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**NORTH CAROLINA PHOSPHATES AND THE TEXAS GULF
SULPHUR COMPANY PROJECT AT THE ASHEVILLE
MINERALS RESEARCH LABORATORY**

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Introduction

In October of 1961 Dr. Leo J. Miller of Texas Gulf Sulphur (TGS) Company contacted the Asheville Minerals Research Laboratory, which is a part of the Engineering Research Department of the North Carolina State University at Raleigh, and requested assistance in the development of a phosphate ore body in Beaufort County. The main objectives of the work at Asheville were defined as follows. The Laboratory should try to determine on the basis of drill core samples, submitted by Texas Gulf Sulphur Company, the highest grade of phosphate concentrate that could be obtained with high recoveries from North Carolina ore, the thickness of the ore body that could be treated economically in one section, and the quantity of concentrate that could be obtained per acre. The results should be compared with data obtained from the treatment of Florida phosphate ores. It was important to use mineral preparation methods during the initial laboratory work that could be verified later by continuous pilot plant operation and could be scaled-up for a commercial plant.

The writer was placed in charge of the project at the Asheville Minerals Research Laboratory and he participated in the development of one of North Carolina's greatest mineral resources from the first drill core examination to the production of the first poundage and tonnage quantities of flotation concentrates. The Asheville Minerals Research Laboratory is the only organization in North Carolina with the staff and facilities to assist in the evaluation and development of mineral properties. At the January 1966 Advisory Committee meeting of the Minerals Research Laboratory at Texas Gulf Sulphur Company's seventy-seven million dollar phosphate plant site at Lee Creek, North Carolina, Dr. Miller stated that without the testing projects the Laboratory had carried out the phosphate operation might have been a year behind its present status. The phos-

phate mine and mill will be in operation in early 1966 and initially will produce three million tons of phosphate concentrate a year.

Now that a phosphate industry has been established in the State, it seems an opportune time to review developments leading up to this, especially the cooperative efforts of a State supported organization and a private company. For a better understanding of North Carolina phosphates, a few words pertaining to the phosphate industry in general, the history of North Carolina phosphates, and the modern approach of Texas Gulf Sulphur Company to the problems of exploration, acquisition and development of the mineral property, which went hand in hand with the work at Asheville, shall precede the project description.

The Phosphate Industry

Almost all commercially produced phosphate in the United States is of marine sedimentary origin. The phosphate granules or pebbles are either in unconsolidated matrix, containing phosphate pebble diluted by quartz sand, calcium carbonate and clay as found in North Carolina, South Carolina and Florida, or in consolidated formations of semi-hard shales as in Idaho, Montana and Utah.

The principal phosphate-bearing mineral in the North Carolina ore has been investigated by James R. Lehr of the Tennessee Valley Authority at Wilson Dam and by Dr. T. Rooney at Columbia University. The phosphate-bearing mineral is a carbonate fluorapatite or francolite, that is, a fluorapatite in which an excess of fluorine and carbonate replaces phosphate in the apatite lattice.

Commercial phosphate is classified according to grade or content of phosphorus. Custom has developed three terms to designate grade: BPL, an abbreviation for bone phosphate of lime (tricalcium-phosphate); P_2O_5 , phosphorus pentoxide; and P, the element phosphorus. The miners in

Florida use BPL, in the West P_2O_5 is used, and the elemental phosphorus industry uses P. Multiplication by a factor of 2.29 converts P into P_2O_5 and by a factor of 2.185 converts P_2O_5 into BPL. There is also confusion in units of weight. Florida uses long tons and the West uses short tons. In the developing North Carolina phosphate industry, grades are expressed in P_2O_5 and weights are expressed in short tons.

In order to produce a commercial phosphate product, the phosphate matrix has to be mined and separated into valuable pebble and wasted sand, clay and calcium carbonate. A phosphate pebble with a high P_2O_5 or BPL content, a low lime to phosphorus ratio (CaO/P_2O_5) and a minimum of other diluents such as fluorine, iron, aluminum, magnesium and hydrocarbon is desired.

About 70 percent of the phosphate pebble produced in the United States goes into fertilizer production. A major part is still used to produce super-phosphate by reacting ground phosphate pebble with sulfuric acid followed by curing. The trend, though, is towards increased production of wet phosphoric acid from phosphate pebble and sulfuric acid and the production of triple super-phosphate by reacting ground phosphate pebble with phosphoric acid. Diammonium phosphate is produced by reacting ammonia with phosphoric acid. Triple super-phosphate and diammonium phosphate are concentrated products which are raw materials for mixed fertilizers.

