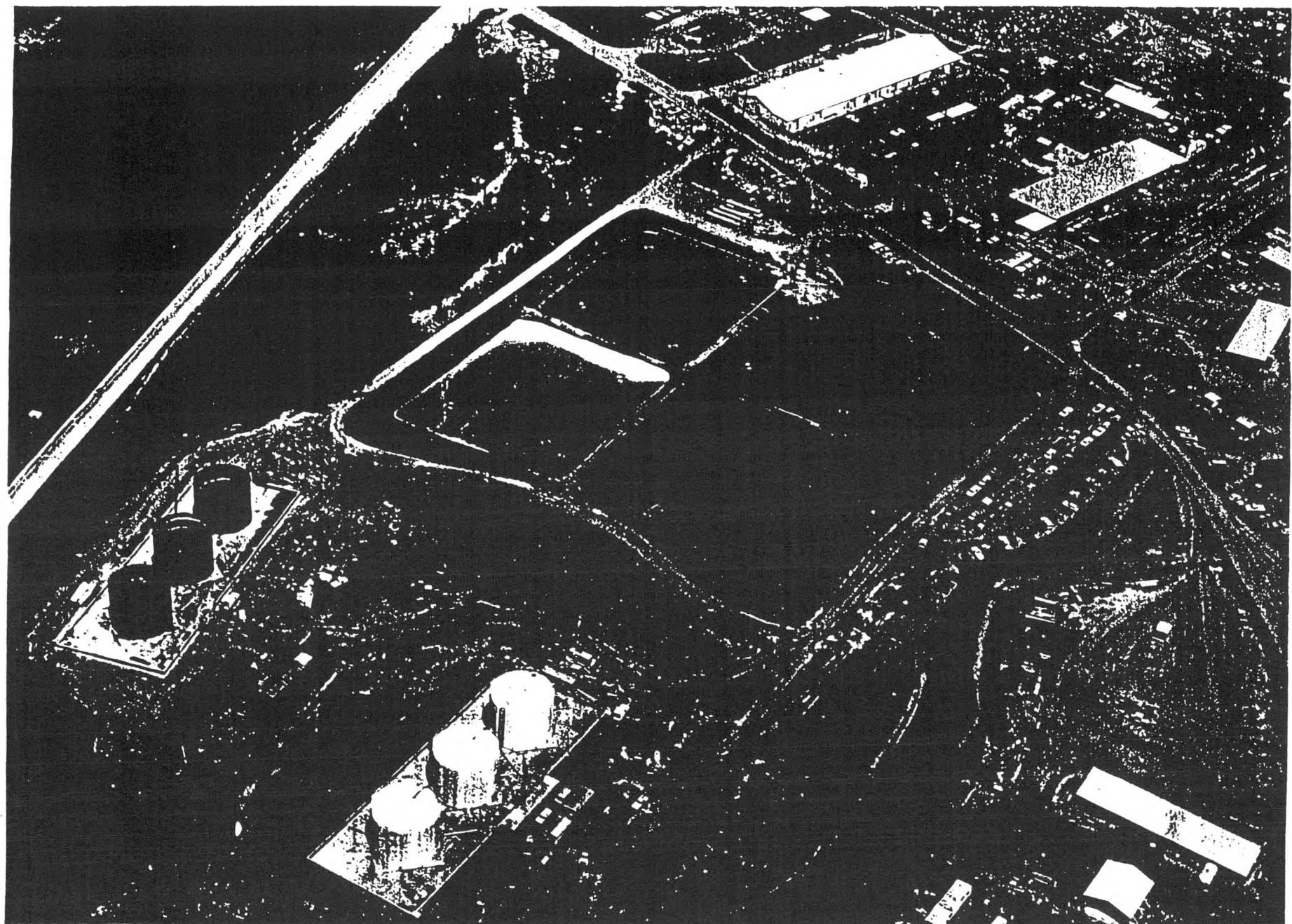


Photo #224500

FIGURE 38

Solids Deposited in The Ponds (Dried Basis)

Chemical	Range (mg/L)
Ag	<.7 - 3
Al	11,400 - 21,100
B	13 - 43
Ba	39 - 130
Be	<1 - 2.9
Ca	29,200 - 47,000
Cd	2 - 7
Cr	95 - 140
Cu	78 - 100
Fe	1300 - 3000
K	100 - 1300
Li	36 - 77
Mg	1500 - 1700
Mn	62 - 95
Mo	9 - 19
Na	990 - 2900
Ni	230 - 570
P	470 - 990
Pb	16 - 36
Sn	18 - 35
Th	61 - 150
Ti	64 - 350
Zn	32 - 58
U	769 - 1040
U ²³⁵ (%)	0.29 - 0.39

Organics

Volatile (mg/L)	
Tetrachloroethene	.0016 - .0036
Freon 113	.0036
All others total	<0.1

Acid base/neutral fraction

Tetrachloroethene	1.2 - 6.6
Phenol	4.8 - 76
1, 1, 2, 2 - Tetrachloroethane	1.2 - 5.5

% moisture

Sludge layer (ft)

