REPORT TO THE CONFEREES OF THE THIRD SESSION OF THE CONFERENCE IN THE MATTER OF POLLUTION OF THE INTERSTATE WATERS OF THE ESCAMBIA RIVER BASIN (ALABAMA-FLORIDA) AND THE INTRASTATE PORTIONS OF THE ESCAMBIA BASIN WITHIN THE STATE OF FLORIDA

WASTE SOURCE STUDY
AND
REVIEW OF WASTE TREATMENT AND CONTROL PRACTICES
AIR PRODUCTS AND CHEMICALS, INC.
PENSACOLA, FLORIDA

May, 1972

Environmental Protection Agency
Surveillance and Analysis Division
Athens, Georgia

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INTRODUCTION

This report presents an evaluation of waste treatment and waste control practices at the Air Products and Chemicals, Inc. plant located near Pensacola, Florida. The study, conducted by the Environmental Protection Agency during March 6-12, 1972, was requested by the conferees of the third session of the Federal-State Escambia Bay Enforcement Conference held on January 24-25, 1972.

The study objectives outlined in this report are to:

- Characterize and quantify all plant wastes discharged into receiving waters;
- Evaluate and document current waste treatment and control practices, and
- Propose alternatives for the best available technology for waste treatment.

The assistance and cooperation of the Florida Department of Pollution Control, Escambia County Health Department, and Air Products and Chemicals, Inc. are gratefully acknowledged.

PROBLEM SUMMARY

As a result of continuing numerous fish kills and general degradation of the water quality in Escambia Bay, the State of Florida in mid-1969 served Air Products and Chemicals, Inc. a citation and orders specifying a 90 percent minimum treatment of their liquid discharges. At a Federal-State Enforcement Conference held in January 1970, the company was directed to further reduce carbonaceous and nitrogenous wastes by 94 percent and phosphorus wastes by 90 percent.(1) During the February 1971 session of the enforcement conference, allowable daily limits of 17 pounds of 5-day Biochemical Oxygen Demand (BOD_5), 477 pounds of total nitrogen and 35 pounds of total phosphorus were established for compliance by December 31, 1972.(2) It was also recommended that should the specified reductions fail to minimize the problems of excessive enrichment of Escambia Bay, plans should be developed to remove all waste discharges from the bay. The Florida Department of Pollution Control (FDPC) concluded that complete removal was necessary and on December 15, 1971, issued an order to Air Products and Chemicals, Inc., advising the company that no wastewaters containing pollutants or contaminants would be discharged into Escambia Bay after December 31, 1972.(3)

SUMMARY AND CONCLUSIONS

1. Wastewater discharges into Escambia Bay from Air Products and Chemicals, Inc., Escambia Plant, do not presently meet conference established limits. It is very doubtful that the company will meet the conference recommendations by December 31, 1972. Significant reductions in waste loads have been accomplished through source controls and the closing of two plant processes (NPK and methanol). However, pollution abatement efforts within the past three years have been partially offset by a two-fold increase in the production of nitrogenous materials. The success of pollution abatement efforts, as they relate to conference recommendations, are demonstrated by the following comparisons:

	Waste Load (lbs/day)				Percent Removals		
Parameter	1969 Base Load	1969 Study	Conf. Limits	Present Study	Base Load	Conf. Recom.	Since 1969 Study
Total Nitrogen	15,100 1/	5,650	477	(3,190) 2/ 2,610	(79) 2/ 83	94	(44) 2/ 54
BOD_5	--	290	17	185	--	94	40
Total Phosphorus	--	320	35	39	--	90	88
Flow (MGD)	--	5.2	--	1.84	--		--

1/ The 1969 base load was established by the company in correspondence with the State of Florida Department of Air & Water Pollution Control.

2/ Estimated added effect on the plant effluent of wastes presently being retained in Echo Pond, assuming a 60 percent nitrogen removal.

2. The dominant pollutants in the plant effluent are nitrogenous compounds. The final waste effluent is a neutral, unheated liquid containing relatively low concentrations of carbonaceous material and nonfilterable residue. Relatively low concentrations of organic compounds from the dinitrotuluene operations were detected in the discharge into Escambia Bay. Some of these compounds are known to be toxic to fish at concentrations greatly in excess of levels found in the effluent and the effects of other compounds on aquatic life are unknown. Other contaminants observed in the effluent were in low concentrations and are not believed to adversely effect water quality in Escambia Bay.

3. Process wastes at the Escambia Plant are collected in a network of open ditches flowing to treatment ponds consisting of four biological ponds (total area - 65 acres), two settling ponds (total area - 4 acres), and one percolation pond containing 14 acres. Wastes from the treatment system flows through a common discharge into Escambia Bay.

4. Alpha, Bravo and Charlie Ponds are unlined ponds constructed on pervious material and, therefore, unsuitable for the treatment or storage of concentrated nitrogenous waste. However, the ponds do intercept a portion of the contaminated groundwater and surface drainage and provide limited regulation of pond discharge. The ponds are located in the natural drainage of a small watershed which tend to flush out during periods of heavy rainfall. Flood gates have been installed on Bravo and Charlie Ponds, creating approximately 75 additional acre feet of storage for discharge regulation.

5. Nitrogenous wastes are difficult to treat effectively and the problem is compounded by contaminated groundwater. Contaminated groundwater seepage, the most complicated aspect of the problem, accounts for over 50 percent of the total nitrogen in the plant effluent. Study data showed a 720 percent increase in the total nitrogen concentration from the effluent of Charlie Pond to the effluent of Alpha Pond -- no wastes are discharged between these points. The groundwater contamination is primarily due to past waste control practices; however, the amines waste percolated through George Pond and the DNT waste seepage from Delta Pond are existing contributors. This problem must be controlled to significantly reduce the nitrogen discharged into Escambia Bay. Although the company has previously installed approximately 85 shallow well points for monitoring groundwater quality, additional investigations will be required to determine the dispersion of wastes in the contaminated groundwater zone and feasibility of corrective methods.

6. The diverse nature of the various plants operated within the complex complicates the development of an effective treatment system. Several methods of waste treatment were examined in preparation of this report. Some of the methods examined include those previously investigated by Air Products and reported to the FDPC. Methods investigated were ion exchange, spray irrigation, deep well disposal, ammonia stripping, and biological (nitrification-denitrification) treatment. The following conclusions were reached:

- Ion exchange would produce a waste stream unusable for product recovery due to high concentrations of salts other than ammonium nitrate. Disposal of the concentrated waste stream would present a difficult problem.
- Spray-irrigation has limited use in the area due to heavy rainfall during the growing season. Over applications of nitrogen wastes would leach rapidly through the pervious soil causing groundwater contamination.
- Deep well disposal was determined by the company to be unsuitable for the total plant effluent due to the large volume of waste and the 90 percent reduction requirement of the FDPC. Although EPA does not encourage this disposal practice, this method could possibly be used for disposal of the treated process wastes, which would not include seepage, runoff, etc.
- Stripping of ammonia with air or steam may be suitable for treatment of the amines and interceptor ditch waste. Steam stripping with ammonia recovery or distillation may be necessary for air quality protection.
- Biological denitrification for nitrate removal in combination with ammonia stripping is considered to be the best alternative.

7. In-plant survey data and discussions with company personnel indicate that it would be feasible to reroute the following plant waste streams for more effective treatment:

- The amines plant waste stream is a very alkaline waste (pH 11.4) with high nitrogen and carbon concentrations. This waste, presently discharged at an average rate of 225 gpm into the percolation pond and contributing to the groundwater contamination problem, could probably be effectively treated in Echo Pond. The excess alkalinity could be used for neutralization of the acidic DNT waste, now being neutralized by caustic. This stream would also provide a carbon source necessary for biological treatment. The high ammonia concentration should be reduced, possibly by stripping prior to neutralization.
- The ammonia plant waste stream, a very alkaline waste (pH 11.9) now flowing into Charlie Pond at an average discharge of 88 gpm, could be combined with the amines waste, piped to the DNT plant for neutralization and treated in Echo Pond.
- Wastes from the PVC plant could be routed through Echo Pond providing a necessary phosphorus supply for the microbial population.

This arrangement appears to be suitable for treatment of the combined streams for nitrogen and carbon removal in Echo Pond plus utilization of the alkalinity and acidity of individual streams for neutralization. The combined flow would average approximately 600 gpm with a COD:NO_3: Total P ratio of 230 to 31 to 1. One potential problem with this scheme of treatment is the unknown biological treatability problems associated with the nitro compounds in the DNT waste. If these compounds greatly retard biological action, as might be suspected, extended holding periods or removal of the compounds at the source may be required. Activated carbon adsorption, a method previously tested by the company, would probably be an effective method of removal.

Studies to determine the compatibility and treatability of these waste streams can be accomplished through the use of jar studies now being conducted by the company and through plant-scale operations with Echo Pond which was recently placed into service.

8. At the present rate of discharge, the BOD_5 effluent concentration would have to be reduced to 1.1 mg/l to meet conference recommendations. A seven-day average effluent BOD_5 concentration of 10 to 15 mg/l (155 to 230 lbs/day), with maximum daily fluctuations of 125 percent, is a much more reasonable, achievable limit.

RECOMMENDATIONS

1. The company should intensify their source control program which has accomplished significant results to date.

2. Alpha, Bravo, and Charlie Ponds should be operated primarily as polishing ponds and not for the treatment of concentrated nitrogen wastes.

3. All untreated DNT waste leaks should be eliminated from Delta Pond.

4. The use of George Pond, a percolation pond treating wastes from the amines plant, should be discontinued.

5. The company should investigate the following compatibility and treatability modifications and submit a report of the findings to the conferees by June 1, 1973:

    - Combine the waste streams from the amines and ammonia plants. Air strip or otherwise remove the ammonia from the combined stream and pipe the treated stream to the DNT plant to neutralize the acidic DNT waste. Treat the combined waste in Echo Pond for nitrate removal. The amines and ammonia waste should provide a necessary source of carbon. Treatment of the amines discharge in this manner would also eliminate George Pond, a percolation pond which is contributing to the groundwater contamination problem.
    - Divert Fox Trot Pond effluent to Echo Pond. This stream will provide part of the phosphorus necessary for proper microbial growth in Echo Pond.

- Investigate the treatability of the nitro compounds in Echo Pond. If these compounds prove to seriously inhibit microbial growth, consideration should be given to removal at the source by use of activated carbon or other methods.
- Additional clay-lined ponding capacity may be required. An aerated treatment pond or another form of aerobic treatment may be desirable following treatment in Echo Pond.

6. The company should conduct an investigation of the groundwater contamination problem and submit a report of the finding to the conferees by June 1, 1973. The report should include:

   - The extent of the contaminated groundwater zone showing vertical and horizontal concentration profiles.
   - Flow patterns of the contaminated groundwater.
   - Alternatives for reducing or eliminating this nitrogen contribution to the Bay.
   - A specific implementation schedule for the selected alternative.

7. A groundwater monitoring system should be established to determine the long-term changes in groundwater nitrogen concentrations. Monthly samples should be collected from a minimum of three points located between Area "B" and the bay. Samples should be analyzed for TKN, NH_3, NO_2-NO_3 and reported to the FDPC.

8. In general, all surface water runoff from the drainage basin should be routed around the treatment system. An exception should be made in any

immediate plant areas where runoff contains high nitrogen concentrations or other contaminants. Wastes from these areas should be collected and treated with process waste.

9. The conferees should increase the allowable BOD_5 effluent limit from 17 to 200 pounds per day. Established loadings should apply as seven-day average values with average daily loads permitted to vary up to 125 percent of this value. The following effluent criteria would apply:

	Maximum Effluent Loadings (lbs/day)	
Parameter	7-day Average	Maximum Daily
5-day Biochemical Oxygen Demand	200	250
Total Nitrogen	477	600
Total Phosphorus	35	44

10. Spray irrigation appears to be one of the most promising schemes for utilizing the waste and should receive further consideration in spite of apparent problems.