History of Development of North Carolina Phosphates

First interest in Beaufort County and surrounding Pamlico and Hyde County phosphates arose, according to Stuckey (1), in 1951 when AMCO Exploration Inc., a subsidiary of American Metals Company, started exploration for phosphate and obtained leases from the State for phosphate-bearing minerals for an area underlying the Pamlico River and tributaries from a point six miles upstream from the city of Washington, North Carolina to thirty miles downstream of Washington, North Carolina. In 1953 AMCO Exploration cancelled the lease with the statement, "... have failed to make any discovery of a commercial ore body of phosphate bearing minerals. . . ." In 1957 Kennecott Copper Corporation through its subsidiary, Bear Creek Mining Company, and Sun Oil Company through its

subsidiary, General Crude Oil Company, acquired leases and began to explore for phosphate in Beaufort County. Both companies withdrew from the area in 1958. In the case of General Crude Oil Company the Asheville Minerals Research Laboratory did a small amount of beneficiation work. A consulting firm discouraged General Crude Oil through an economic comparison with Florida practice. Texas Gulf Sulphur looked at North Carolina phosphates in 1959 but considered the mining cost prohibitive at that time.

Two main problems have beset the North Carolina phosphate development from the beginning. The first problem is mining of a deposit which is below sea level and which is near an ocean-connected water body. It is overlain by 50 to 200 feet of overburden and underlain by a major aquifer, the Castle Hayne formation. The second problem is the production of high-grade phosphate concentrate from the ore or matrix without dilution by coquina, shell and dolomitic limestone which is found in some sections of the ore body.

The first encouraging report on the phosphate potential in the area was presented by Brown (2) in 1958, indicating a major phosphate ore body of economic importance together with a theory of the possible origin of the phosphate deposit of Beaufort County, North Carolina. An optimistic look at Brown's data reveals a mineral potential of around four billion tons of phosphate concentrate under an area of 450 square miles or approximately 14,000 tons of phosphate concentrate per acre. These are interesting figures indeed, when compared with Florida, the number one phosphate producing state, where most of the mined land yields less than 10,000 tons of phosphate pebble per acre.

In 1961 A. L. Nash, from Concord, North Carolina, brought the first tonnage quantity of North Carolina phosphate to the surface while trying to develop a hydraulic mining technique especially suited to the deeper areas of the Beaufort County deposit north of the Pamlico River. The method was based on the hope of removing large quantities of phosphate ore from the deposit by pumping, without removing or caving the caprock and overburden. The coquina caprock caved in and the venture failed as an economical mining method. However, Nash's work was extremely helpful from another standpoint, as it supplied ore for beneficiation studies at the Asheville Minerals Research Laboratory, for the Texas Gulf Sulphur pilot plant in Asheville in 1962, for the

larger Texas Gulf Sulphur pilot plant at Lee Creek in 1964, and for a Food Machinery Corporation (FMC) pilot plant in Asheville in 1965.

In 1961 interest in North Carolina phosphates was renewed by three companies: Texas Gulf Sulphur Company, Magnet Cove Barium Corporation, and North Carolina Phosphate Corporation (a joint venture of Kennecott Copper Corporation and American Agricultural Chemical Company). The State received bids in 1962 for leases of State-owned river bottoms under Pamlico River blocks A, B, C, D, E, F, and under Pungo River blocks G, H, I (Figure 1). Texas Gulf Sulphur Company, as highest bidder, was awarded the option to lease block D. Magnet Cove Barium Corporation bid on, and was awarded, options to lease blocks G, H, and I of the Pungo River. In 1964 FMC Corporation leased blocks J and K. The various interested companies have also acquired many thousands of acres of private land adjacent to the State leases.

The Texas Gulf Sulphur Approach to North Carolina Phosphates—A Text Book Example of Scientific Geology

Texas Gulf Sulphur Company's bid for the lease on the D block and acquisition of surrounding land was preceded by rapid collection of knowledge about the ore body by means of modern methods and by a comparison with other producing phosphate deposits in the United States. A major tool in the early work of Texas Gulf Sulphur Company was gamma ray logging of existing water wells. The small amount of radioactive uranium, and possibly thorium, minerals in the North Carolina phosphate pebble, as mentioned by Brown (2), gives a gamma ray count proportionate to the phosphate content of the ore. Gamma ray logging therefore shows the approximate ore grade and also defines the contacts between the Pungo River phosphate formation and the discordantly overlying Yorktown formation and the discordantly underlying Castle Hayne formation.

By March 1962 Dr. Miller of Texas Gulf Sulphur Company had gained knowledge about the extent of the ore body, especially south of the Pamlico River, the thickness of the phosphate formation and of the overburden. A map with overburden and ore isopachs was drawn and the limits of an ore body with a minimum thickness of 40 feet, a maximum ratio of overburden to ore of 2 : 1 and with less than 200 feet total depth

were delineated. Dr. Miller believed that a deposit meeting these requirements had possibilities for dry, open-pit mining if the amount of water inflow could be determined and controlled. The area around Lee Creek met the above requirements and could be secured by Texas Gulf Sulphur Company partly because not too much was known up to then about the extent of the ore body south of the river. This information was made public through a gamma ray logging project by the North Carolina Department of Conservation and Development, in cooperation with the U. S. Geological Survey, started in March 1962 and which has been on open file since 1963 and was published in 1965 (4). A map showing overburden thickness and phosphate ore body thickness, as computed from the data given in the report, is presented in Figure 2.