78 - 82

2 - 4.6

FIGURE 39

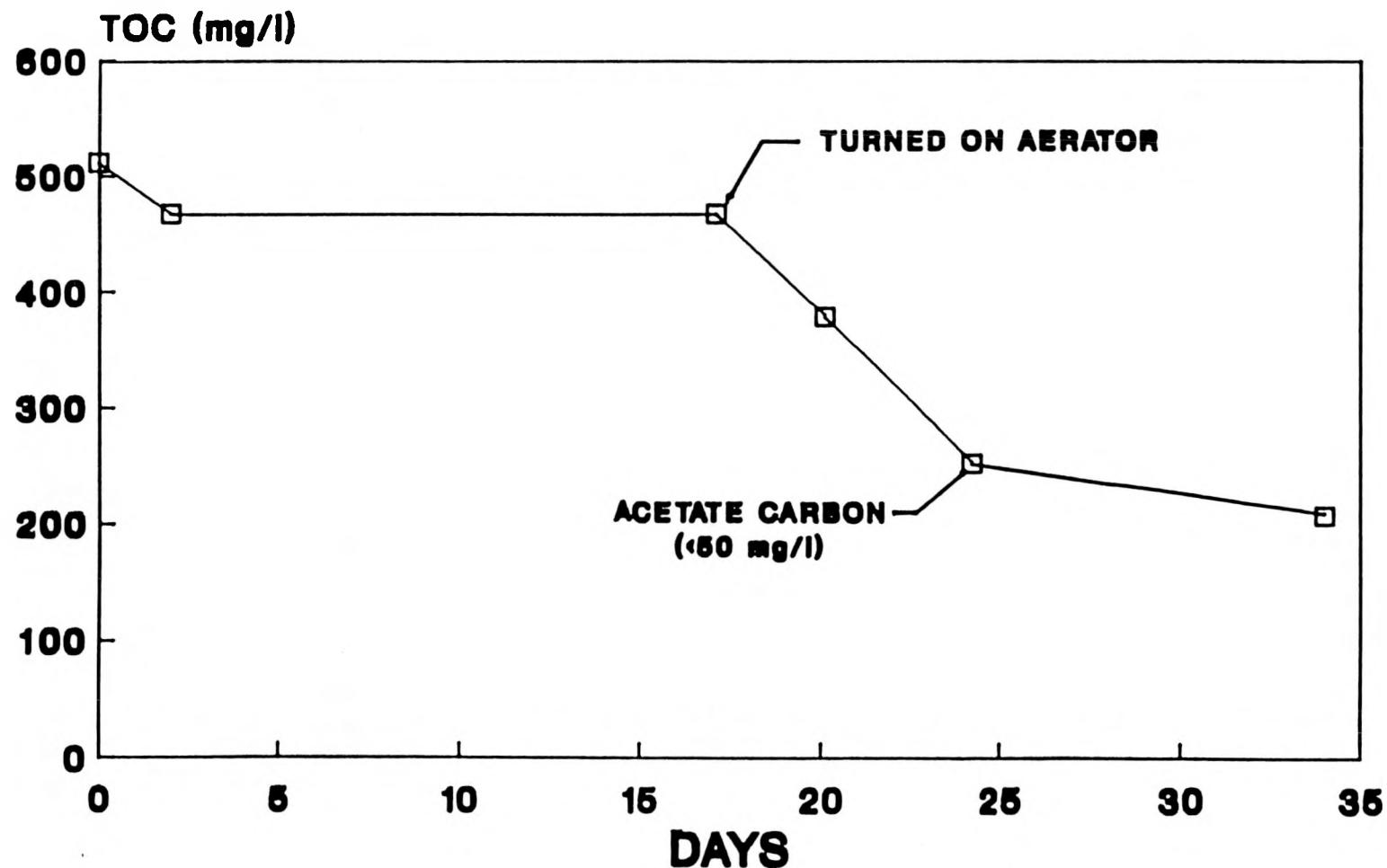
WATER TREATMENT AFTER DENITRIFICATION

- * AERATION TO REMOVE EXCESS ORGANICS
- * AREA IS BEING MONITORED BY WELLS
- * DECANT WATER TO FINAL TREATMENT PROCESS