PLANT DESCRIPTION

GENERAL

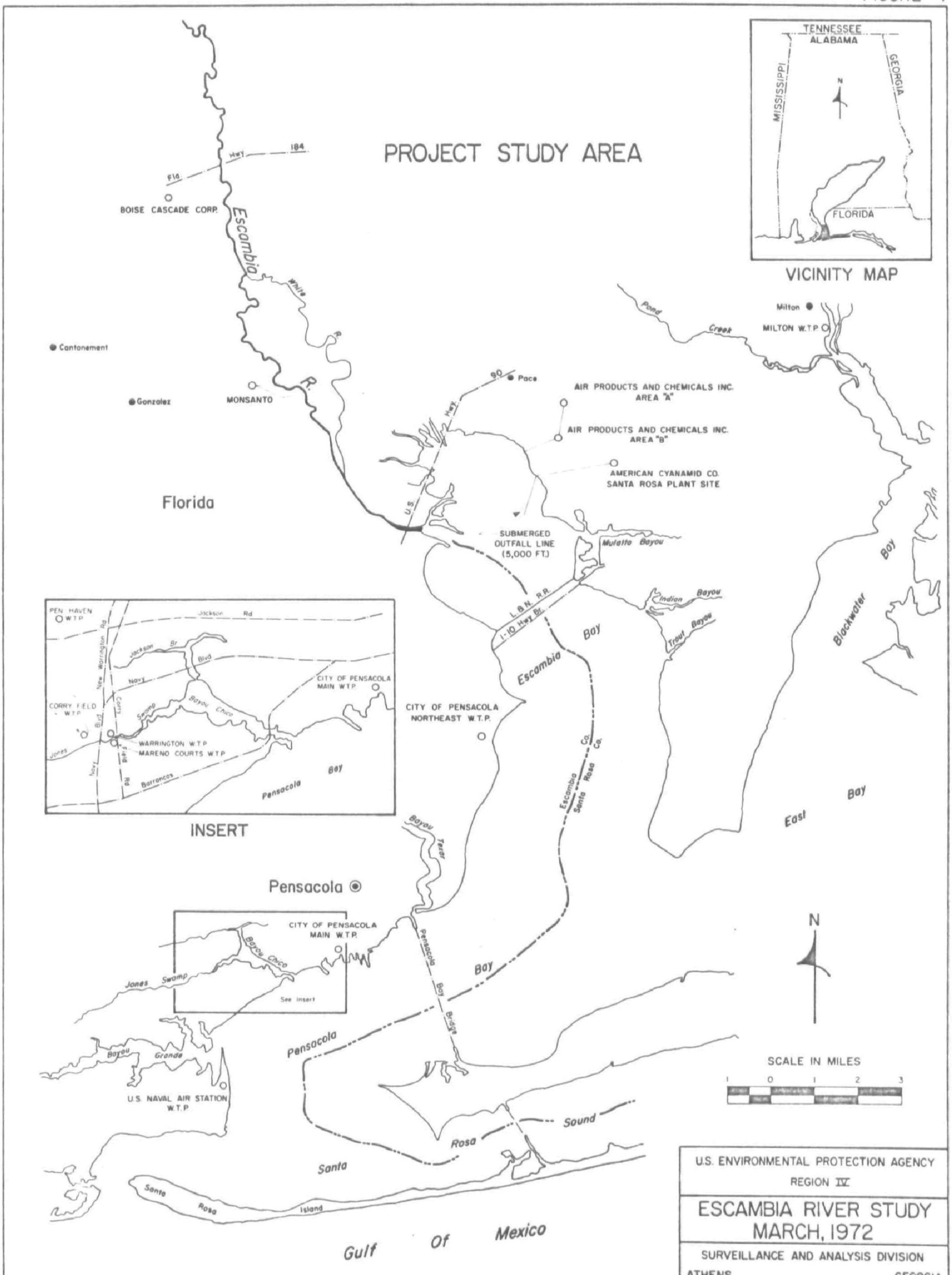
The Air Products and Chemicals, Inc., Escambia Plant, was originally constructed in 1955 on a 1600-acre tract of land located north of Pensacola near Pace, Florida (Figure 1). The plant produces ammonium nitrate, ammonia, nitric acid, amines, urea, dinitrotoluene, and polyvinyl chloride. Total plant production of nitrogenous materials has approximately doubled in the past three years. Since the 1970 Enforcement Conference, the methanol and mixed fertilizer (NPK) plants have been closed. The company reported that the methanol operation was temporarily shut down because of a shortage of natural gas and the NPK operation was terminated because of the difficulty and expense of controlling the nutrient waste load. Treated process wastes are discharged through a common outfall into the upper northeastern area of Escambia Bay (Figure 1). Domestic wastes are treated in several septic tank systems and discharged to subsurface drain fields.

Operations are grouped into two separate areas approximately one mile apart (Figure 2). The complex of plants to the north are contained in Area "A" and the plants to the south in Area "B". Separate grouping of the processes was required by insurance companies for safety reasons.

PLANT PROCESSES

Ammonia - Natural gas is reformed and reacted with atmospheric nitrogen to produce ammonia which is used in other plant processes. Additional ammonia is shipped in to augment plant usage.

FIGURE 1

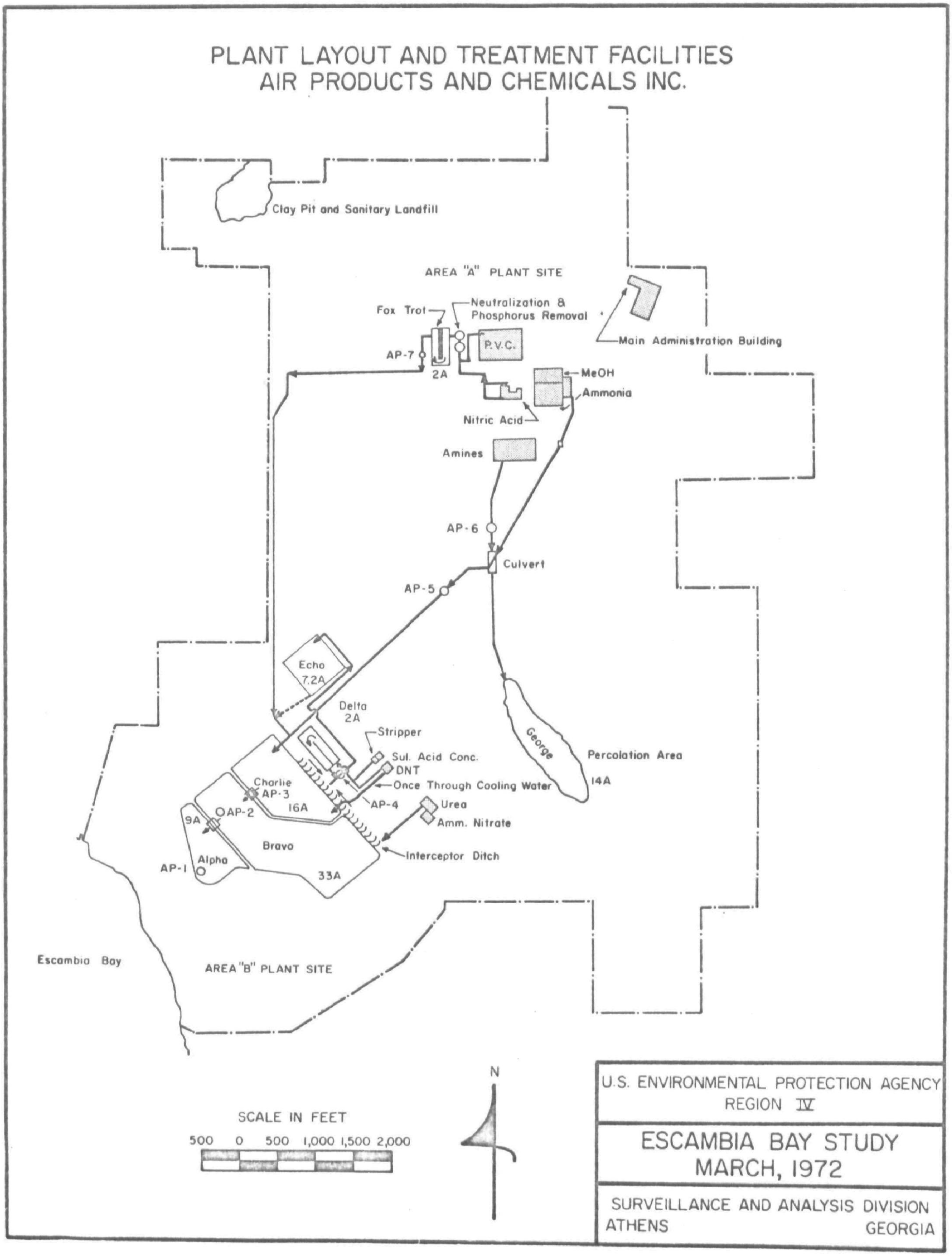


U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION IV

ESCAMBIA RIVER STUDY
MARCH, 1972

SURVEILLANCE AND ANALYSIS DIVISION
ATHENS GEORGIA

FIGURE 2


PLANT LAYOUT AND TREATMENT FACILITIES
AIR PRODUCTS AND CHEMICALS INC.
Clay Pit and Sanitary Landfill
AREA "A" PLANT SITE
Fox Trot
Neutralization & Phosphorus Removal
Main Administration Building
AP-7
P.V.C.
2A
MeOH
Ammonia
Nitric Acid
Amines
AP-6
Culvert
AP-5
Echo 7.2A
Delta 2A
Stripper
Sul. Acid Conc.
DNT
Once Through Cooling Water
Charlie
AP-3
AP-4
16A
OAP-2
9A
Urea
Amm. Nitrate
Bravo
Interceptor Ditch
AP-1
Alpha
33A
George
Percolation Area
14A
Escambia Bay
AREA "B" PLANT SITE
SCALE IN FEET
500 0 500 1,000 1,500 2,000
N
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SURVEILLANCE AND ANALYSIS DIVISION
ATHENS GEORGIA

Amines - Methanol and ammonia are used to produce mono-, di-, and tri-methylamines. These are intermediates used by other companies in production of various products.

Nitric Acid - This product is a major component in the production of ammonium nitrate, and is produced by oxidizing ammonia.

PVC - Vinyl chloride is imported, polymerized and shipped to other processors. This operation is not integrally connected with other plant operations since nitrogenous materials are not utilized.

Ammonium Nitrate - Ammonia and nitric acid are reacted to form ammonium nitrate, which is used principally for fertilizer.

Urea - Ammonia is reacted with carbon dioxide to form urea. This product has a high nitrogen content and is used for fertilizer, protein feed supplement and other uses.

Dinitrotoluene - Toluene is imported and reacted with a mixture of nitric and sulfuric acid to form dinitrotoluene. The product is sold for use in the production of urethane products.

NPK - The NPK plant is now permanently closed, but formerly produced mixed agricultural fertilizers containing nitrogen, phosphate and potash.

WASTE TREATMENT FACILITIES

The existing treatment facilities (Figure 2) consist of a system of settling, percolation and biological treatment ponds. Waste is conveyed from Area "A" to the treatment ponds by two paved ditches. Prior to construction of Alpha, Bravo and Charlie Ponds in 1971, the total plant effluent, with only source control treatment, was discharged into the swampy area where the ponds are now located. Echo Pond was recently constructed for aerobic/anaerobic biological treatment of the concentrated waste from the DNT plant. Delta and Fox Trot ponds were designed primarily for settling while George Pond was designed for percolation.

Alpha, Bravo and Charlie Ponds encompass 9, 33, and 16 acres, respectively. Construction on the ponds began in late 1970, and the system was placed in service during March 1971. The ponds, constructed in the natural drainage of a watershed, were formed by constructing dikes on the typical lowland muck. Bottom material permits a free interchange of groundwater and wastewater. Waste streams discharge into Charlie Pond through two paved ditches from the PVC and ammonia plants; each carry 100 to 150 gpm of waste. The company reported that approximately 700 gpm of once-through cooling water from Area "B" also discharges into Charlie Pond. These are the only surface discharges flowing into the three ponds; however, a considerable amount of contaminated groundwater apparently does infiltrate into this system. Groundwater infilteration is evident by both the increased volume of wastewater and the increased nitrogen concentration levels as the waste flows through the pond system. At the present time, Echo Pond is being filled and has no discharge. When it begins overflowing later this summer, the discharge will be into Charlie Pond.

These ponds are located in the natural drainage of a watershed and are susceptible to flushing from heavy rainfall. To help alleviate this problem, the State of Florida required the company to install floodgates above the weirs in Charlie and Bravo Ponds which increases the storage by approximately 75 acre feet. The installation of floodgates has prevented complete flushing; however, the flow varies considerably with rainfall.

The failure to clear the pond area of trees and other vegetation prior to construction has initiated a need for the frequent removal of floating debris. However, the vegetation removal accounts for some nitrogen reduction. The heavy growth of algae and duck weed in the ponds cause a noticeable green color. Prior to pond construction, the total plant effluent flowed across the muck area where the ponds are now located. Leaching of waste materials from the pond bottoms will continue to occur until the system is stabilized.

Delta Pond, a two-acre asphalt-lined pond, provides a holding period for the DNT waste. At the time of the survey, the lining above the liquid level was badly cracked. The yellow color of the waste in the interceptor ditch below Delta Pond and the presence of toluene compounds in the effluent of Alpha Pond indicated that seepage was occurring. Water from Delta Pond is pumped into Echo Pond for biological treatment. An emergency overflow is provided from Delta to Charlie Ponds.