Without drilling, Texas Gulf Sulphur Company gained control over a most valuable part of the North Carolina phosphate ore body. A relatively small number of drill holes put down in 1962 provided more exact geologic knowledge and material for studies about the physical quality of the phosphate ore, the amenability to concentration and the quality and quantity of concentrate that could be extracted.

Evaluation of Texas Gulf Sulphur Company Cores

Drill Core Preparation—Texas Gulf Sulphur split the moist drill core into quarters, kept one quarter for reference, sent a quarter to the Colorado Research Foundation for test work, and sent one quarter to Southern Testing and Research Laboratories for P_2O_5 analysis. The fourth quarter was sent to the Asheville Laboratory for evaluation and flotation test work.

Defining of Low-Grade and High-Grade Sections—From each hole, composites were prepared on the basis of P_2O_5 analysis and physical characteristics. Efforts were made to treat section composites of at least 30 to 40 feet thickness representing the high-grade (plus 10 percent P_2O_5) ore zone. The phosphate section was divided into three zones, the upper low-grade, containing coquina, the high-grade ore zone, and the lower low-grade ore (Figure 3). The Minerals Research Laboratory developed methods for beneficiating both high-grade and low-grade zones, but most of the core work was done on the high-grade section only.

Physical Separation—The high-grade core com-

posite from each hole was first mixed with water, sized at 14 and 28 (or 35) mesh and deslimed on 200 mesh. The relatively small amount of plus 14 mesh material consists of phosphate pebbles diluted by dolomitic limestone and quartz. The small amount of minus 14 plus 28 mesh material consists of phosphate pebble and quartz which can be up-graded by agglomeration flotation. The large amount of minus 28 plus 200 mesh material, consisting of round phosphate pebble with angular and round quartz, was treated by flotation to obtain the principal phosphate concentrate. The minus 200 mesh slimes, mainly silt and montmorillonite-type clay, were wasted.

Flotation Concentration of North Carolina Phosphate—Flotation is a separation method based on the fact that one mineral can be rendered hydrophobic (water repellent) preferentially over another which stays hydrophylic (water wetted). After conditioning with selected reagents in aqueous suspension, the hydrophobic mineral can be separated from the hydrophylic mineral by agitation and aeration of the aqueous suspension. The hydrophobic mineral attaches to air bubbles, floats to the surface and is removed. The hydrophylic mineral stays in the aqueous suspension. A separation is thereby achieved. This procedure is of utmost importance because it provides a reliable tool for beneficiating large tonnages efficiently. More than 750,000 tons of ore is treated daily by flotation in the United States. Texas Gulf Sulphur Company will treat initially around 24,000 tons per day of flotation feed.

For the separation of North Carolina phosphate pebble from quartz three flotation methods can be employed. (1) First, it is possible to condition the deslimed sands with an anionic, fatty acid-type collector and float the hydrophobic phosphate away from the quartz. A fair-grade rougher concentrate with 26 to 29 percent P_2O_5 and with phosphate recoveries of over 95 percent can be obtained. By multiple cleaning, a concentrate grade of 29 to 30 percent P_2O_5 can be obtained with a fair recovery (80 to 90 percent). (2) Second, it is possible to render the quartz hydrophobic and float it away from the phosphate pebble with a cationic, amine-type collector. Acceptable concentrate grades of 30 to 31 percent P_2O_5 with fair recovery of 80 to 90 percent can be obtained. The amine system is very sensitive to slimes and reagent variations and it therefore causes operation problems. There are also problems involved in

floating the coarser quartz without losing fine phosphate in the float waste product. (3) The most reliable system for obtaining high concentrate grades, 30 to 31 percent P_2O_5 , with high flotation recoveries, 90 to 95 percent, is a combination of an anionic float followed by dereagentizing and then a cationic float. The cationic float removes the fine silica from the fatty acid concentrate with little loss of phosphate. This double flotation method is used extensively in Florida where it was developed by Crago (5) in 1941. It allows more variation in flotation feed and plant conditions than either the single anionic or single cationic flotation procedures. The double flotation procedure was advocated by the Minerals Research Laboratory and a technique was developed to treat the core samples from the Texas Gulf Sulphur Company drill project which consistently gave high-grade concentrates with high recoveries.

Phosphate Core Evaluation Procedure—The feed preparation, flotation, and agglomeration flotation steps used in evaluating the phosphate cores are described as follows:

Feed Preparation

Tumble 600 grams of wet sample at 40 to 50 percent solids in 8" x 9" laboratory mill with 440 gram rubber rod for 15 minutes.

Screen out plus 14 mesh material.

Wash remaining mud balls, if any, through screen with water spray.

Decant minus 14 mesh material on 200 mesh screen to 60 to 70 percent solids.

Save minus 200 mesh slimes.

Scrub minus 14 mesh material at 60 to 70 percent solids for 10 minutes with 4-bladed, 3-inch impeller at 1200 rpm.

Screen out plus 28 mesh material.

Deslime minus 28 mesh twice on 200 mesh after diluting to 10 percent solids.

Settle minus 28 mesh material to about 60 to 65 percent solids.