FIGURE 40

TOC CONCENTRATIONS DURING BIO-OXIDATION SOUTHWEST POND

19



83P-21 MAR 1991

FIGURE 41
AERATION OPERATION



62

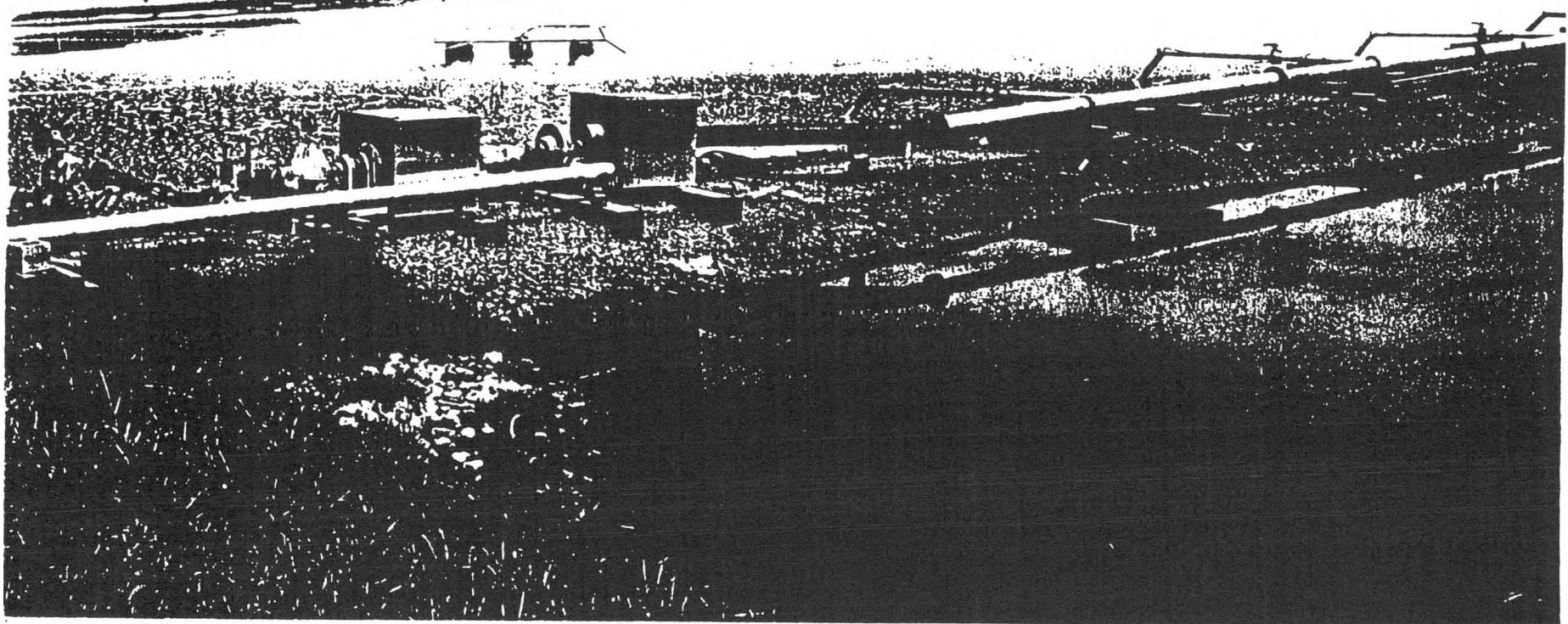
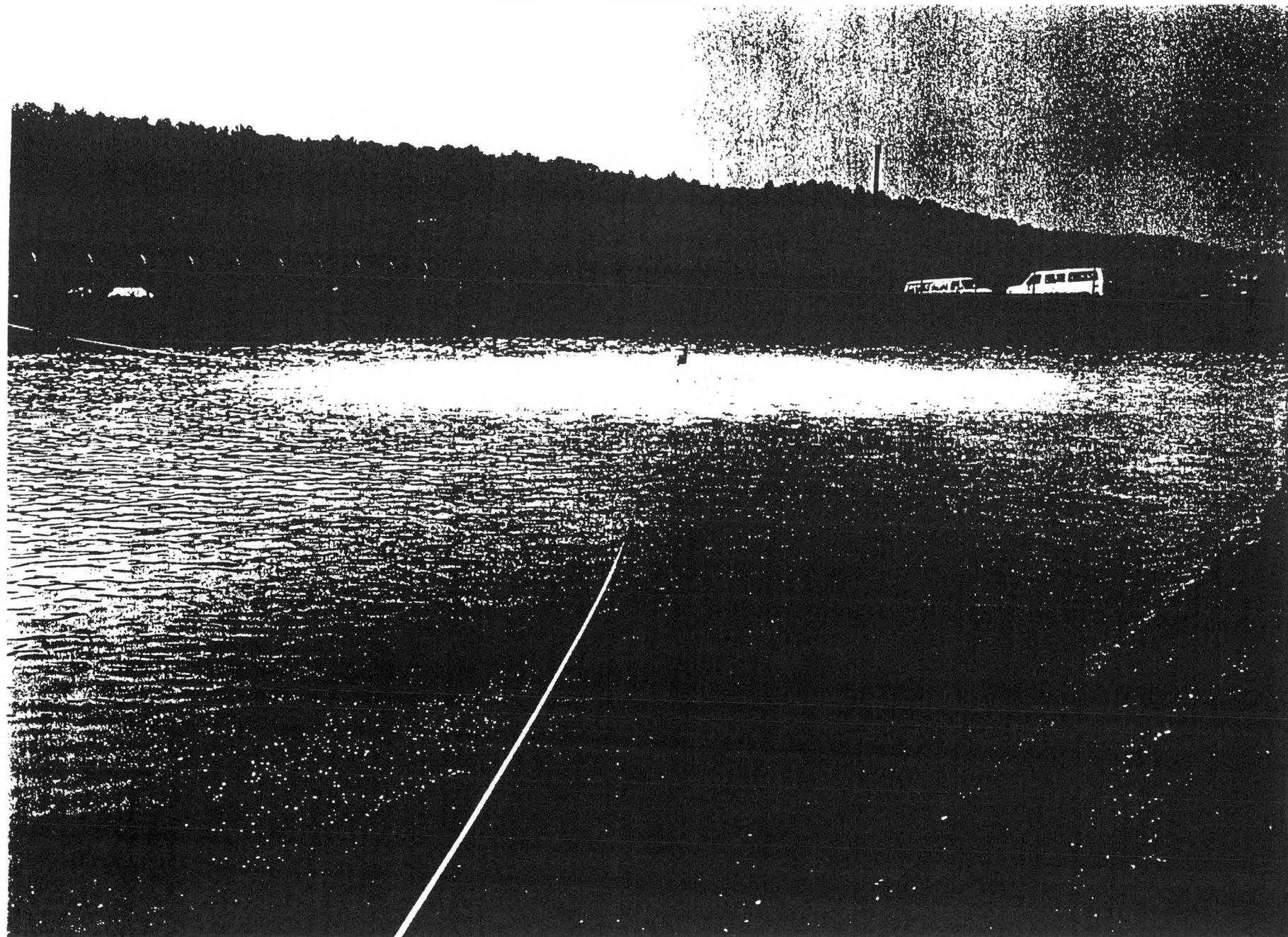