The interceptor ditch is a shallow excavation immediately above the northwest portion of Charlie and Bravo Ponds. The pond dikes form the lower bank of the collection ditch (Figure 2). The primary purpose of this ditch is to intercept contaminated groundwater which seeps from the slope below Area "B". Approximately 70 gallons per hour of waste discharged from the urea and ammonia nitrate operations flow into this collection ditch along with surface runoff from Area "B" and some groundwater seepage. A pump, installed in the northwest end of the ditch, pumps waste into Echo Pond. It appears that a large portion of the infiltration into Bravo and Charlie Ponds comes from the collection ditch. Since the water level in the ditch is normally higher than the water surface in the ponds, a positive head is available for flow through and under the dike. An emergency spillway is provided from the ditch to Bravo and Charlie Ponds.

The company has approximately 85 shallow monitoring wells distributed around the property. Company information indicates that nitrogen levels within the wells are very sporadic, varying from trace concentrations up to 1,000 mg/1. This condition was probably caused by former indiscriminate discharge practices, including leaking flumes, plant leaks, spills, leakage from Delta Pond and the amines percolation pond.

Echo Pond is a newly constructed 7.2 acre, clay-lined pond. Wastes from Delta Pond and the collection ditch are pumped through a common four-inch header to this new facility. At the time of this study, the pond was being filled and had no discharge. The pond is designed for nitrification-denitrification, neutralization and biological oxidation of the concentrated nitrogen waste. After treatment, the effluent will flow into Charlie Pond.

Fox Trot Pond is a two-acre, clay-lined pond designed to settle material from the PVC operation. Pretreatment of this waste is accomplished by the addition of magnesium oxide for phosphate precipitation. The polymers and magnesium oxide slurry from this operation settle rather easily, and the first section of the pond has a pronounced blanket of white solids visible through the water. Cooling water from the nitric acid plant is also discharged to this pond. After settling and biological treatment, the waste flows via a paved ditch into Charlie Pond.

George Pond is an unlined, 14-acre pond constructed of sand. It is designed as a percolation pond for the amines waste and undoubtedly contributes heavily to the nitrogen problem in the groundwater. Although the upper end of the ditch conveying the amines water is lined, the lower portion is unlined permitting loss of some of the waste before it reaches the pond.

Waste from the ammonia plant flows into Charlie Pond through a concrete-lined ditch. This ditch is in need of repair and permits the loss of some waste before it reaches Charlie Pond.

PREVIOUS WASTEWATER STUDIES

REPORT REVIEWS

Two wastewater studies were performed for the Company by Black, Crow and Eidsness, Inc., consulting engineers, located in Gainesville, Florida.(4)(5) The first report, dated October 8, 1969, resulted from a citation issued by the Florida Air and Water Pollution Control Board charging the Company with the pollution of Escambia Bay. The directive required the company to submit to the Board within 45 days an engineering report on proposed waste treatment. Major conclusions and recommendations of this report were:

- Treatment facilities at Air Products consisted of source controls, a percolation system and an impoundment.
- Process wastewater flow was about 5.0 MGD with nitrogen and Chemical Oxygen Demand loadings ahead of source controls about 14,800 and 58,900 pounds per day, respectively.
- Estimated treatement removals, in percent were:

	Nitrogen	COD
Source control equipment only	62	59
Overall system	62	97

- Recommended additional studies:
  - A. Wastewater characterization
  - B. Evaluation of treatment methods, including:
    1. Additional source controls
    2. Spray percolation

3. Spray irrigation; and

4. Subsurface disposal

C. Treatability and pilot plant studies.

D. Design, engineering, construction, and startup.

- The proposed program could be accomplished in 35 months with a target date of October, 1972.

The second report, dated June 12, 1970, was in response to a directive from the Florida Department of Air and Water Pollution Control to report on accomplishments and proposed further actions. Major conclusions were:

- All improvements proposed for installation by January 1, 1970 had been accomplished,
- Existing waste control improvements afforded 89 percent nitrogen removal from plant wastewater streams,
- Overall nitrogen removal at the outfall had been reduced to about 68 percent due to seepage.
- Estimated loading due to seepage were:

	Amount
Flow	1.3 MG
Nitrogen (total)	3,400 lbs.
Phosphorus (total)	250 lbs.
Potassium	410 lbs.

The Company, with the assistance of their consultant, investigated and reported findings on various waste treatment methods in a 1971 report to the FDPC.(6) Listed below are the conclusions taken directly from the report.

Spray Irrigation Problems

1. Nitrogen must be applied at rates vegetation can assimilate it. Water volume must be applied at rates that can hydrologically be handled.
2. In the case of the Escambia Plant, hydrological considerations will govern irrigation application. The land area and reservoir requirements make the entire approach impractical.
3. The Escambia Plant area experiences some 80 inches of rainfall per year. The greater portion of this rainfall coincides with the local agricultural growing season.

Ion Exchange Application Problems

1. Ion exchange of the outfall would generate 28,800 gallons per day of 15% salt solution which cannot be disposed of.
2. Ion exchange results in a 50-fold increase in ion concentration.
3. Chloride concentrations in the outfall exceed permissible limits for safely concentrating the regenerant stream.
4. The regenerant solution would contain 60% of salts other than ammonium nitrate, excluding recycle to process.

Subsurface Disposal Application Problems

1. Escambia must inject its entire outfall to achieve desired treatability.

2. Based on data developed from Monsanto's injection well, limited receiving aquifier permeability precludes subsurface disposal of Escambia's entire outfall.

3. Recent legislation may require 90% treatment prior to injection.

Activated Carbon Application Problems

1. The removal of DNT plant wastewater COD load was achieved.

2. Activated carbon did not significantly reduce the total nitrogen load of the wastewater.

3. The expanded biological pond system is anticipated to require this carbon source to achieve the maximum nitrogen treatability.

4. A source-control project has resulted in a 50% reduction of the DNT plant total N-load.

SOURCE CONTROLS

This report will not attempt to discuss fully the in-plant controls which have resulted in waste load and flow reductions; however, a listing of the controls and resulting reductions are shown. This listing was provided by a company letter dated April 24, 1972, which is included in Appendix D.

Escambia Plant Source-Control Program

Timetable	Project
Pre-1970	1. Cooling towers.
	2. Catalytic combustion on HNO_3 plant.
	3. Ammonia recovery from copper liquor system.

4. Aqua ammonia stripper and vaporizer system.
5. Methanol by-product disposal (to boilers).
6. Compressor building oil recovery and disposal.
7. Amines by-product disposal (flare).
8. Nitric acid oil recovery and disposal.
9. Methyl methacrylate effluent pH control.
10. Methyl methacrylate effluent stabilization basin.
11. Ammonium nitrate remelt and reclaim system.

After Jan. 1970

1. NPK recycle pond.
2. Recovery of weak aqua ammonia from percolation.
3. HNO_3 concentrator recycle and reclaim system.
4. HNO_3 plant start-up weak acid recovery and recycle.
5. NPK plant shutdown.
6. DNT organics recovery and recycle.
7. Ammonia condensate stripper.
8. Ammonia cascade cooler on cooling tower recycle.
9. Nitric acid recovery and recycle from DNT spent acid stripper.
10. Methanol distillation system put on reboilers.
11. Methanol still bottoms recycled as cooling tower makeup.
12. Ammonium nitrate solutions recovery and recycle.
13. Totally enclosed ammonium nitrate prill loading and bulk conveyor system.
14. NPK gypsum pond removal for agricultural use.
15. Cooling towers.

Load reductions reported by the Company in their 1971 report to the FDPC from source control were:

Flow	1.31 mgd
COD	50,300 lbs/day
Total Nitrogen	12,800 lbs/day
Heat	3.5 mm BTU/hr

Because of difficulties in treating nitrogen waste and in particular a mixture of nitrogen waste from several different processes, this area merits careful study for possible further development. Air Products and Chemicals reported, in the 1971 report to the FDPC, that 99.706 percent of the total nitrogen handled is contained as product and 0.294 percent is lost to the effluent.(6)

STUDY FINDINGS

GENERAL DISCUSSION

Sampling began on March 7, 1972, and continued for five consecutive days. Twenty-four hour composite samples were collected with automatic sampling equipment provided by the company at seven in-plant stations. Station locations are described in Table I and plotted in Figure 2. Automatic samplers and flow measuring equipment are permanently installed at these stations since the company routinely samples these locations. Equal aliquots of samples were collected at roughly 30-minute intervals and pumped into a 2-1/2 gallon jug packed in ice. The sample collected at Station AP-4 was an exception since it was collected by a dripping faucet via a tube attached to the glass jug. This arrangement was necessary because the sample was taken from a force main, and conventional automatic samplers could not be used. All samples were divided at the point of collection for duplicate analysis by the company. Samples collected at Stations AP-1 and AP-4 were also split with the FDPC. Two grab samples of well water were collected. The study plan listing the sample stations and analysis for each sample is included in Appendix E. Table II contains the tabulated data obtained from this study.

Weather conditions were variable during the study, typical of springtime. Temperatures averaged 4^{o}F below normal with a maximum temperature of 72^{o}F and a minimum of 40^{o}F. Rainfall recorded at the Pensacola airport totaled 0.58 inches and occurred on March 8. The company reported 0.55 inches of rain on March 7 and 0.07 inches on March 8 at the plant

Table I

SAMPLING STATIONS AND LOCATIONS
AIR PRODUCTS AND CHEMICALS, INC.

AP-1	Effluent from Alpha Pond
AP-2	Effluent from Bravo Pond
AP-3	Effluent from Charlie Pond
AP-4	Open ditch in Area "B" between the plant and ponds
AP-5	East ditch flowing from Area "A" to the ponds
AP-6	Ditch flowing from Amines Plant to George Pond
AP-7	Effluent from Fox Trot Pond
AP-8	Well water supply

site. Waste flows during the first part of the study were still being influenced by runoff from a 1.85 inch rain which occurred on March 2. The flood control gates installed above the weirs on Bravo and Charlie Ponds provide a considerable amount of extra storage (up to approximately 1.5 feet head) which slowly drains out with the normal plant effluent. This condition makes the flow measuring devices installed at the discharge of Bravo and Charlie Ponds inoperative. It also creates a non-steady state condition which makes it difficult to relate influent to effluent for the three pond system.

Chemical analysis of samples split with the company were in good agreement with the exception of the nitrogen series, particularly the NO_2-NO_3 concentrations. Subsequent resampling of AP-1 through AP-7

TABLE II

AVERAGE ANALYTICAL DATA FOR INPLANT WASTE STREAMS
**** AIR PRODUCTS AND CHEMICALS INC. ****
MARCH 7-12, 1972

STATION	FLOW RATE GPM	T ACDITY CACO3 MG/L	T ALK CACO3 MG/L	BOD 5 DAY MG/L	COD HI LEV MG/L	T ORG C C MG/L	PHOS-T P-WET MG/L	RESDUE V NFLT MG/L	RESDUE T NFLT MG/L	RESDUE TOTAL MG/L	RESDUE T VOL MG/L	T KJEL N MG/L	NH3-N TOTAL MG/L
AP-1	1272	36	23	11.4	52	13.4	2.64	12	17	772	398	86.2	84.9
AP-2	--	32	30	24.5	49	12.0	3.69	9	9	470	135	67.9	65.9
AP-3	--	10	43	41.0	27	7.4	1.95	8	8	248	56	13.7	11.0
AP-4	119	115	73	107.3	487	161.8	5.14	22	56	5962	971	231.1	230.6
AP-5	88	0	2552	57.0	119	64.8	1.00	15	44	3672	398	26.4	22.8
AP-6	225	0	1664	--	1974	662.4	0.05	--	14	783	100	269.7	139.6
AP-7	124	0	121	134.5	51	11.8	12.08	9	19	314	90	9.1	6.2
AP-8	--	14	9	--	6	--	--	--	--	21	7	1.8	1.6

	RESDUE DISS-105 C MG/L	RESDUE VOL FLT MG/L	NO3-N TOTAL MG/L	WATER TEMP CENT	PH SU
AP-1	755	386	83.4	14.5	6.6
AP-2	463	131	67.4	16.0	6.7
AP-3	240	49	9.8	17.4	7.1
AP-4	5906	954	556.0	--	5.8
AP-5	769	100	9.2	23.1	11.9
AP-6	769	100	1.0	46.9	11.4
AP-7	299	83	6.3	20.2	8.8
AP-8	21	9	1.8	--	5.8

****

STATION	FLOW RATE GPM	T ACDITY CACO3 LB/D	T ALK CACO3 LB/D	BOD 5 DAY LB/D	COD HI LEV LB/D	T ORG C C LB/D	PHOS-T P-WET LB/D	RESDUE V NFLT LB/D	RESDUE T NFLT LB/D	RESDUE TOTAL LB/D	RESDUE T VOL LB/D	T KJEL N LB/D	NH3-N TOTAL LB/D
AP-1	1272	549	351	184.6	800	206.9	38.71	190	260	12089	6362.93	1326.22	1305
AP-4	120	152	127	146.7	823	266.2	7.22	38	91	9698	6362.93	1326.22	1305
AP-5	88	0	2094	62.7	126	52.5	1.03	18	52	3592	332.59	36.54	33
AP-6	225	0	4435	--	5476	1823.2	0.12	--	37	2030	265.36	734.78	380
AP-7	124	0	179	233.4	77	17.8	17.92	12	22	466	131.94	14.09	9

	RESDUE DISS-105 C LB/D	RESDUE VOL FLT LB/D	NO3-N TOTAL LB/D
AP-1	11829	6173.0	1280.32
AP-4	9606	1307.6	845.80
AP-5	3540	315.0	8.23
AP-6	1993	265.4	2.70
AP-7	444	122.6	9.22

produced comparable results between the two laboratories and also compared very closely with company results on the original survey. EPA and company sample collection and laboratory procedures were thoroughly reviewed and no explanation for the wide variation could be determined. Since the original company results agreed very closely with both EPA and company results from a sample collected later at each of the sampling locations, it was decided to use company results for the nitrogen series.