Flotation of Minus 28 Plus 200 Mesh Material

Condition at 60 to 65 percent solids for 5 minutes with 4-bladed, 3-inch impeller at 700 rpm after adding saponified fatty acid (0.3 to 0.8 lb/ton of feed) and fuel oil (0.5 to 1.0 lb/ton of feed).

Float in Denver 500-gram laboratory flotation machine.

Clean float product once.

Scrub cleaned concentrate with H₂SO₄ (2 to 4 lb/ton of feed) at high solids for 5 minutes.

Wash twice after diluting to about 10 percent solids.

Float remaining quartz after conditioning for 15 seconds in cell with amine acetate (0.15 to 0.3 lb/ton of feed).

Agglomeration of Minus 14 Plus 28 Mesh Material

Condition this coarse fraction with saponified fatty acid and fuel oil for 5 minutes.

Agglomerate float with water spray.

The results of a typical hole are presented in Table 1.

Table 1

Flotation Test Results of Hole 23—106 to 142 ft.

	% P ₂ O ₅ *	% Insol.	Gross Wt. P ₂ O ₅		
			in Tons	Tons	% of Total
Phosphate Matrix	15.32	—	100.0	15.32	100.0
+14 Mesh Pebble	23.70	—	3.4	0.79	5.1
—14+28 Mesh Conc.	28.65	4.06	2.2	0.63	4.1
—28+200 Mesh Conc.	30.60	2.16	43.0	13.15	86.0
Total Flot. Conc.	30.50	—	45.2	13.78	90.1
Waste Tailings	1.60	—	32.8	0.53	3.4
Slimes, —200 Mesh	1.20	—	18.6	0.22	1.4

* P₂O₅ analyses by Southern Testing & Research Laboratories, Wilson, N. C.

Comparison of Asheville Practice With Others
—Texas Gulf Sulphur Company was very encouraged when concentrate grades of up to 31 percent P₂O₅ with high recoveries were produced at the Asheville Laboratory from the first drill core in January 1962 and from succeeding drill cores during the following month. The concentrate grade was so high that the other investigator on the project questioned the methods and assay procedures used by the Asheville Laboratory (Table 2).

Table 2

Concentrate Analysis Before and After Calcining—
Hole 8-B

	As Is*	Calcined at 1750°F 2 hrs**	
% P ₂ O ₅	31.2	33.50	33.73
% CaO	49.09	52.57	52.63
% CO ₂	4.66	2.16	2.06
% Acid Insol.	1.80	—	—
% Weight Loss	—	10.0	—

* Analysis by Three Gee Dee Company, Pembroke, Florida

** Analysis by Southern Testing & Research Laboratories, Wilson, N. C.

A single float procedure had been used by the other investigator, rather than the double float

procedure as used at the Asheville Laboratory. After a comparison of the assay procedures and flotation methods used by the two laboratories, verification of the high-grade concentrate and high recoveries at the Asheville Laboratory was obtained.

The Asheville flotation method for North Carolina phosphate ores differs from Florida practice, and other investigators' methods, in that partially saponified, tall oil fatty acid of definite quality is used instead of crude tall oil and NaOH. The apparent advantages over Florida practice are mentioned here in order of importance.

1. Percent solids in the conditioner can be kept lower when using saponified fatty acid than when using crude tall oil and NaOH, thereby saving wear and power and minimizing slime formation.

2. Less fatty acid is necessary because of better dispersion and higher fatty acid content. This more than compensates for the higher cost of refined tall oil of uniform quality.

3. Less NaOH is necessary and conditioning and flotation pH can be held below 9.

4. Less H₂SO₄ is needed in the following fatty acid removal step because of uniformity of the Ca-fatty acid salt coating on the phosphate pebble.

Results of Core Evaluation—In the first phase of the drill core program, 67 drill holes were evaluated and Texas Gulf Sulphur (TGS) Company was informed that from a large part of their property approximately 30,000 tons of high-grade concentrate could be recovered from each acre by treating only the 40-foot high-grade ore zone. The hard layers of high calcium carbonate and high magnesium carbonate content in the high-grade ore body could be separated from the bulk of the material by simple screening methods. A very good picture about the uniformity of the ore body was obtained.

The core evaluation also included calcining of the concentrates. By calcining under controlled conditions, the concentrate is up-graded through removal of hydrocarbon and CO₂. A higher grade phosphoric acid can be produced from calcined concentrate and the weight loss of about 10 percent will result in freight savings.

Comparison of North Carolina Phosphate Ore With Florida Ore

Samples from nine Florida phosphate pits, representing eight operating companies, were

processed in the Minerals Laboratory in April 1962. To make comparisons with North Carolina results the same laboratory procedures were used to process the Florida samples. The comparison of Florida and North Carolina data looked very encouraging. The area Texas Gulf Sulphur Company was interested in would yield more than five times the amount of phosphate concentrate per acre than was obtained in Florida. The amount of overburden and matrix to be mined per ton of concentrate was the same as in Florida. The reagent amounts necessary to up-grade the ore by flotation were much less per ton of concentrate than in Florida. The North Carolina phosphate pebble is not weathered and is harder than Florida pebble. The amount of phosphate lost in the slimes is only a tenth as great as the loss in Florida per ton of ore treated. Less favorable factors are that the bulk of North Carolina phosphate concentrate contains only 30.5 to 31 percent P_2O_5 , whereas the average Florida concentrate grade is 33 percent P_2O_5 . North Carolina phosphate concentrate has a slightly higher CaO to P_2O_5 ratio than Florida phosphate pebble, which means that slightly more sulfuric acid is necessary in producing of super-phosphate or phosphoric acid. A lower alumina and iron content in the North Carolina phosphate concentrate, about half the amount generally found in Florida phosphate material, is favorable. Also, the reaction rate of North Carolina phosphate with sulfuric acid in the conversion process is faster than with Florida phosphates.