Photo #224081

FIGURE 42
AERATION TO BIODECOMPOSE ORGANICS



63

Photo #216750

FINAL TREATMENT PROCESS

FOR WATER

- LOWER pH TO 2-3 TO REMOVE CARBONATES
- ADD FERRIC SULFATE
- ADJUST pH TO 7-8
- FLOCCULATE USING FERRIC HYDROXIDE
- FILTER WATER
- DISCHARGE WATER
- PUT FILTERED SOLIDS INTO EMPTY POND BASINS

FIGURE 44
CHEMICAL TREATMENT TO REMOVE SOLIDS

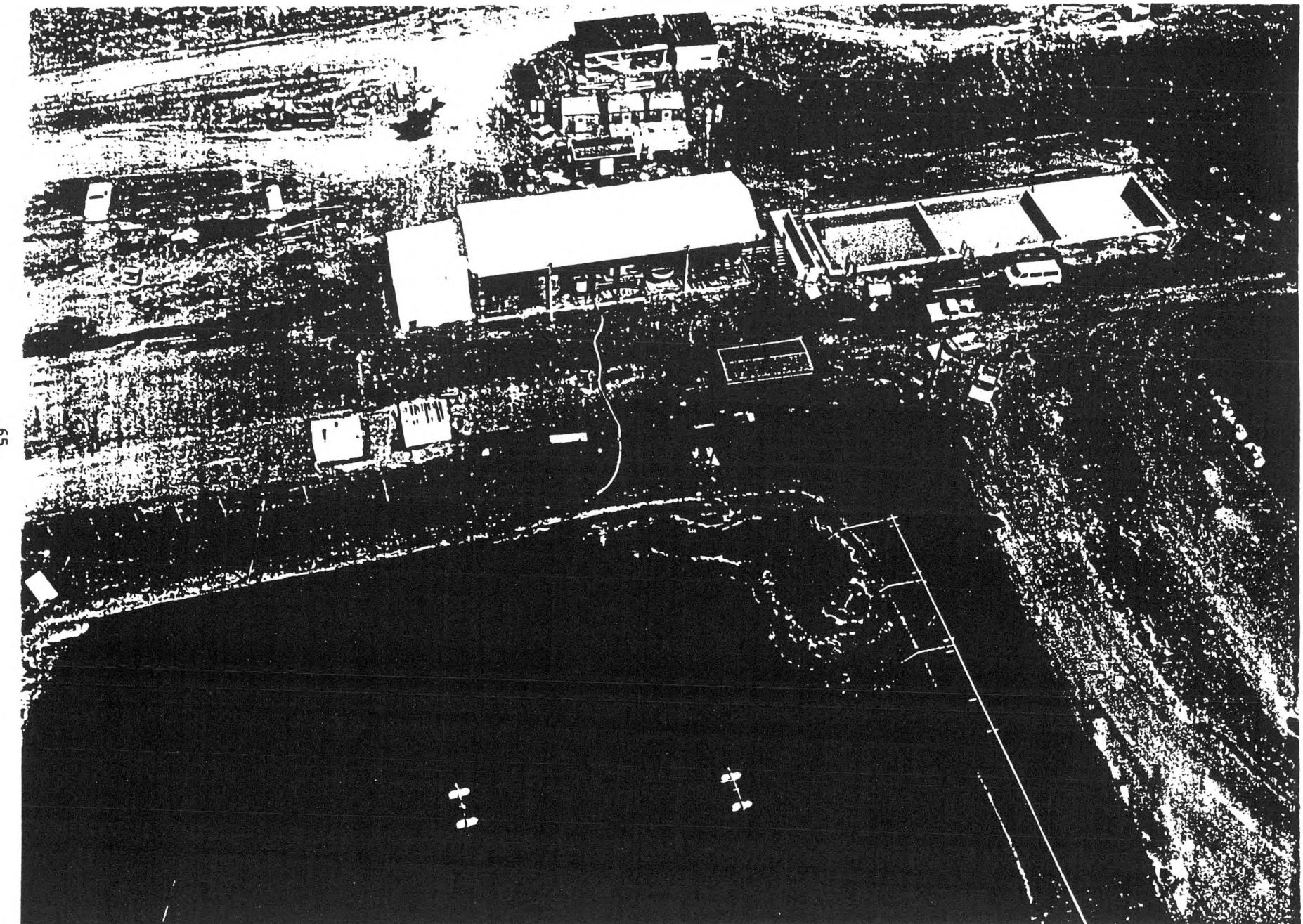


Photo #231946

FIGURE 45

CHEMICAL TREATMENT PLANT

99

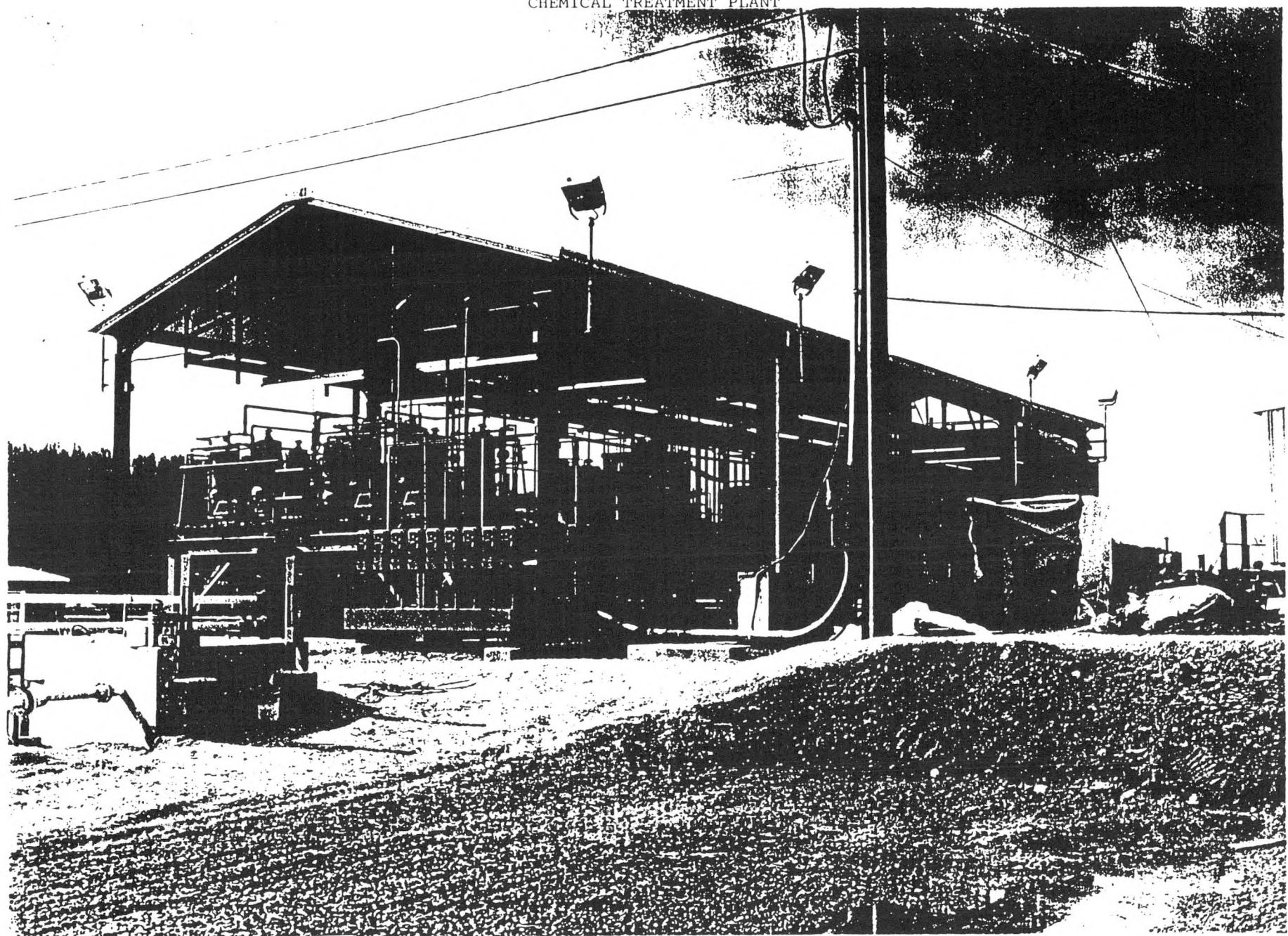


Photo #231490

FIGURE 46

TREATED S-3 POND WATER

	MG/KG	
	BEFORE	AFTER
NO ₃	7,900-46,000	< 50
TOTAL TOXIC ORGANICS	-	0.33
OIL/GREASE	26	2.6
TOTAL SUSPENDED SOLIDS	31	14
Al	583	< 0.06
Ag	-	< 0.02
Ca	847	55
Cd	0.23	< 0.002
Cr	4.8	< 0.014
Cu	5.6	< 0.03
Ni	31	0.32
Pb	2	< 0.1
Zn	4.2	< 0.005
CN	-	< 0.002
pH	1	7-8

L9

REC-8 MAR 1991

FIGURE 47

SOLIDS DEPOSITED IN THE PONDS

(DRIED SOLIDS)