COMPARISON OF 1969 AND 1972 DATA (PLANT EFFLUENT)

The 1969 Federal report identified Air Products and Chemicals, Inc. (formerly Escambia Chemicals) as a major contributor of nitrogen and phosphorus wastes discharged into Escambia Bay. A comparative tabulation of data is shown below:

	Flow mgd	BOD_5 lbs/day	TOC lbs/day	TKN-N lbs/day	NH_3-N lbs/day	NO_2-NO_3-N lbs/day	Total N lbs/day	Total Phos-P lbs/day
1969	5.2	290	194	3,270	2,280	2,380	5,650	320
		12	13.5	86.5	85.0	83.5	170	2.5
1972	1.84	185	207	1,327	1,306	1,281	2,608	39
Diff	-3.36	-105	+13	-1,943	- 974	-1,099	-3,042	-281

Biochemical Oxygen Demand

Considerable variation (2.0 to 23 mg/l) was noted in the BOD_5 discharged into Escambia Bay. The final daily discharge contained 185 pounds of BOD_5 — 168 pounds greater than the conference recommended limit. At the present rate of discharge (1.84 MGD), the BOD_5 effluent concentration would have to be reduced to 1.1 mg/l to meet the 17 pounds per day limit recommended. A seven-day average effluent BOD_5 concentration of 10 to 15 mg/l (155 to 230 pounds per day), with a maximum daily fluctuation of 125 percent, is a much more reasonable, achievable limit.

Except for the amines plant discharge, BOD_5 concentrations generated by plant processes are rather minor. Concentrations of BOD_5 in excess of 800 mg/l were noted in the amines plant discharge into George Pond, the percolation pond. The discharge from Fox Trot Pond, which receives the PVC plant waste was the largest single direct contributor of BOD_5 (233 lbs/day) to the pond system. The urea and ammonia nitrate operation (Station AP-4) was the second largest contributor with an average of 147 pounds of BOD_5 discharged daily into Echo Pond.

Phosphorus

Significant reductions have been achieved in phosphorus removal (88 percent). The total phosphorus load has been reduced from 320 pounds per day to 39 pounds per day. Reductions have resulted primarily from closing the NPK operation. Pretreatment of the PVC waste with magnesium oxide before discharge to Fox Trot Pond also reduced the effluent phosphorus load. At the time of the recent study, the average phosphorus load from all plant processes was 26 pounds per day.

Nitrogen

Nitrogen reductions for the same period have been less dramatic than phosphorus reductions. A complicating factor in comparing the two sets of data is the waste being retained in Echo Pond. Since this pond was recently (November 1971) placed into operation and is not yet full, the plant effluent measured at Station AP-1 did not reflect this waste. Waste from the two ponds and the collection ditch which receives waste from the urea and ammonium nitrate operations plus groundwater seepage is diverted to Echo Pond. During the survey, the flow into Echo Pond averaged 0.173 MGD with the following loads in pounds per day: BOD - 138; TOC - 266; TKN - 295; NH_3 - 359; NO_3-N - 1,154, and total phosphorus - 7.2. It is impossible to accurately predict the effect of the Echo Pond discharge on the total plant effluent. However, for comparison purposes, it is assumed that 60 percent of the nitrogen entering Echo Pond will be removed, resulting in an adjusted plant effluent of 3,214 pounds per day or a 43 percent reduction from the 1969 load. This reduction is attributed to closing of the NPK and methanol plant, additional source controls, and treatment in the biological pond system.

Metals

Analyses were made for specific metals by atomic adsorption on samples collected from Stations AP-1, AP-4, and AP-8. In addition, a metals scan was performed by the spark source mass spectrometer on samples from Stations AP-1 and AP-4. The plant effluent (Station AP-1) contained 1.2 pounds per day of chromium, 1.5 pounds per day of zinc, and 3 pounds per day of manganese as well as many other metals in low concentrations. The contribution of metals from Air Products and Chemicals, Inc., to Escambia Bay is very small.

Organic Compounds

An organic scan was performed on samples collected from Stations

AP-1, AP-4, and AP-8 (Table III). Results indicated low concentrations (.03 mg/1) of organic compounds from the DNT operation in the plant effluent.

Table III

ORGANIC COMPOUND DATA

Station No.	Compound	Concentration (mg/1)
AP-1	0-Nitrotoluene	0.012
	2,6-Dinitrotoluene	0.02
	15 other unidentified compounds were observed in the 0.01-1.0 mg/1 range.	
AP-4	0-Nitrotoluene	7.8
	P-Nitrotoluene	8.8
	2,4-Dinitrotoluene	190
	2,6-Dinitrotoluene	150
	3,4-Dinitrotoluene	40
	10 other organic compounds were also detected in the 0.01-5.0 mg/1 range.	
AP-8	No compounds greater than 0.01 mg/1 detected.	

Concentrations of organic compounds in the plant effluent were low; however, the waste from Station AP-4 is presently being retained on site in Echo Pond. When this pond starts overflowing later this year, an increase in the concentration of organics from the DNT operation may be observed. Although fish toxicity for dinitrotoluene is unknown, concentrations of 0-Nitrotoluene in the range of 18 to 40 mg/1 were observed to be lethal for minnows.(7)

Nitro compounds are subject to biochemical oxidation in dilute concentrations; however, higher concentrations retard biological growth.

Trinitrotoluene (TNT) has a deleterious effect at concentrations greater than 1.0 mg/l.(5) The biological toxicity limits for nitrotoluene and dinitrotoluene are not known, but it is likely that toxicity will occur at relatively low concentrations. This may severely limit the treatability of this waste in conventional biological ponds requiring much greater retention times.

Groundwater

Groundwater seepage was a major factor in the total nitrogen load discharged from the plant. Studies performed by the company have shown that nitrogen concentrations in the upper zones of the groundwater in Area "B" ranged up to 1,000 mg/l of total nitrogen. This situation is caused by poor waste control practices in the past. Source controls, waste treatment, better materials handling, and awareness of groundwater contamination problems by company personnel should greatly reduce this problem in the future. The U. S. Geological Survey reports that the average rate of movement of groundwater in the Pensacola area is approximately 100 feet per year. (8) The distance from Area "B" to the upper dikes of the ponds is approximately 1,000 feet, and the approximate distance to the final pond effluent is 3,000 feet. It is theorized that water in the upper ground water zone should move considerably faster than 100 feet per year. Contributions of nitrogen from the ground water will likely be present for several years.

The groundwater problem becomes apparent when a flow or materials balance is attempted on the treatment system. Data, reported by the company to the FDPC, tabulated in Table IV and plotted in Figure 3, illustrate this problem. These data show approximately a 60 percent increase in flow and 240 percent increase in total nitrogen through the three pond system. Results of the recent EPA survey indicated a 28 percent increase in flow (1.3 vs 1.83 mgd) and a 4,000 percent increase in nitrogen (66 vs 2,603 pounds per day of total nitrogen) (Figure 4). These figures do not include the 694 pounds per day discharged into the percolation pond or the 1,515 pounds per day retained in Echo Pond. The 1 MGD of once-through cooling water from the DNT operation was not sampled; however, company spokesman stated that no additives or contaminants were added through the process.

DNT, Urea, and Ammonia Nitrate Waste

During the survey, the pump in the interceptor ditch ran continuously while the pump in Delta Pond ran intermittently. The two composite samples collected at Station AP-4 on March 9-10 and 11-12 represent only waste from the ditch since the Delta Pond pump did not operate during this period. Data indicate a considerable difference in the character of the two wastes (Appendix C). Waste from Delta Pond, which receives the DNT waste, is a concentrated dark brown liquid containing approximately 15,000 mg/l of total residue and 1,500 mg/l of COD. Most of the nitrogen discharged from this operation is in the form of nitrate nitrogen with concentrations of about 1,000 mg/l. The high solids concentration is due primarily to the soda ash wash operation used to purify the dinitrotoluene and to caustic neutralization of the wastes. A 380 mg/l dinitrotoluene concentration was observed in the combined flow at Station AP-4.

TABLE IV

MONTHLY SUMMARY OF AIR PRODUCTS & CHEMICALS, INC. MONTORING DATA
JUNE 1970 THROUGH FEBRUARY 1972

	(1970) JUNE		JULY		AUGUST		SEPTEMBER		OCTOBER		NOVEMBER		DECEMBER	
	INF	EFF	INF	EFF	INF	EFF	INF	EFF	INF	EFF	INF	ENF	INF	ENF
FLOW *	4.13	6.39	3.55	5.97	4.07	6.87	3.82	6.07	4.50	7.94	3.98	7.68	3.49	6.98
COD	7763	4357	3714	2563	8113	2422	9553	1944	11843	3847	14511	4486	5107	3721
NITROGEN:														
AMMONIA	841	3755	571	2886	583	3063	487	2406	639	4963	576	5003	540	4465
ORGANIC	286	894	204	329	340	318	456	273	253	362	508	167	224	374
NITRATE	1658	3304	465	2329	532	2764	513	1985	964	3561	238	3509	546	3682
NITROGEN TOTAL	2735	7909	1232	5490	1399	6064	1457	4665	1858	8886	1323	8684	1611	8521
PHOPHORUS TOTAL	134	676	50	478	113	581	124	492	343	581	95	409	65	957
POTASSIUM	160	992	81	466	71	609	72	485	75	678	36	693	36	408

	(1971) JANUARY		FEBRUARY		MARCH		APRIL		MAY		JUNE		JULY	
	INF	EFF	INF	EFF	INF	EFF	INF	EFF	INF	EFF	INF	ENF	INF	ENF
FLOW *	3.73	5.26	3.87	5.74	3.03	4.42	2.28	3.69	2.27	3.77	2.32	3.78	2.24	3.91
COD	6683	3960	7508	4586	7471	2341	7811	3175	7992	2823	18336	2649	5529	2184
NITROGEN:														
AMMONIA	478	4314	396	3960	894	4066	1936	3393	1463	3039	247	1742	287	1691
ORGANIC	317	243	153	208	283	217	466	319	109	233	213	282	168	343
NITRATE	982	4139	811	4905	1380	4648	1203	2981	2415	2744	1128	1094	871	961
NITROGEN TOTAL	1797	8597	1360	8817	2558	9010	3604	6687	4649	5893	1588	3109	1323	3005
PHOPHORUS TOTAL	128	604	112	348	55	402	249	359	290	441	8	185	36	168
POTASSIUM	31	833	42	366	174	733	122	537	167	404	19	195	27	205

	AUGUST		SEPTEMBER		OCTOBER		NOVEMBER		DECEMBER		(1972) JANUARY		FEBRUARY	
	INF	EFF	INF	EFF	INF	EFF	INF	EFF	INF	EFF	INF	ENF	INF	ENF
FLOW *	2.39	4.05	2.29	5.06	--	2.43	--	1.58	--	1.75	--	1.89	--	2.05
COD	8191	1956	7033	2177	--	1144	--	967	--	938	--	903	--	858
NITROGEN:														
AMMONIA	684	1956	877	2352	--	1248	--	1174	--	1209	--	1493	--	1469
ORGANIC	146	221	318	297	--	90	--	46	--	45	--	64	--	21
NITRATE	833	1659	777	2408	--	1290	--	1161	--	1231	--	1211	--	1272
NITROGEN TOTAL	1521	3836	1959	5049	--	2628	--	2362	--	2484	--	2764	--	2759
PHOPHORUS TOTAL	23	138	--	--	--	--	--	--	--	--	--	--	--	--
POTASSIUM	59	246	--	--	--	--	--	--	--	--	--	--	--	--

* FLOW REPORTED AS MGD; ALL OTHER VALUES ARE LBS/DAY

(DATA THROUGH AUGUST 1971 WAS TAKEN FROM COMPANY REPORT DATED OCTOBER 1, 1971 TO THE FDPC. LATER DATA WAS OBTAINED FROM COMPANY PREPARED REPORTS TO THE FDPC.)