The TGS Phosphate Flotation Pilot Plant at the Asheville Minerals Research Laboratory—Pilot Plant Objective and Operation

Samples of concentrate weighing up to 50 pounds were produced by batch procedures during the drill core evaluation for shipment to prospective customers. Tonnage quantities of concentrate were needed for super-phosphate and phosphoric acid test work. In November 1962 Texas Gulf Sulphur Company requested rapid erection at Asheville of a pilot plant that could produce tonnage quantities of phosphate concentrate from ore which A. L. Nash had pumped to the surface at Gum Point on the northern shore of Pamlico River on land leased by Texas Gulf Sulphur Company. The pilot plant later treated a number of large diameter drill cores.

A 500-pound-per-hour pilot plant was designed

around the equipment available at Asheville and was set up within one month with financial assistance from Texas Gulf Sulphur Company. On December 18, 1962 the first experimental run produced acceptable concentrate. By March 1963 about 35 tons of ore had been processed. The final pilot plant run on April 3, 1963 was made on drill core in order to verify the process on ore like that which would be obtained by dry mining methods. Representatives of Texas Gulf Sulphur Company and engineering companies observed the pilot plant run on drill cores and were satisfied that the process could treat North Carolina phosphate ore reliably. In all pilot plant runs acceptable concentrates grades of plus 30 percent P_2O_5 were obtained. The recovery was very high in a few runs, but was lower in others depending on variables studied, such as reagent quantities and screening methods.

The flowsheet of the Texas Gulf Sulphur Company pilot plant at Asheville is presented in Figure 4.

Pilot Plant Description

Feed Preparation—The ore was fed on a belt feeder at a constant rate of 500 pounds per hour into an impeller-type scrubber, was diluted and pumped onto a 28 mesh trommel or stationary Wedge Wire screen. The small amount of screen oversize was kept separate. The screen undersize was diluted to about five percent solids and deslimed in the first cyclone. The overflow from No. 1 cyclone contained all slimes removed. Slime samples were kept for settling characteristics and thickener requirement determinations. The cyclone underflow was densified in a screw classifier and scrubbed at 65 percent solids in a Wemco-type attrition scrubber. The scrubbed material was deslimed in No. 2 cyclone after dilution in the No. 3 pump sump. The No. 2 cyclone overflow was recirculated to the No. 2 pump sump. The cyclone underflow was densified to 75 to 80 percent solids in the No. 2 screw classifier.

Fatty Acid Flotation—The sized, deslimed and densified material was fed to the fatty acid conditioner where the percent solids was adjusted by adding water in metered quantities. The flotation feed was conditioned with saponified fatty acid and fuel oil. The feed was then diluted to flotation density and fed to the Denver pilot plant flotation machine. A rougher float was followed by two cleaner steps. The tailings from the cleaners were

circulated to the rougher machine. The fatty acid tailings were sampled and discarded. Ninety-six to ninety-eight percent of the P_2O_5 in the flotation feed was recovered in a fatty acid concentrate containing five to ten percent insolubles. The type concentrate produced in the fatty acid float could be used only for the production of phosphoric acid in a captive plant and would require upgrading by an amine float for open-market sales.

Acid Scrub and Amine Flotation—The fatty acid concentrate was conditioned in an impeller-type scrubber with sulfuric acid. The concentrate was then washed in a fluid-bed-type, V-box washer to remove the fatty acid-sulfuric acid reaction products. In the next float a small amount of sodium hydroxide was used for pH control and the remaining silica was floated away from the phosphate concentrate by an amine acetate collector. Ninety-five to ninety-six percent of the P_2O_5 in the flotation feed was recovered in a concentrate containing 30 to 31 percent P_2O_5 and less than three percent insolubles. The final concentrate was dried in a rotary dryer before shipment to prospective customers in the United States and as far as Japan and Europe.

Pilot Plant Control—All feed streams into the pilot plant, such as ore, water, and reagents, were metered continuously. The product streams out of the pilot plant were sampled at intervals by taking timed samples on a routine schedule. Material balances were made on the basis of sample weights and P_2O_5 assays. The assay method employed is based on the color intensity of molybdovanadophosphoric acid complex which is measured in a colorimeter. A number of samples were sent out to other laboratories for checks and standardization of the method.

The pilot plant circuit ran very reliably and could be operated by one engineer, two technicians and one utility man.

Pilot Plant Results—In Table 3 the material balances for pilot plant runs on pumped ore are presented together with the amount of reagent used.