ION	RANGE (MG/KG)	
ION	LOW	HIGH
Ag	< 0.7	3
Al	11,400	21,100
B	18	43
Ba	39	130
Be	< 1	2.9
Ca	29,200	47,000
Cd	2	7
Cr	95	140
Cu	78	100
Fe	1300	3000
Mg	1500	1700
Mn	82	95
Mo	990	2900
Ni	230	570
P	470	990
Pb	18	38
Sn	18	35
Th	61	150
Tl	64	350
Zn	32	58
U	769	1040

FIGURE 48

SOLIDS DEPOSITED IN THE PONDS (CONT'D)

(DRIED SOLIDS)

	RANGE (MG/KG)	
	LOW	HIGH
VOLATILE ORGANICS		
FREON 113		0.0038
ALL OTHER - TOTAL		< 0.1
ACID BASE/NEUTRAL ORGANICS		
PHENOL	4.8	76
1,1,2,2 TETRACHLOROETHANE	1.2	5.5
% MOISTURE (BEFORE DRYING)	78	82
SLUDGE LAYER (FEET, BEFORE DRYING)	2	4.6

FIGURE 49
PONDS AFTER TREATMENT OF LIQUIDS



Photo #236987

FIGURE 50

TREATED PONDS

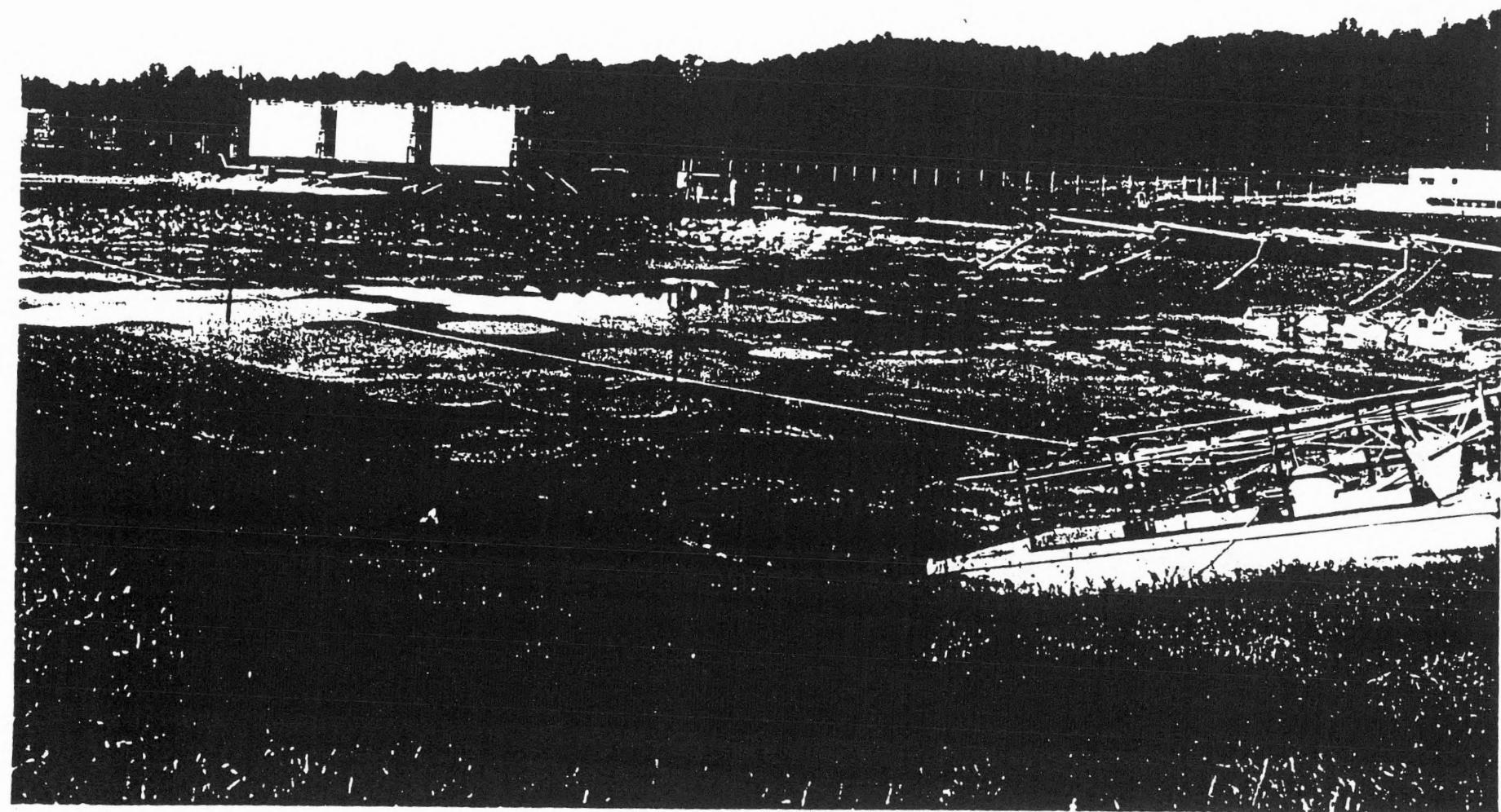


Photo #236563

FIGURE 51
EMPTY PONDS

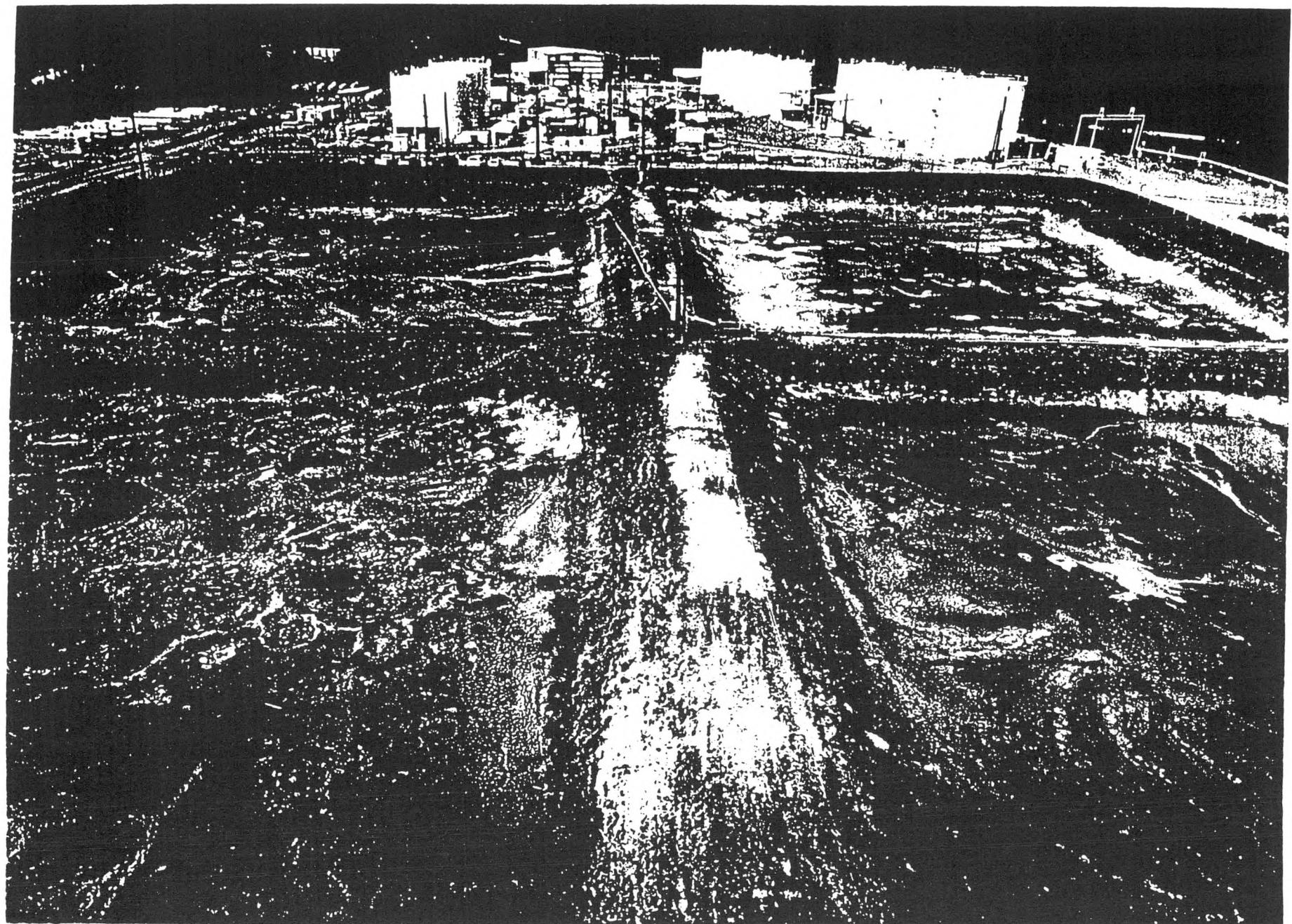


Photo #241762

FIGURE 52
S-3 PONDS AFTER REMOVAL OF LIQUIDS

73



Photo #242130

FILLING EMPTY PONDS

- * ADDED LARGE ROCKS
- * ADDED CLAY LAYER
- * ADDED FLEXIBLE MEMBRANE
- * DRAINAGE NET
- * GEOTEXTILE FABRIC
- * COMPACTED CLAY
- * CRUSHED STONE
- * ASPHALTIC CONCRETE