FIGURE 3
TOTAL NITROGEN (LBS / DAY) LOADS
AIR PRODUCTS AND CHEMICALS INC.

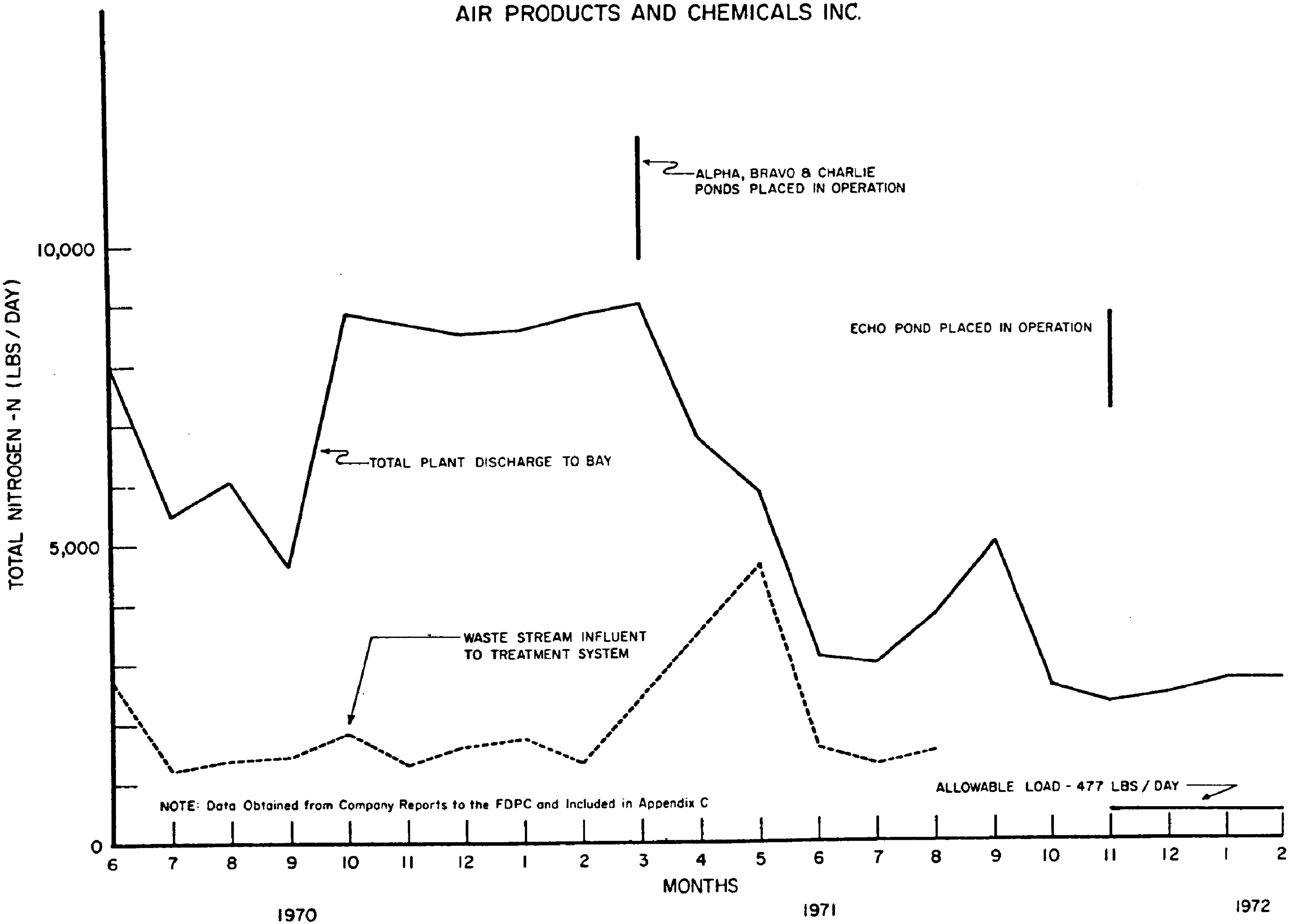
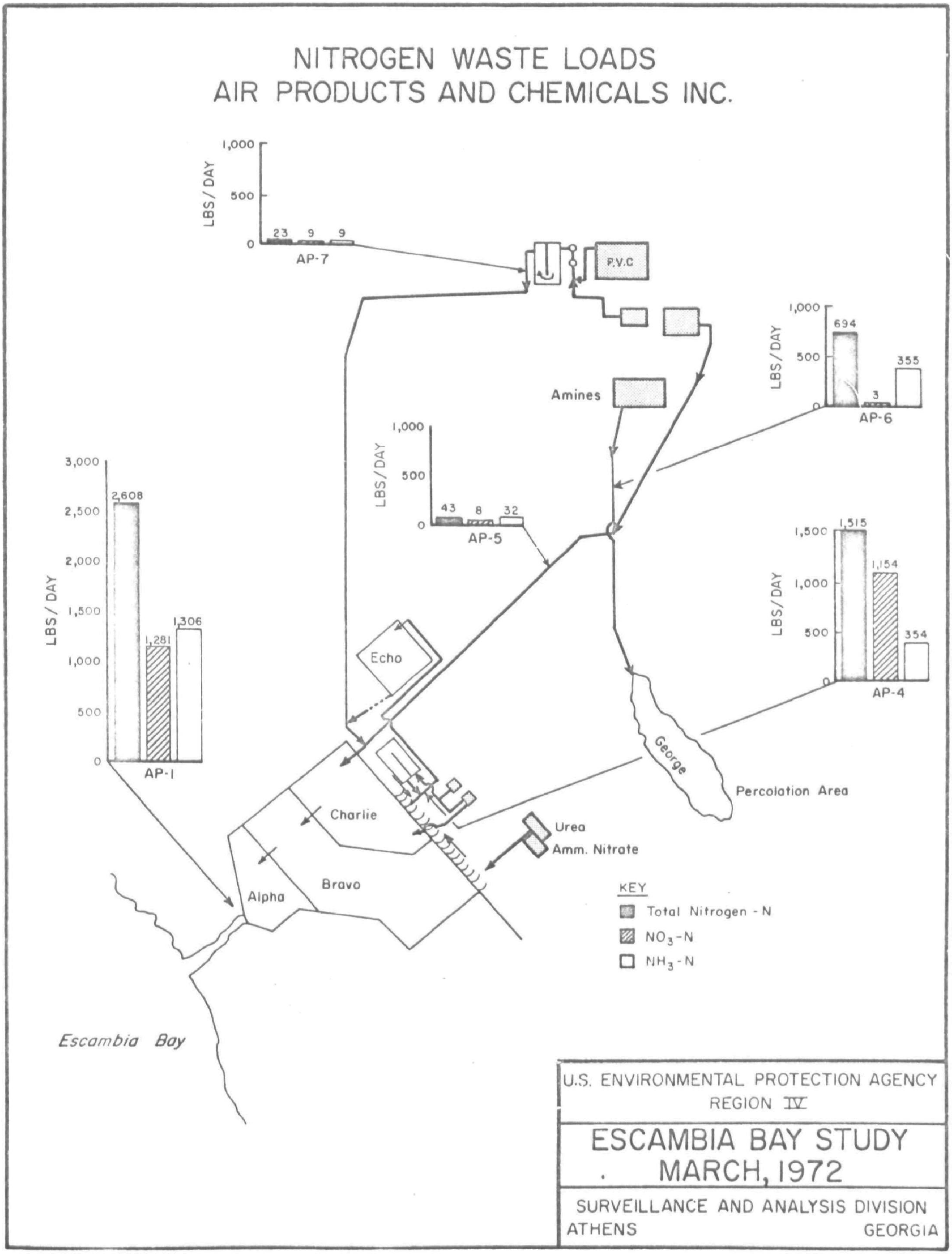


FIGURE 4


NITROGEN WASTE LOADS
AIR PRODUCTS AND CHEMICALS INC.
LBS / DAY
1,000
500
0
23
9
9
AP-7
P.V.C
1,000
694
500
355
3
0
AP-6
Amines
1,000
500
0
43
8
32
AP-5
3,000
2,608
2,500
2,000
1,500
1,281
1,306
1,000
500
0
AP-1
1,515
1,500
1,154
1,000
500
354
0
AP-4
Echo
George
Percolation Area
Charlie
Urea
Amm. Nitrate
Alpha
Bravo
KEY
Total Nitrogen - N
NO3-N
NH3-N
Escambia Bay
U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION IV
ESCAMBIA BAY STUDY
MARCH, 1972
SURVEILLANCE AND ANALYSIS DIVISION
ATHENS GEORGIA

Considering the time that each pump was in operation, a concentration of 700 to 800 mg/l of dinitrotoluene in Delta Pond was estimated. The interceptor ditch receives approximately 500 gallons per hour of condensate waste and barometric condenser condensate from the urea and ammonia nitrate operations with the remainder of the flow being surface runoff and ground water infiltration. This waste had an approximate ammonia and nitrate concentration of 350 mg/l and 385 mg/l, respectively, with an average flow of 84 gpm. Since the combined flow from Delta Pond and the interceptor ditch represents 67 percent of the total nitrogen load from process discharges, the company has isolated this waste for additional treatment in Echo Pond.

Ammonia Waste

The ammonia waste flows through the concrete lined East Ditch to Charlie Pond (Station AP-5). This is a highly alkaline waste (2,550 mg/l as $CaCO_3$) with an average pH of 11.9. The average total nitrogen concentration is relatively low at 26 mg/l with 23 mg/l as ammonia. This waste stream contains 2 percent of the total nitrogen waste loading from process operations.

Amines Waste

Waste from the amines plant is clear with an average temperature of 47°C and pH of 11.4 (Station AP-6). Average concentrations are: alkalinity - 1,664 mg/l as $CaCO_3$; BOD_5 - greater than 808 mg/l (company reported one value of 2,463 mg/l); COD - 2,368 mg/l; TOC - 750 mg/l;

and total nitrogen as N - 694 mg/l. This source accounts for 30 percent of the total nitrogen discharged from plant processes and is probably a large contributor of nitrogen seepage into the lower pond system. The 1969 report by Black, Crow and Eidsness, Inc., states that "The data tend to indicate that the Amines plants effluent, discharged into the percolation area with an estimated nitrogen loading of 872 pounds per day, appears at the outfall."

PVC and Nitric Acid Waste

The discharge from Fox Trot Pond (Station AP-7) is slightly alkaline with an average pH of 8.8. The waste is well settled and presents no significant nitrogen load to the lower pond system. Nitrogen contributions from this source represent one percent of the total from process operations. This stream is the only direct process discharge of phosphorus to Charlie Pond. The load is 18 pounds per day.

Well Water

Air Products is presently using three 150-foot deep wells to supply the plant's water requirements. One well, located in Plant Area "B", supplies the cooling water requirements of this area. The other two wells are located east of the Area "A" (Figure 2). Two grab samples were collected from these wells during the survey (Station AP-8). Because of discrepancies in the nitrogen data, two more grab samples were collected in April. All Parameters analyzed, other than nitrogen, were in good agreement with well samples taken from the same general area. Nitrogen concentrations were higher than concentrations in samples taken outside the plant area.

Analysis of well water samples taken at American Cyanamid's Santa Rosa Plant, located only a few miles away, showed nitrogen concentrations less than 1 mg/l of total nitrogen. By contrast, these results indicate that nitrogen waste from the Air Products Plant have spread through a relatively large area of the ground water (Table V).