Table 3

Pilot Plant Flotation of Pumped Ore From Gum Point —
Material Balance and Reagent Schedule

Material	% Weight	% P_2O_5	% Insol.	% P_2O_5 Distr.	Flot. Recovery
+28 Mesh Oversize	10.8	20.9	—	11.4	—
—28 Mesh Concentrate	55.5	30.5	3.2	82.6	96.3
Fatty Acid Tailings	28.8	1.1	—	1.8	—
Amine Tailings	3.2	8.6	—	1.4	—
—200 Mesh Slimes	3.7	15.9	—	3.0	—
Feed Sample	100.0	19.8	—	100.0	—

Reagent Consumption
(lbs/Ton of Feed)

Tall Oil	0.4
NaOH	0.06
H_2SO_4	2.4
NaOH	0.13
Amine Acetate	0.18
Fuel Oil	0.8

Pilot plant photos of the general layout, ore feeder, desliming circuit, flotation section, phosphate drying, and a pilot plant equipment description are presented in the Appendix.

The Larger Pilot Plant Operated by Texas Gulf Sulphur Company at Lee Creek

Texas Gulf Sulphur Company planned to test the feasibility of wet, and possibly dry, mining by experimental dredging with a conventional cutter-head dredge by lowering of the water table in a test pit to reach the ore body. It was desirable to determine if the recommended process would give satisfactory results with freshly-mined ore contaminated with overlying material. The Asheville Minerals Research Laboratory was therefore asked to present plans for a larger pilot plant to be erected at the test pit site at Lee Creek, North Carolina. The objectives of this pilot plant were stated as follows:

- 1) Find minimum equipment requirements to reliably concentrate dredged ore and obtain design data for the projected plant.
- 2) Obtain exact reagent and water requirements in continuous operation using local water.
- 3) Become familiar with the dredged ore and handling problems and train personnel for the projected plant.
- 4) Establish standard sampling and mill control procedures in the field.
- 5) Provide large-tonnage samples for customer evaluation, and larger-scale calcining and processing.

Based on the experience and data obtained in Asheville, a proposal for a larger pilot plant was presented to Texas Gulf Sulphur Company in May 1963. It recommended a 12-ton-per-hour feed preparation circuit, stockpiling facilities for the flotation feed, and a 2.7-ton-per-hour flotation circuit. The proposal contained a flowsheet (see Figure 5), equipment size and cost, estimated construction and operating cost and a construction and start-up time plan. A general layout for the larger pilot plant was also presented. It was recommended that the equipment be unitized so

that quick changes could be made and minimum equipment needs determined. The plans presented were based on equipment available through Denver Equipment Company which specializes in flotation pilot plant equipment. Most of the equipment used in the smaller Asheville pilot plant was Denver Equipment Company type.

To expedite the construction of the larger pilot plant, Denver Equipment Company was engaged by Texas Gulf Sulphur Company to assist in the detailed design and to furnish the equipment. The pilot plant was erected at Lee Creek in the fall of 1963. An engineer of the Asheville Laboratory joined the Texas Gulf Sulphur Company staff, and during 1964 the Lee Creek phosphate pilot plant was operated and the objectives were fulfilled. In July of 1964 Texas Gulf Sulphur Company awarded Brown and Root of Houston, Texas and Rea Construction Corporation of Charlotte, North Carolina the contract to erect a phosphate mill, with all supporting facilities, that can produce 3,000,000 tons of phosphate concentrate per year from North Carolina ore. This plant was designed and erected in 1965 and it began operation in March 1966.

Additional Metallurgical Studies at the Asheville Laboratory

For support of the pilot plant work at Lee Creek, studies of important variables in the concentration circuit were made at Asheville. The influence of the different possible water sources at Lee Creek on the flotation results was tested. The influence of dilution of high-grade ore by low-grade ore was studied. Methods to up-grade the low-grade ore sections were developed and the economics of treating the low-grade sections of the ore body were worked out. Further flotation test work with saponified fatty acid established that a very wide range of conditions, such as conditioning percent solids, conditioning time, and saponification ratios, would give high concentrate grades and recoveries. Variables in the flotation feed preparation were studied especially with respect to amount of slimes that could be tolerated in the flotation circuit. A differential grinding method was tested that could recover a fair percentage of the phosphate values in the plus 14 mesh oversize which was rejected in earlier work.

By-Products of Phosphate Mining

When the Lee Creek pilot plant began to

operate successfully in 1964, the attention of the Asheville Laboratory shifted from phosphate concentration studies to evaluation of possible by-products. Excellent samples of overburden and low-grade ore for use in by-product investigations had been made available by the experimental mining.

On the basis of physical properties, chemical analysis and concentratability, the main by-product sources were defined. Shells, coquina, dolomitic limestone and some ilmenite, which can be recovered from the overburden, and the flotation tailings from the concentrator represent the interesting by-products. Each is described below.