FIGURE 54

ADDING ROCKS TO PONDS

75

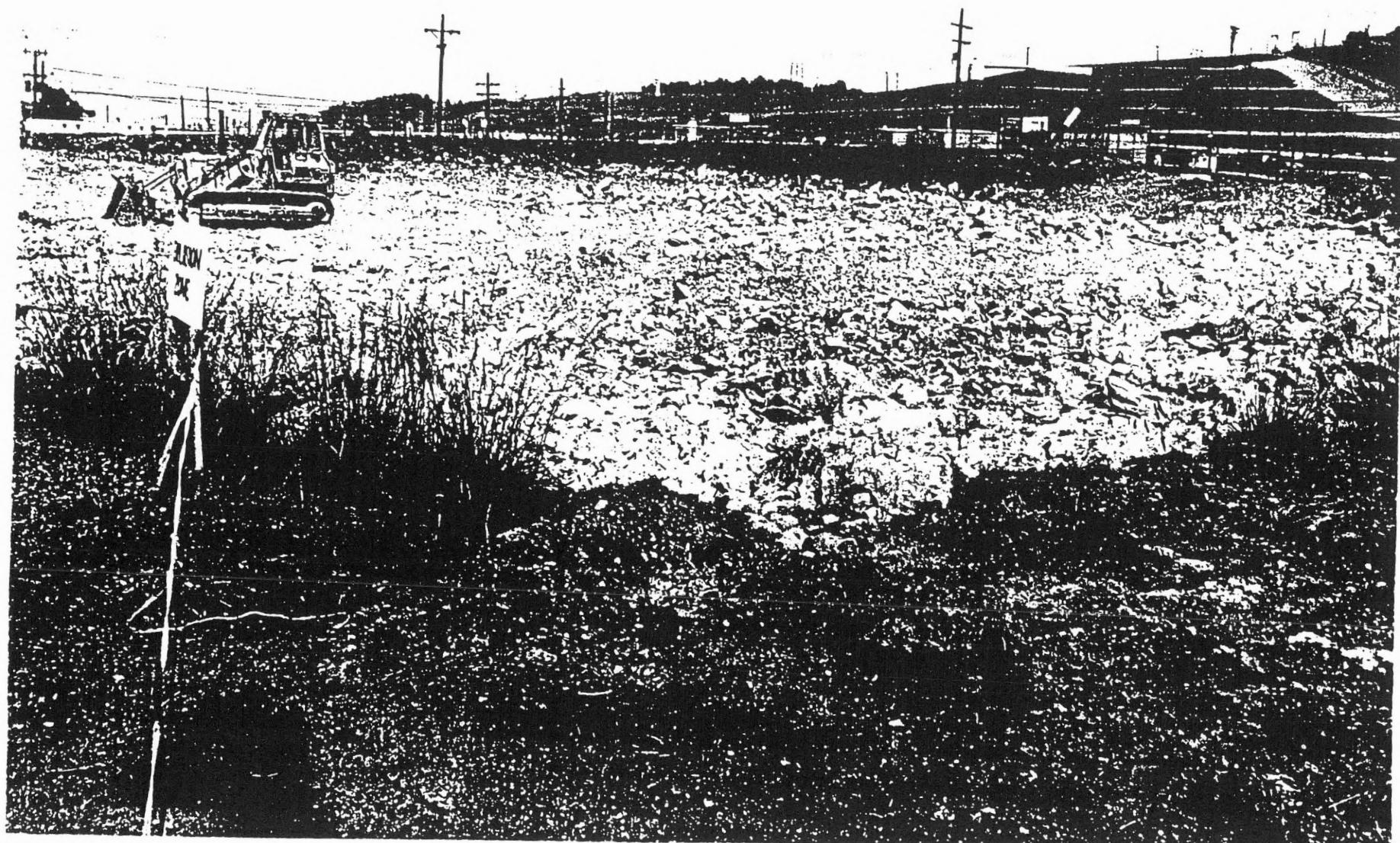


Photo #258129

FIGURE 55

PONDS DURING FILLING PROCESS



76

Photo #258133

FIGURE 56

ADDING CLAY TO PONDS

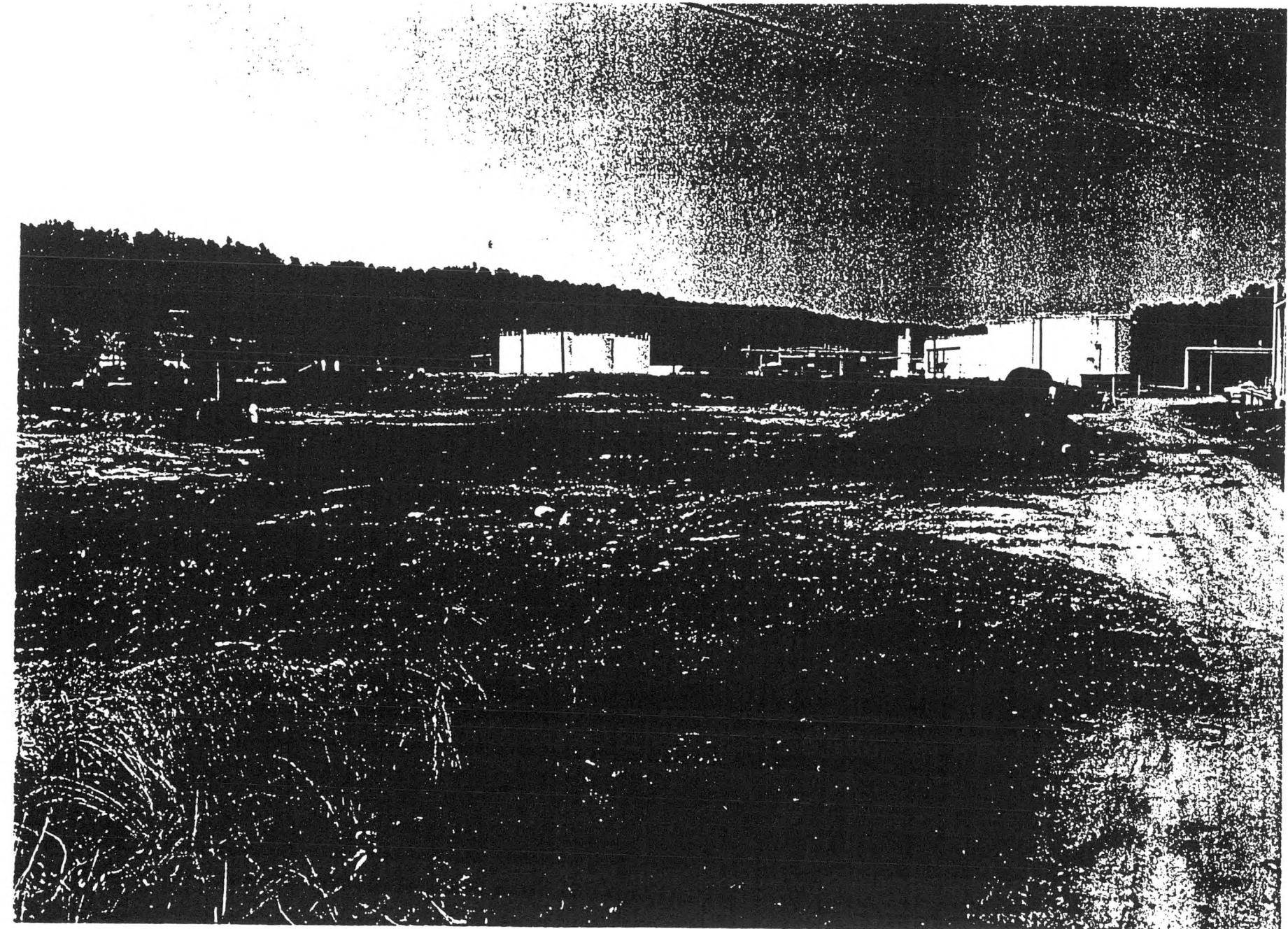


Photo #258539

FIGURE 57
CLAY CAP

78

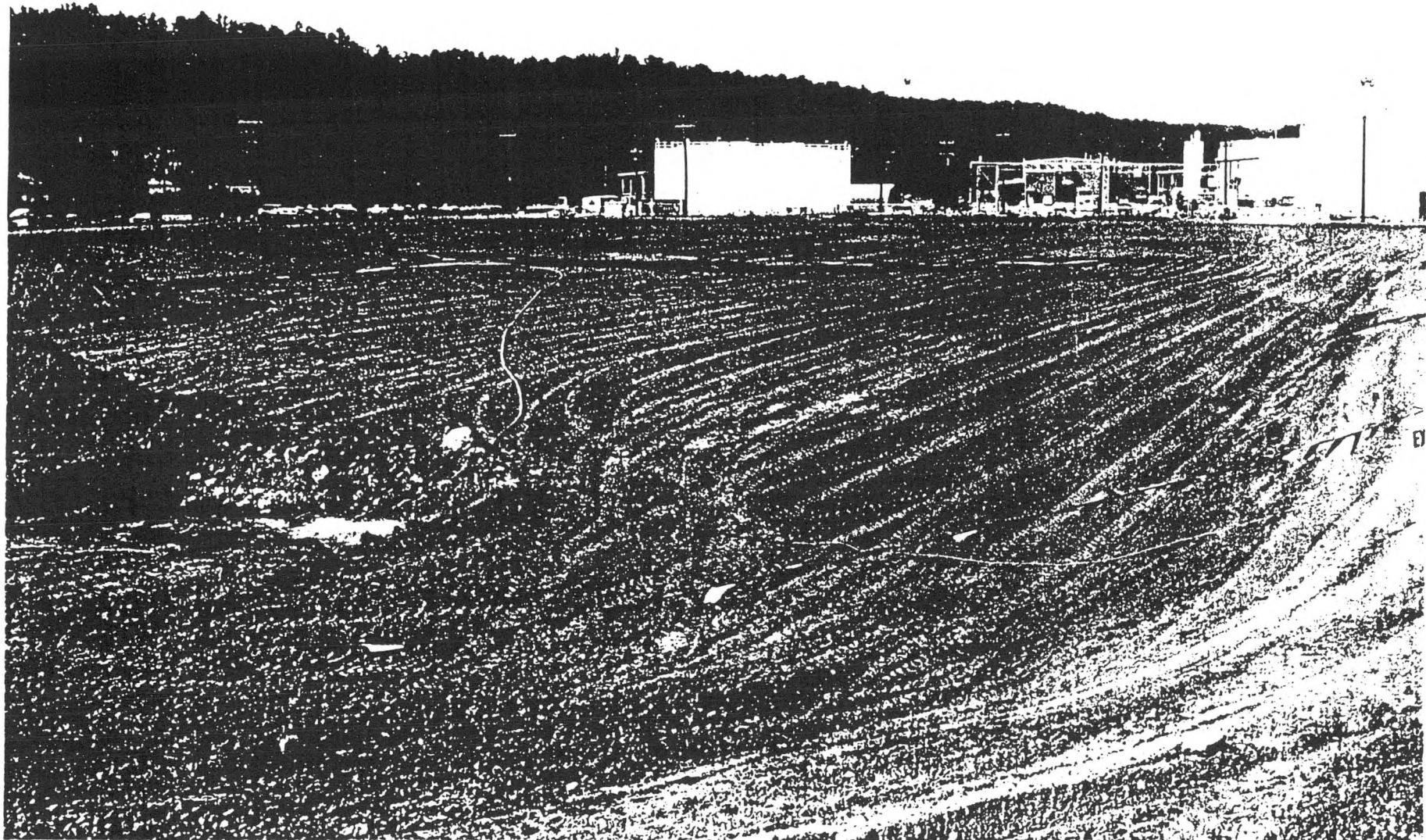