Table V

WELL WATER NITROGEN DATA

	Well No. 1		Well No. 3		Well No. 5	
	4/13	4/14	4/13	4/14	4/13	4/14
TKN (mg/l)	4.2	3.7	1.1	1.0	0.6	0.0
NH_3-N (mg/l)	4.2	3.7	1.1	1.0	0.4	0.0
NO_3-N (mg/l)	0.7	0.6	5.1	3.6	0.2	0.2
Total N (mg/l)	4.9	4.3	6.2	4.6	0.8	0.2

TREATMENT ALTERNATIVES AND SUGGESTIONS

The major objective in waste treatment considerations at Air Products is further reduction in the nitrogen discharge. Nitrogen in the plant effluent originates from two principal sources -- the total discharge from the individual plant processes and contaminated groundwater seepage into the treatment system. During the recent survey, 46 pounds per day of nitrogen were discharged into the three-pond system, and 2,610 pounds were discharged from the ponds. However, process wastes being stored in Echo Pond accounted for the unusually low nitrogen load (46 lbs/day) discharged into the ponds. During the study, water was being released from storage in the three-pond system; however, the major portion of this difference must be attributed to groundwater seepage. It is reasonable to assume that roughly 700 pounds per day comes directly from the percolation pond and the remaining 1,800 pounds per day comes from contaminated groundwater and seepage from the Delta Pond.

Because of geophysical characteristics of the plant area, such as the porous surface sand zone, the shallow water table, and existing problems encountered with contaminated groundwater, all waste transmission and treatment facilities should be as leakproof as possible. The following are suggestions for improving waste handling procedures:

- Eliminate George (percolation) Pond.
- Reline Delta Pond.
- Line future ponds with clay or other impervious materials.

- Use unlined Alpha, Bravo, and Charlie Ponds only for polishing pretreated wastes.
- Transport all waste (especially from Area "A") in water-tight pipes or channels.
- Re-evaluate in-plant handling procedures to prevent any nitrogen spillage of intermediate or finished products.

TREATMENT METHODS

The following alternative methods of waste treatment were considered:

- Ion exchange
- Spray irrigation
- Deep well disposal
- Ammonia stripping
- Biological ponds

Ion Exchange

Initially, the ion exchange process designed by Chemical Separations Corporation for Farmers Chemical Association, Inc., in Chattanooga, Tennessee, appeared to provide a possible solution for the problems at Air Products. After discussions with Chemical Separations and Air Products personnel, it was learned that methods have been studied to adapt this system. However, the high concentrations of salts, other than ammonium nitrate, would produce a waste stream unusable for product recovery. Disposal of this unusable waste stream (estimated by Air Products to be 28,800 gpd) would present a problem of the same magnitude as disposing of the original waste.

Spray Irrigation

Spray irrigation provides some possibilities; however, it needs additional study. It is doubtful that this method could be used for disposal of the total plant effluent, including ground water seepage to the ponds, because of the large volume (2 to 4 MGD); however, it could possibly be used for ultimate disposal of the process waste.

Deep Well Disposal

The company has determined that deep well disposal is not suitable for disposal of the total effluent because of the large waste volume and the 90 percent FDPC reduction requirement. This method could, however, be used for disposal after treatment of the process waste.

Ammonia Stripping

Stripping appears feasible for removal of the 355 pounds per day of ammonia in the amines plant waste. The existing high pH and temperature make this waste suitable without pre-conditioning for stripping. Steam stripping or distillation with ammonia recovery may be necessary in order to prevent air pollution problems. Any of these methods should remove virtually all of the ammonia which accounts for roughly 50 percent of the total nitrogen in this stream. Ammonia stripping of the other large source of ammonia waste (DNT and the interceptor ditch) would require pre-conditioning since the pH is 5.8. If ammonia stripping is used, it would probably be advantageous to separate these two wastes streams since the major portion of the ammonia comes from the interceptor ditch. The pH of the waste in the interceptor ditch averaged 4.4.

Biological Treatment

Air Products has placed in operation a system of biological ponds for treatment of the total plant waste. Because of the problems encountered with previously-discussed treatment methods, biological treatment combined with ammonia stripping appears to be the more promising. For example, Dr. William Oswald has stated that "Input-output balances show disappearance of up to 80 percent of the total input nitrogen in a single faculative pond treating domestic waste."(9) Laboratory results from the report prepared by Associated Water and Air Resources Engineers, Inc. for Farmers Chemicals Association, Inc., indicated that 80 to 85 percent of the nitrate nitrogen could be removed in an anaerobic unit in 10 to 35 days providing the proper nutrient balance was maintained for bacterial growth.(10)

The organic nitro compounds from the DNT operation will probably retard biological growth in Echo Pond. If given sufficient time, these compounds will break down; however, a sizable increase in ponding capacity may be required.

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2. Proceedings of the Second Session of the Conference in the Matter of Pollution of Interstate Waters of the Escambia River Basin (Alabama-Florida) and the Interstate Portions of the Escambia Basin Within the State of Florida, EPA, Pensacola, Florida, February 23-24, 1971.

3. Case No. IW-226-69, Order No. 356, "Orders for Corrective Action," the Florida Department of Pollution Control, December 15, 1971.

4. Preliminary Report to Escambia Chemical Corporation on Wastewater Treatment, Black, Crow and Eidsness, Inc., Engineers, Gainesville, Florida, October 1969.

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7. Water Quality Criteria, California Water Resources Control Board, Publ. 3-A, 2nd Ed., 1963.

8. Water Resources of Escambia and Santa Rosa Counties, Florida, U. S. Geological Survey, Report of Investigation No. 40, Tallahassee, Florida, 1965.

9. Oswald, William J., "Status of Oxidation Pond Processes." Presented at the conference, Southeast Water Laboratory, Athens, Georgia, "Advances in Treatment of Domestic Wastes." October 18, 1972.

10. Laboratory Investigations into the Removal and Control of High Concentrations of Nitrogenous Compounds, Associated Water and Air Resources Engineers, Inc., Nashville, Tennessee, May 1970.

APPENDICES

APPENDIX A

ACKNOWLEDGMENT & PROJECT PERSONNEL

APPENDIX A

ACKNOWLEDGMENT

Appreciation is expressed to Mr. Phil Doherty and his staff at the FDPC Gulf Breeze Laboratory for their able assistance in conducting this study by providing laboratory space and field personnel for assisting in sample collection. Company reports provided by the FDPC were very useful in obtaining background information on previous pollution abatement activities. Company assistance and cooperation were outstanding. Mr. Roy Duggan and his crew made available the Company's automatic sampling and flow measuring equipment for this survey.

EPA FIELD PERSONNEL

Rod Davis - Field Chemist

Charles Sweatt - Sanitary Engineer

Tom Bennett - Chemist

Pat Lawless - Chemist

FDPC FIELD PERSONNEL

Ron Breeding - Technician

APPENDIX B

ANALYTICAL METHODOLOGY

ANALYTICAL METHODS

All chemical analysis will be done in accordance with those methods listed in Methods for Chemical Analysis of Water and Wastes 1971.

1. Acidity - Listed on page 5, uses a NaOH titration to an end-point of pH 8.3. Results are reported as mg/l $CaCO_3$.
2. Alkalinity - Listed in Standard Methods for the Examination of Water and Wastewater, 13th Edition, p. 52, Method 102 (1971).
3. Biochemical Oxygen Demand (BOD_5) - Standard Methods for the Examination of Water and Wastewater, 13th Edition, p. 489, Method 219 (1971).
4. Chemical Oxygen Demand - Standard Methods for the Examination of Water and Wastewater, 13th Edition, p. 495, Method 220 (1971).
5. Cyanide - EPA Methods for Chemical Analysis of Water and Wastes 1971, p. 42.
6. Metals - EPA Methods for Chemical Analysis of Water and Wastes 1971, p. 83.
7. Nitrogen-Ammonia - (Automated Method) EPA Methods for Chemical Analysis of Water and Wastes 1971, p. 141.
8. Nitrogen - Total Kjeldahl - (Automated Phenolate Method) EPA Methods of Chemical Analysis of Water and Wastes 1971, p. 157.
9. Nitrogen, Nitrate-Nitrite - (Automated Cadmium Reduction Method) EPA Methods for Chemical Analysis of Water and Wastes 1971, p. 175.
10. Oil and Grease - Hexane Extraction - EPA Methods of Chemical Analysis of Chemical Analysis of Water and Wastes 1971, p. 217.
11. Total Organic Carbon - Carbonaceous Analyzer - EPA Methods of Chemical Analysis of Water and Wastes 1971, p. 221.

12. Phenolics - (Automated 4-AAP Method with Distillation) Standard Methods for the Examination of Water and Wastewater, 13th Edition, pp. 501-510, Method 222 through 222E (1971), Modified for Automation.

13. Phosphorus - (Automated Single Reagent Method) EPA Methods of Chemical Analysis of Water and Wastes 1971, p. 246.

14. Solids, Filterable (Dissolved) - EPA Methods of Chemical Analysis of Water and Wastes 1971, p. 275.

15. Solids, Total - EPA Methods of Chemical Analysis of Water and Wastes 1971, p. 280.

16. Solids, Non-Filterable (Suspended) - By difference between total and filterable solids.

17. Solids, Volatile - EPA Methods for Chemical Analysis of Water and Wastes 1971, p. 282.

PROCEDURE FOR ORGANIC ANALYSES

Samples AP-1, AP-4, and AP-8 were approximately neutral pH when received. They were extracted with 15% methylene chloride in hexane, made strongly basic with KOH and re-extracted with 15% methylene chloride in hexane, acidified with sulfuric acid and extracted for the third time with methylene chloride. The samples were dried by passing through glass wool and then concentrated to 1 ml in a Kuderna-Danish concentrator. The acid extract was esterified using diazomethane. All extracts were injected into a gas chromatograph equipped with a flame ionization detector. The column was glass 6'x2.5 mm I.D. packed with 3% SE-30 on Chromosorb W, HP. The oven was programmed from 75^{o}-240^{o} @ 10^{o}/min. We attempted to identify any peaks detected using the Finnigan GC/MS Model 1015 system. The compounds listed were confirmed by injecting a known standard into the GC/MS. Quantitation was done on the gas chromatograph.

All samples were extracted in duplicate and the concentrations found represent the average of the duplicates. Concentrations found should be considered minimum concentrations as the percent recovery of each compound from water is unknown.

METHODS OF CHEMICAL ANALYSES

Water and Wastewater

PARAMETER	METHOD	REFERENCE	MODIFICATION
Acidity	Volumetric, NaOH Titration	1	Potentiometric Endpoint
Alkalinity	" H_2SO_4 Titration	1	" "
Ammonia	Automated Phenolate Method	2	None
Biochemical Oxygen Demand	Winkler D.O., 5-day	1,2	Azide Modification Formula "C" Dilution Water w/Sewage Sed.
Chemical Oxygen Demand	Acid-Dichromate Oxidation	2	None
Chromium, Total	Atomic Absorption	1,2	HCl-HNO_3 Digestion
Cobalt	Atomic Absorption	1,2	HCl-HNO_3 Digestion
Copper	Atomic Absorption	1,2	HCl-HNO_3 Digestion
Dissolved Solids	Difference Between Total and Suspended Solids	-	--
Cyanides	Pyridine-Pyrazolone	1,2,4	Distillation Cleanup
Manganese	Atomic Absorption	1,2	HCl-HNO_3 Digestion
Nitrate-Nitrite	Copper-Cadmium Reduction, Automated	2	None
Oil and Grease	Solvent Extraction	1	None
pH	Electrometric	1	None
Phenols	4-Aminoantipyrine	1	Distillation, Automated Colorimetric Analysis

APPENDIX B

(Con't)

PARAMETER	METHOD	REFERENCE	MODIFICATION
Phosphorus	Ascorbic Acid Method	2	Automated, Manual Digestion
Suspended Solids	Gravimetric	1	None
Thiocyanates	Ferric Nitrate Complex	3	-
Total Kjeldahl Nitrogen	Automated Phenolate Method	2	None
Total Organic Carbon	Carbon Analyzer	2	None
Total Solids	Gravimetric, $105^{\circ}C$	1	None
Turbidity	Hellige Turbidimeter	2	None
Volatile Suspended Solids	Gravimetric, $550^{\circ}C$	1	None
Volatile Total Solids	Gravimetric, $550^{\circ}C$	1	None
Zinc	Atomic Absorption	1,2	$HCl-HNO_3$ Digestion
Titanium	Atomic Absorption	1,2	$HCl-HNO_3$ Digestion
Vanadium	Atomic Absorption	1,2	$HCl-HNO_3$ Digestion

References

1. Standard Methods for Examination of Water and Wastewater, 13th Edition, 1971.
2. EPA Manual of Methods for Chemical Analyses of Water and Wastes, 1971.
3. Colorimetric Methods of Analysis, Vol. 11A, 1959, Snell and Snell.
4. ASTM Book of Standards, Part 23, 1971.