The Shells—In the upper 25 feet of the overburden in the upper Yorktown formation there is a 10 to 15-foot layer of shell matrix. This layer contains calcium carbonate in the form of fossil clam, oyster and coral shells. Laboratory tests showed that a part of the calcium carbonate material could be obtained, free of clay and sand contamination, by simple washing and screening methods. Since North Carolina has no commercial production of high-calcium limestone, this possible source is of special interest. The Asheville Laboratory assisted in a shell-recovery pilot plant project at Lee Creek using the available scrubbing and sizing equipment. Two-hundred-and-ten tons of shell matrix from the mining test pit was treated in a trommel scrubber-screen combination to provide tonnage samples for further testing.

A fraction of the clean shell material was tested as raw material for a lime plant to produce quicklime and hydrated lime. The analyses of the lime raw material and products are given in Table 4.

Table 4

	Lime Raw Material and Products				
	CaO	MgO	Insol.	Fe ₂ O ₃	CaCO ₃
-1½" + 4 Mesh Kiln Feed	54.4	0.1	1.1	0.16	97.5
-1½" + 4 Mesh Quicklime	96.08	0.38	0.3	0.52	—
-200 Mesh Hydrate	74.05	0.20	0.8	0.36	—

A flowsheet and capital and operating-cost estimates for a rotary-kiln lime plant to produce 300 tons per day of quicklime from 600 tons per day of shells were presented to Texas Gulf Sulphur Company by the Laboratory.

The ground shell was also evaluated as a calcium source in poultry feed supplements and it proved to be equal to material now imported into the State. The Laboratory prepared the samples

for the test work conducted by the North Carolina State University Poultry Science Department and also the samples sent to various poultry-feed mills.

Shell material can also be used as a cement raw material and as aggregate for road building and concrete block products.

There is a potential of about 350,000 tons per year of clean shell product at the planned phosphate production rate.

The Coquina—In the upper low-grade section of the phosphate horizon there are layers of hard, cemented coquina containing some phosphate. Tests showed that this material was suitable for aggregate after washing, crushing and sizing. A portion of the material meets the State abrasion specifications. This potential by-product is interesting because sources of hard rock for aggregate are scarce in the coastal area. The tonnages available, in the range of 700,000 tons per year, make it an attractive raw material for an aggregate plant. A flowsheet for a 300 ton-per-hour aggregate plant with capital and operating-cost estimates was prepared for Texas Gulf Sulphur Company by the Asheville Laboratory.

The finer, softer coquina material, containing low-grade phosphate ore, can be used for agricultural liming purposes after drying and grinding. Tonnage samples of this material were prepared by the Laboratory for testing on eastern Carolina soils.

The Dolomitic Limestone—In the upper ten feet of the high-grade ore section there occurs an irregular layer of very hard, dolomitic limestone which can be used, after crushing and sizing, as an aggregate source. It also makes a good agricultural liming material when dried and ground to North Carolina ag-lime specifications. The magnesium carbonate content of this material is very desirable for agricultural liming purposes. The Asheville Laboratory ran abrasive tests for road aggregate on this material and prepared tonnage samples of ground dolomitic limestone for agricultural testing. The dolomitic limestone meets the State abrasion specifications.

It is difficult to estimate the quantity of this material available because of the non-uniformity of this layer.

The Ilmenite—The top part of the Texas Gulf Sulphur Company overburden is a sand layer which forms a low ridge across the property. During the test pit operation in 1964, Laboratory

personnel sampled the pit faces and determined the heavy mineral content. The sand contained 0.97 percent by weight of heavy minerals, mainly ilmenite, and it analyzed 0.47 percent TiO_2 . Ilmenite concentrates containing 52.6 percent TiO_2 were produced in the Asheville Laboratory by gravity separation followed by magnetic separation. Because of the localized nature and economics of the heavy-mineral-bearing sands, no recovery of ilmenite is planned.

Sand-Lime Bricks From Shell Lime and Flotation Tailings—One possible use for two of Texas Gulf Sulphur Company's by-products is the manufacturing of sand-lime bricks. Sand-lime bricks or calcium silicate building products consist of high-quartz sand bonded with hydrated calcium silicates. These hydrated calcium silicates, similar to the bonding agents in Portland cement, are formed by the reaction of lime and silica under steam pressure. Sand-lime bricks can be made with high compressive strength, low absorption, and freeze and thaw resistance, in colors of natural silver gray or colored as desired. The colors are weather resistant and reproducible. The brick can be split for a textured surface.

The general manufacturing process consists of mixing of ground quicklime or hydrate with high-silica sand, in ratios of 85 to 90 percent sand to 15 to 10 percent lime, and adjusting of moisture for easy pressing and forming. The mixture is pressed at 4000 to 8000 psi and steam-cured in autoclaves at pressures of 150 to 250 psi for four to five hours. The bricks retain their exact dimensions during curing and can be shipped immediately after removal from the pressure vessel. The sand-lime brick manufacturing process can be completely automated so that the bricks are never touched by human hands.

Quicklime produced from Texas Gulf Sulphur Company's shells and flotation tailings sand was tested in the Asheville Laboratory for sand-lime brick production. Preliminary testing showed that an attractive silver gray sand-lime brick can be produced from these by-products of phosphate mining. The test bricks met the requirements of minimum compressive strength for individual bricks of grade SW of ASTM C-73-51.