Photo #258720

FIGURE 58
CLOSED POND

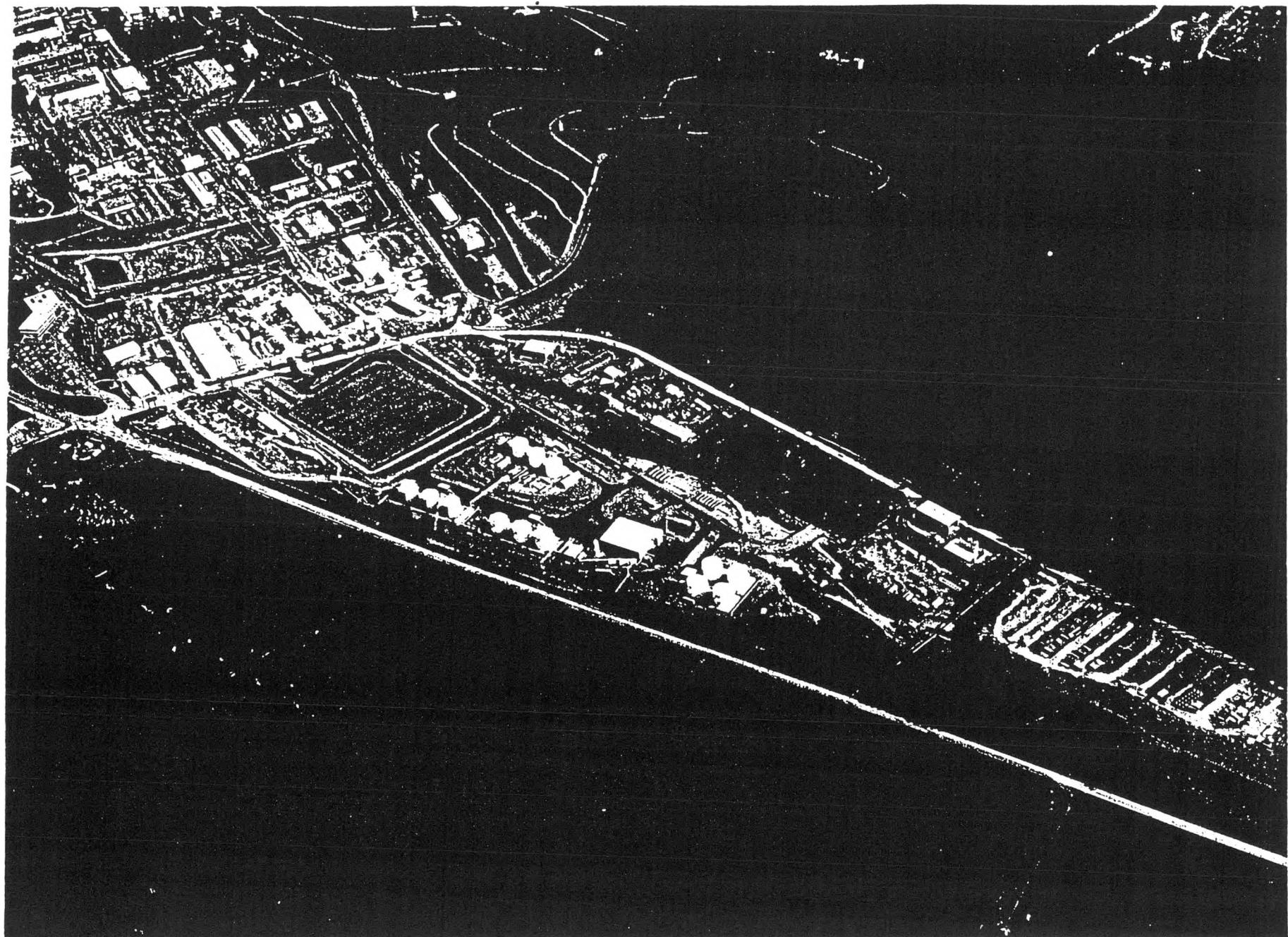


Photo #272658

SUMMARY

- * PONDS ARE CLOSED AND CAPPED**
- * AREA IS BEING MONITORED BY WELLS**
- * NEW TREATMENT PLANT INSTALLED:**
 - * 500,000 GAL TANKS USED AS BIO-REACTORS**
 - * SOLIDS ARE STORED IN STEEL TANKS**

CLOSURE OF OTHER PONDS AT Y-12

- NEW HOPE POND
SETTLING POND FOR PLANT DISCHARGE
CLOSED WITH A MULTI-LAYER CAP
- WASTE OIL PONDS
2 SMALL OIL RETENTION PONDS
PCB/OIL/URANIUM CONTAMINATED SOIL
SOIL REMOVED AND STORED
CLOSED WITH A MULTI-LAYER CAP

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