APPENDIX C

WASTE SAMPLING DATA

STORET RETRIEVAL DATE 72/06/10

641050 AP-01 F-113-46

AIR PRODUCTS AND CHEMICALS INC.
ESCAMBIA RIVER STUDY

ESCAMBIA R. AIR PRODUCTS & CHEM.
12 FLORIDA
SOUTHEAST
ESCAMBIA RIVER
1113S000 2444220

DATE FROM TO	TIME OF DAY	DEPTH FEET	00058 FLOW RATE GPM	00435 T ACDITY CACO3 MG/L	00410 T ALK CACO3 MG/L	00310 BOD 5 DAY MG/L	00340 COD HI LEVEL MG/L	00680 T ORG C C MG/L	00665 PHOS-T P-WET MG/L	00535 RESIDUE VOL NFLT MG/L	00530 RESIDUE TOT NFLT MG/L
72/03/07	09 27										
CP(T)-G			1382.00	28	32	9.1	29	18.0	2.00	6	13
72/03/08	08 00										
72/03/08	09 30										
CP(T)-G			1382.00	38	22	11.0	48	11.0	2.00	6	6
72/03/09	09 30										
72/03/09	09 35										
CP(T)-G			1417.00	39	21	23.0	78	14.0	2.40	28	36
72/03/10	09 30										
72/03/10	09 35										
CP(T)-G			1208.00	46	20	12.0	55	12.0	2.40	10	15
72/03/11	09 15										
72/03/11	09 45										
CP(T)-G			972.00	27	18	2.0	51	12.0	4.40	11	13
72/03/12	09 15										

DATE FROM TO	TIME OF DAY	DEPTH FEET	00500 RESIDUE TOTAL MG/L	00505 RESIDUE TOT VOL MG/L	00625 TOT KJEL N MG/L	00610 NH3-N TOTAL MG/L	00515 RESIDUE DISS-105 C MG/L	00520 RESIDUE VOL FLT MG/L	00620 NO3-N TOTAL MG/L	00010 WATER TEMP CENT	00403 LAB PH SU
72/03/07	09 27									12.5	
	14 45									16.0	
72/03/07	09 27										
CP(T)-G			689	320	94.800	93.700	676	314	87.00		6.8
72/03/08	08 00										
	09 30									16.0	
72/03/09	09 30									14.0	
72/03/08	09 30										
CP(T)-G			619	258	85.500	85.400	613	252	88.00		6.5
72/03/09	09 30										
72/03/10	09 30									14.0	
72/03/09	09 35										
CP(T)-G			1380	961	87.600	84.300	1344	933	80.00		6.7
72/03/10	09 30										
72/03/11	09 15									14.0	
72/03/10	09 35										
CP(T)-G			541	202	84.300	82.300	526	192	85.80		6.5
72/03/11	09 15										
72/03/12	09 15									15.0	
72/03/11	09 45										
CP(T)-G			631	250	78.900	78.700	618	239	76.00		6.5
72/03/12	09 15										

STORET RETRIEVAL DATE 72/06/10

641052 AP-02 F-113-47

AIR PRODUCTS AND CHEMICALS INC.
ESCAMBIA RIVER STUDY

ESCAMBIA R. AIR PRODUCTS & CHEM.
12 FLORIDA
SOUTHEAST
ESCAMBIA RIVER
1113S000 2444220

DATE FROM TO	TIME OF DAY	DEPTH FEET	00058 FLOW RATE GPM	00435 T ACDITY CACO3 MG/L	00410 T ALK CACO3 MG/L	00310 BOD 5 DAY MG/L	00340 COD HI LEVEL MG/L	00680 T ORG C C MG/L	00665 PHOS-T P-WET MG/L	00535 RESIDUE VOL NFLT MG/L	00530 RESIDUE TOT NFLT MG/L
72/03/07	09 50										
CP(T)-G				22	35	72.0K	44	14.0	2.13	5K	8
72/03/08	08 05										
72/03/08	09 45										
CP(T)-G				35	31	8.0	38	13.0	2.00	5K	5K
72/03/09	09 45										
72/03/09	09 45										
CP(T)-G				36	28	41.0	71	10.0	2.72	5K	6
72/03/10	09 45										
72/03/10	09 50										
CP(T)-G				38	30	64.0K	50	11.0	2.50	8	11
72/03/11	09 50										
72/03/11	10 00										
CP(T)-G				31	27	68.0K	41	12.0	9.10	9	10
72/03/12	10 00										

DATE FROM TO	TIME OF DAY	DEPTH FEET	00500 RESIDUE TOTAL MG/L	00505 RESIDUE TOT VOL MG/L	00625 TOT KJEL N MG/L	00610 NH3-N TOTAL MG/L	00515 RESIDUE DISS-105 C MG/L	00520 RESIDUE VOL FLT MG/L	00620 NO3-N TOTAL MG/L	00010 WATER TEMP CENT	00403 LAB PH SU
72/03/07	09 50									15.5	
	14 55									17.5	
72/03/08	08 05									16.5	
72/03/07	09 50										
CP(T)-G			429	113	66.000	59.300	421	113	57.00		6.9
72/03/08	08 05										
72/03/09	09 45									14.5	
72/03/08	09 45										
CP(T)-G			578	214	70.700	69.900	578	214	74.00		6.6
72/03/09	09 45										
72/03/10	09 45									16.0	
72/03/09	09 45										
CP(T)-G			463	105	83.700	83.200	457	105	79.40		6.8
72/03/10	09 45										
72/03/11	09 50									16.0	
72/03/10	09 50										
CP(T)-G			404	121	60.400	58.800	393	113	64.00		6.6
72/03/11	09 50										
72/03/12	10 00									16.0	
72/03/11	10 00										
CP(T)-G			478	120	58.500	58.200	468	111	62.60		6.6
72/03/12	10 00										

STORET RETRIEVAL DATE 72/06/10

641054 AP-03 F-113-48

AIR PRODUCTS AND CHEMICALS INC.
ESCAMBIA RIVER STUDY

ESCAMBIA R. AIR PRODUCTS & CHEM.
12 FLORIDA
SOUTHEAST
ESCAMBIA RIVER
1113S000 2444220

DATE FROM TO	TIME OF DAY	DEPTH FEET	00058 FLOW RATE GPM	00435 T ACDITY CACO3 MG/L	00410 T ALK CACO3 MG/L	00310 BOD 5 DAY MG/L	00340 COD HI LEVEL MG/L	00680 T ORG C C MG/L	00665 PHOS-T P-WET MG/L	00535 RESIDUE VOL NFLT MG/L	00530 RESIDUE TOT NFLT MG/L
72/03/07	10 13										
CP(T)-G				8	52	72.0K	17	6.0	1.85	8	9
72/03/08	10 05										
72/03/08	10 08										
CP(T)-G				9	46	68.0K	24	6.0	1.70	5	6
72/03/09	09 15										
72/03/09	10 00										
CP(T)-G				12	41	41.0	26	8.0	2.30	5K	6
72/03/10	10 00										
72/03/10	10 05										
CP(T)-G				12	40	65.0K	35	8.0	1.89	7	8
72/03/11	10 05										
72/03/11	10 15										
CP(T)-G				9	37	68.0K	32	9.0	2.00	11	12
72/03/12	10 15										

DATE FROM TO	TIME OF DAY	DEPTH FEET	00500 RESIDUE TOTAL MG/L	00505 RESIDUE TOT VOL MG/L	00625 TOT KJEL N MG/L	00610 NH3-N TOTAL MG/L	00515 RESIDUE DISS-105 C MG/L	00520 RESIDUE VOL FLT MG/L	00620 NO3-N TOTAL MG/L	00010 WATER TEMP CENT	00403 LAB PH SU
72/03/07	10 13									17.0	
	15 10									18.5	
72/03/08	10 05									18.0	
72/03/07	10 13										
CP(T)-G			252	72	13.900	11.600	241	64	8.60		7.2
72/03/08	10 05										
72/03/08	10 08										
CP(T)-G			260	66	13.600	11.100	254	61	9.70		7.2
72/03/09	09 15										
	09 50									15.5	
72/03/10	10 00									17.0	
72/03/09	10 00										
CP(T)-G			250	36	15.000	11.600	244	36	11.30		7.1
72/03/10	10 00										
72/03/11	10 05									17.5	
72/03/10	10 05										
CP(T)-G			234	60	13.900	10.500	226	53	10.40		6.9
72/03/11	10 05										
72/03/12	10 15									18.5	
72/03/11	10 15										
CP(T)-G			245	44	12.200	10.000	233	33	9.10		7.2
72/03/12	10 15										

STORET RETRIEVAL DATE 72/06/10

641056 AP-04 F-113-49

AIR PRODUCTS AND CHEMICALS INC.
ESCAMBIA RIVER STUDY

ESCAMBIA R. AIR PRODUCTS & CHEM.
12 FLORIDA
SOUTHEAST
ESCAMBIA RIVER
11135000 2444220

DATE FROM TO	TIME OF DAY	DEPTH FEET	00058 FLOW RATE GPM	00435 T ACDITY CACO3 MG/L	00410 T ALK CACO3 MG/L	00310 BOD 5 DAY MG/L	00340 COD HI LEVEL MG/L	00680 T ORG C C MG/L	00665 PHOS-T P-WET MG/L	00535 RESIDUE VOL NFLT MG/L	00530 RESIDUE TOT NFLT MG/L
72/03/07	10 35										
CP(T)-G			162.00	85	157	79.0	878	250.0	4.48	45	127
72/03/08	10 20										
72/03/08	10 25										
CP(T)-G			151.00	77	99	50.0	668	200.0	6.00	13	35
72/03/09	10 10										
72/03/09	10 20										
CP(T)-G			84.00	172	0	200.0	100	31.0	8.60	5K	28
72/03/10	10 15										
72/03/10	10 20										
CP(T)-G			117.00	87	108	100.0	746	300.0	2.90	25	72
72/03/11	10 20										
72/03/11	10 30										
CP(T)-G			84.00	156	0	100.0K	44	28.0	3.70	5	17
72/03/12	10 20										

DATE FROM TO	TIME OF DAY	DEPTH FEET	00500 RESIDUE TOTAL MG/L	00505 RESIDUE TOT VOL MG/L	00625 TOT KJEL N MG/L	00610 NH3-N TOTAL MG/L	00515 RESIDUE DISS-105 C MG/L	00520 RESIDUE VOL FLT MG/L	00620 NO3-N TOTAL MG/L	00010 WATER TEMP CENT	00403 LAB PH SU
72/03/07	10 35										
CP(T)-G			9410	788	138.600	136.400	9283	743	670.00		6.9
72/03/08	10 20										
72/03/08	10 25										
CP(T)-G			8060	872	142.500	142.400	8025	859	645.00		6.7
72/03/09	10 10										
72/03/09	10 20										
CP(T)-G			2010	784	353.300	353.200	1982	784	395.00		4.3
72/03/10	10 15										
72/03/10	10 20										
CP(T)-G			7790	873	170.800	170.700	7718	848	695.00		6.7
72/03/11	10 20										
72/03/11	10 30										
CP(T)-G			2540	1540	350.500	350.400	2523	1535	375.00		4.6
72/03/12	10 20										

STORET RETRIEVAL DATE 72/06/10

641058 AP-05 F-113-50

AIR PRODUCTS AND CHEMICALS INC.
ESCAMBIA RIVER STUDY

ESCAMBIA R. AIR PRODUCTS & CHEM.
12 FLORIDA
SOUTHEAST
ESCAMBIA RIVER
11135000 2444220

DATE FROM TO	TIME OF DAY	DEPTH FEET	00058 FLOW RATE GPM	00435 T ACDITY CACO3 MG/L	00410 T ALK CACO3 MG/L	00310 BOD 5 DAY MG/L	00340 COD HI LEVEL MG/L	00680 T ORG C C MG/L	00665 PHOS-T P-WET MG/L	00535 RESIDUE VOL NFLT MG/L	00530 RESIDUE TOT NFLT MG/L
72/03/07	11 31										
CP(T)-G			114.00	0	1130	55.0	149	32.0	1.27	37	115
72/03/08	11 10										
72/03/08	16 15										
CP(T)-G			147.00	0	1030	11.0	90	20.0	0.84	7	18
72/03/09	10 25										
72/03/09	10 30										
CP(T)-G			84.00	0	3200	138.0	111	84.0	0.86	12	41
72/03/10	10 35										
72/03/10	10 45										
CP(T)-G			58.00	0	2300	24.0	175	60.0	0.63	14	31
72/03/11	10 35										
72/03/11	10 45										
CP(T)-G			37.00	0	5100	70.0K	69	128.0	1.40	7	15
72/03/12	10 45										

DATE FROM TO	TIME OF DAY	DEPTH FEET	00500 RESIDUE TOTAL MG/L	00505 RESIDUE TOT VOL MG/L	00625 TOT KJEL N MG/L	00610 NH3-N TOTAL MG/L	00515 RESIDUE DISS-105 C MG/L	00520 RESIDUE VOL FLT MG/L	00620 NO3-N TOTAL MG/L	00010 WATER TEMP CENT	00403 LAB PH SU
72/03/07	11 31									24.5	
	15 25									25.5	
72/03/08	11 10									20.0	
72/03/07	11 31										
CP(T)-G			2210	185	23.800	23.200	2095	148	5.10		11.8
72/03/08	11 10										
72/03/09	10 25									22.0	
72/03/08	16 15										
CP(T)-G			3300	182	66.000	59.900	3282	175	6.00		11.6
72/03/09	10 25										
72/03/09	10 30										
CP(T)-G			4760	539	17.200	13.300	4719	527	11.00		11.9
72/03/10	10 35										
72/03/11	10 35									24.0	
72/03/10	10 45										
CP(T)-G			2780	244	20.000	17.200	2749	230	7.70		11.9
72/03/11	10 35										
72/03/12	10 45									22.5	
72/03/11	10 45										
CP(T)-G			5310	841	5.000	0.600	5295	834	16.00		12.1
72/03/12	10 45										

STORET RETRIEVAL DATE 72/06/10

641060 AP-06 F-113-S1

AIR PRODUCTS AND CHEMICALS INC.
ESCAMBIA RIVER STUDY

ESCAMBIA R. AIR PRODUCTS & CHEM.
12 FLORIDA
SOUTHEAST
ESCAMBIA RIVER
11135000 2444220

DATE FROM TO	TIME OF DAY	DEPTH FEET	00058 FLOW RATE GPM	00435 T ACDITY CACO3 MG/L	00410 T ALK CACO3 MG/L	00310 BOD 5 DAY MG/L	00340 COD HI LEVEL MG/L	00680 T ORG C C MG/L	00665 PHOS-T P-WET MG/L	00535 RESIDUE VOL NFLT MG/L	00530 RESIDUE TOT NFLT MG/L
72/03/07	11 12										
CP(T)-G			245.00	0	810	830.0L	3210	800.0	0.01	5K	22
72/03/08	11 25										
72/03/08	11 33										
CP(T)-G			240.00	0	1810	810.0L	2570	1000.0	0.03	5K	9
72/03/09	11 20										
72/03/09	11 25										
CP(T)-G			217.00	0	1600	820.0L	1960	600.0	0.01K	5K	10
72/03/10	11 05										
72/03/10	11 10										
CP(T)-G			211.00	0	2200	740.0L	1730	600.0	0.02	5K	15
72/03/11	11 00										
72/03/11	11 10										
CP(T)-G			211.00	0	1900	840.0L	402	312.0	0.13	5K	12
72/03/12	10 50										

DATE FROM TO	TIME OF DAY	DEPTH FEET	00500 RESIDUE TOTAL MG/L	00505 RESIDUE TOT VOL MG/L	00625 TOT KJEL N MG/L	00610 NH3-N TOTAL MG/L	00515 RESIDUE DISS-105 C MG/L	00520 RESIDUE VOL FLT MG/L	00620 NO3-N TOTAL MG/L	00010 WATER TEMP CENT	00403 LAB PH SU
72/03/07	11 12									44.0	
72/03/08	11 25									40.0	
72/03/07	11 12										
CP(T)-G			273	80	255.600	127.500	251	80	1.50		11.2
72/03/08	11 25										
72/03/09	11 20									43.0	
72/03/08	11 33										
CP(T)-G			270	84	356.500	179.100	261	84	1.10		11.0
72/03/09	11 20										
72/03/10	11 05									44.5	
72/03/09	11 25										
CP(T)-G			282	30	369.200	214.600	272	30	0.80		11.1
72/03/10	11 05										
72/03/11	11 00									56.0	
72/03/10	11 10										
CP(T)-G			1430	103	249.500	117.500	1415	103	0.70		11.7
72/03/11	11 00										
72/03/12	10 50									54.0	
72/03/11	11 10										
CP(T)-G			1660	201	117.500	59.300	1648	201	0.80		11.9
72/03/12	10 50										

STORET RETRIEVAL DATE 72/06/10

641062 AP-07 F-113-52

AIR PRODUCTS AND CHEMICALS INC.
ESCAMBIA RIVER STUDY

ESCAMBIA R. AIR PRODUCTS & CHEM.
12 FLORIDA
SOUTHEAST
ESCAMBIA RIVER
11135000 2444220

DATE FROM TO	TIME OF DAY	DEPTH FEET	00058 FLOW RATE GPM	00435 T ACDITY CACO3 MG/L	00410 T ALK CACO3 MG/L	00310 BOD 5 DAY MG/L	00340 COD HI LEVEL MG/L	00680 T ORG C C MG/L	00665 PHOS-T P-WET MG/L	00535 RESIDUE VOL NFLT MG/L	00530 RESIDUE TOT NFLT MG/L
72/03/07	11 50		112.00	0	134	70.0K	44	12.0	14.00	10	19
72/03/08	11 50										
CP(T)-G			138.00	0	114	70.0K	51	14.0	11.60	5K	5K
72/03/09	11 35										
72/03/09	11 40										
CP(T)-G			152.00	0	120	203.0	50	12.0	12.00	5	20
72/03/10	11 40										
72/03/10	11 30										
CP(T)-G			121.00	0	118	66.0	71	12.0	11.40	8	23
72/03/11	11 30										
72/03/11	11 35										
CP(T)-G			95.00	0	120	72.0K	39	9.0	11.40	11	15
72/03/12	11 10										

DATE FROM TO	TIME OF DAY	DEPTH FEET	00500 RESIDUE TOTAL MG/L	00505 RESIDUE TOT VOL MG/L	00625 TOT KJEL N MG/L	00610 NH3-N TOTAL MG/L	00515 RESIDUE DISS-105 C MG/L	00520 RESIDUE VOL FLT MG/L	00620 NO3-N TOTAL MG/L	00010 WATER TEMP CENT	00403 LAB PH SU
72/03/07	11 50		315	88	6.100	5.000	296	78	5.50	20.0	8.8
72/03/08	11 50									19.0	
72/03/09	11 35									18.0	
72/03/08	11 50										
CP(T)-G			312	94	11.100	6.100	312	94	5.50		8.8
72/03/09	11 35										
72/03/10	11 40									20.5	
72/03/09	11 40										
CP(T)-G			319	76	13.300	6.600	299	71	5.70		8.8
72/03/10	11 40										
72/03/11	11 30									22.0	
72/03/10	11 30										
CP(T)-G			302	84	7.800	6.100	279	76	7.60		8.8
72/03/11	11 30										
72/03/12	11 10									21.5	
72/03/11	11 35										
CP(T)-G			322	109	7.200	7.100	307	98	7.10		8.8
72/03/12	11 10										

STORET RETRIEVAL DATE 72/06/10

641064 AP-08 F-113-53

AIR PRODUCTS AND CHEMICALS INC.
ESCAMBIA RIVER STUDY

ESCAMBIA R. AIR PRODUCTS & CHEM.
12 FLORIDA
SOUTHEAST
ESCAMBIA RIVER
1113S000 2444220

DATE FROM TO	TIME OF DAY	DEPTH FEET	00058 FLOW RATE GPM	00435 T ACDITY CACO3 MG/L	00410 T ALK CACO3 MG/L	00310 BOD 5 DAY MG/L	00340 COD HI LEVEL MG/L	00680 T ORG C C MG/L	00665 PHOS-T P-WET MG/L	00535 RESIDUE VOL NFLT MG/L	00530 RESIDUE TOT NFLT MG/L
72/03/09	11 45			14	6		5	1.0K	0.01K	5K	5K
72/03/10	11 25			14	12		6	1.0K	0.01K	5K	5K

DATE FROM TO	TIME OF DAY	DEPTH FEET	00500 RESIDUE TOTAL MG/L	00505 RESIDUE TOT VOL MG/L	00625 TOT KJEL N MG/L	00610 NH3-N TOTAL MG/L	00515 RESIDUE DISS-105 C MG/L	00520 RESIDUE VOL FLT MG/L	00620 NO3-N TOTAL MG/L	00010 WATER TEMP CENT	00403 LAB PH SU
72/03/09	11 45		23	5			23	5K			5.5
72/03/10	11 25		18	9			18	9			6.0
72/04/13					2.000	1.900			2.00		
72/04/14					1.600	1.600			1.50		

ELEMENTS IN WASTEWATER STREAMS
AIR PRODUCTS AND CHEMICALS, INC.
ESCAMBIA PLANT
MARCH 6-12, 1972

Elements detected by spark source-mass spectrometer scan:

Final Pond Effluent (Station AP-1)

Lead	Silver	Bromine	Cobalt	Calcium	Aluminum
Cerium	Rhodium	Selenium	Iron	Potassium	Magnesium
Lanthanum	Molybdenum	Arsenic	Manganese	Chlorine	Sodium
Barium	Zirconium	Zinc	Chromium	Sulfur	Fluorine
Cesium	Strontium	Copper	Vanadium	Phosphorus	Boron
Tin	Rubidium	Nickel	Titanium	Silicon	

Echo Pond Influent (Station AP-4)

Lead	Barium	Rubidium	Nickel	Potassium
Terbium	Cesium	Bromine	Cobalt	Chlorine
Gadolinium	Antimony	Selenium	Iron	Sulfur
Neodymium	Ruthenium	Arsenic	Manganese	Phosphorus
Praseodymium	Molybdenum	Gallium	Chromium	Silicon
Cerium	Niobium	Zinc	Vanadium	Aluminum
Lanthanum	Strontium	Copper	Calcium	Sodium
Borow	Fluoride	Magnesium		

Metal concentrations determined by atomic asorption methods:

Station AP-1		Station AP-4		Station AP-8	
Parameter	Conc (μg/l)	Parameter	Conc (μg/l)	Parameter	Conc (μg/l)
Chromium	80	Chromium	2,150	Chromium	<50
Cobalt	<100	Cobalt	<100	Cobalt	<100
Copper	<50	Copper	<50	Copper	<50
Manganese	200	Manganese	1,050	Manganese	<50
Titanium	<500	Titanium	<500	Titanium	<500
Vanadium	<500	Vanadium	<500	Vanadium	<500
Zinc	100	Zinc	420	Zinc	<10

APPENDIX D

CORRESPONDENCE

Air Products and Chemicals INC.
ESCAMBIA PLANT
P.O. Box 467, Pensacola, Florida 32502

April 24, 1972

Mr. Charles Sweatt
Environmental Protection Agency
Surveillance & Analysis Division
College Station Road
Athens, Ga. 30601

Re: Escambia Plant, Air Products and Chemicals, Inc. - Source-Control Program

Dear Mr. Sweatt:

Per your request of April 19, 1972 at the Escambia Plant meeting for reviewing the wastewater survey, I have worked up the following list of source-control projects. These projects are organized as before and after 1970, according to your request.

Escambia Plant Source-Control Program

Timetable		Project
Pre-1970	1.	Cooling towers.
	2.	Catalytic combustor on HNO_3 plant.
	3.	Ammonia recovery from copper liquor system.
	4.	Aqua ammonia stripper and vaporizer system.
	5.	Methanol by-product disposal (to boilers).
	6.	Compressor building oil recovery and disposal.
	7.	Amines by-product disposal (flare).
	8.	Nitric acid oil recovery and disposal.
	9.	Methyl methacrylate effluent pH control.
	10.	Methyl methacrylate effluent stabilization basin.
	11.	Ammonium nitrate remelt and reclaim system.
After Jan. 1970	1.	NPK recycle pond.
	2.	Recovery of weak aqua ammonia from percolation.
	3.	HNO_3 concentrator recycle and reclaim system.
	4.	HNO_3 plant start-up weak acid recovery and recycle.
	5.	NPK plant shutdown.
	6.	DNT organics recovery and recycle.
	7.	Ammonia condensate stripper.

Timetable		Project
After Jan. 1970 (cont'd)	8.	Ammonia cascade cooler on cooling tower recycle.
	9.	Nitric acid recovery and recycle from DNT spent acid stripper.
	10.	Methanol distillation system put on reboilers.
	11.	Methanol still bottoms recycled as cooling tower makeup.
	12.	Ammonium nitrate solutions recovery and recycle.
	13.	Totally enclosed ammonium nitrate prill loading and bulk conveyor system.
	14.	NPK gyp pond removal for agricultural use.
	15.	Cooling towers.

The above list of projects details source-control projects and not treatment projects (i.e., biological ponds, chemical treatment, percolation, etc.). Should you require additional information, please let us know.

Very truly yours,

AIR PRODUCTS AND CHEMICALS, INC.
Escambia Plant

Roy L. Duggan

R. L. Duggan
Environmental Engineer

RLD/as