Summary and Conclusions

The work described in this bulletin shows how a properly equipped and staffed minerals research organization can be of valuable help in establish-

ing mining industries in the State. Through close cooperation with Texas Gulf Sulphur Company, the Minerals Research Laboratory assisted in the evaluation of one of North Carolina's most valuable mineral resources, the phosphate in Beaufort County. A process for the concentration of North Carolina phosphate ores was developed at the Laboratory and tonnage samples for customer evaluation were produced in a pilot plant. Based on the Asheville results, plans for a larger pilot plant were presented to Texas Gulf Sulphur Com-

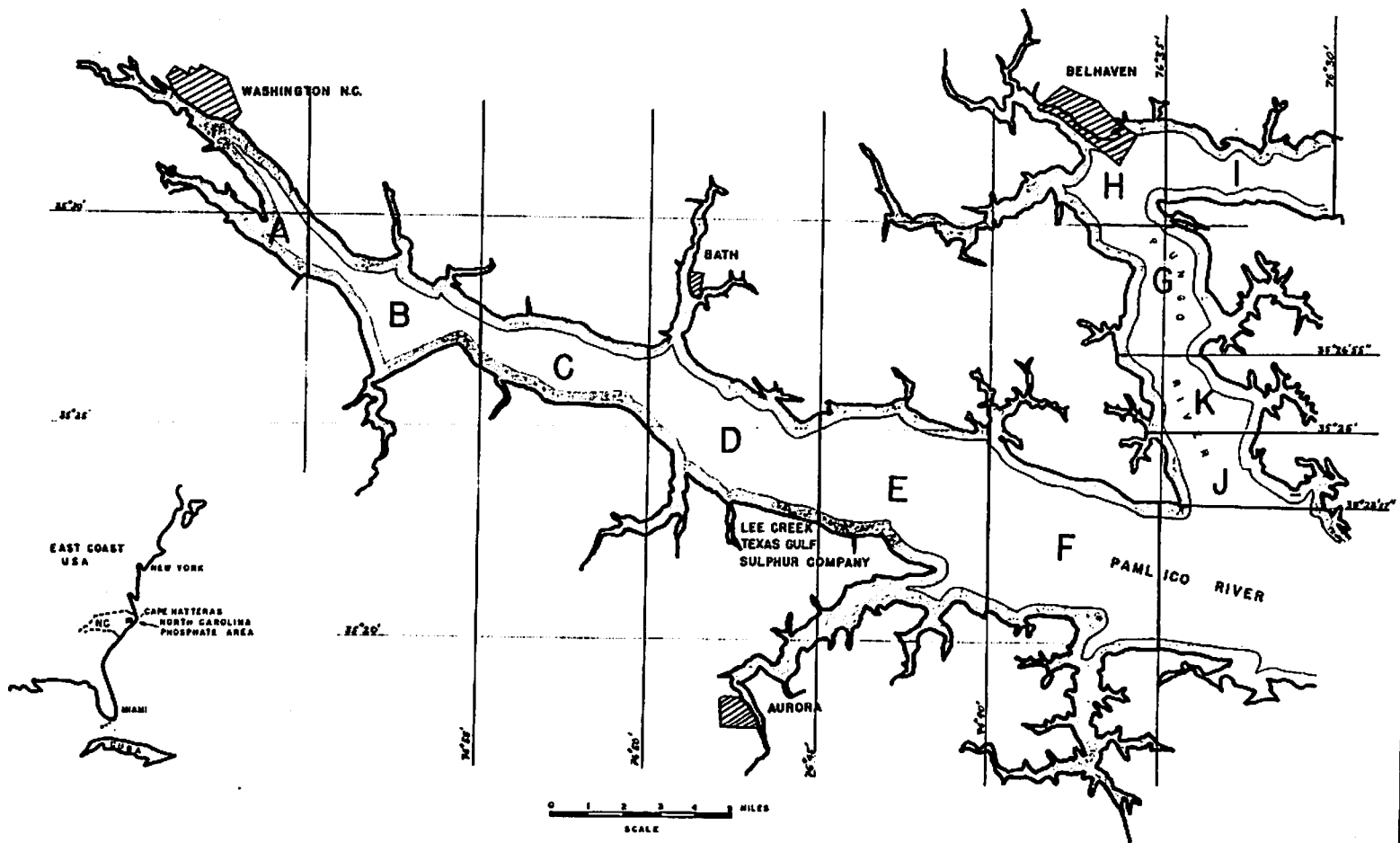
pany. This pilot plant provided larger samples from dredged ore using local water and it also provided design data for the production plant.

Studies on by-products from the phosphate mining operation outlined possible uses for high-calcium shells as lime raw material, coquina as aggregate, dolomitic limestone for agricultural liming purposes or aggregate, and flotation tailings and lime for the production of sand-lime bricks.

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5. Arthur Crago, U. S. Patent No. 2,293,640 (1942).

FIGURE I
NORTH CAROLINA PHOSPHATE AREA
LOCATION OF STATE LEASE BLOCKS A TO K



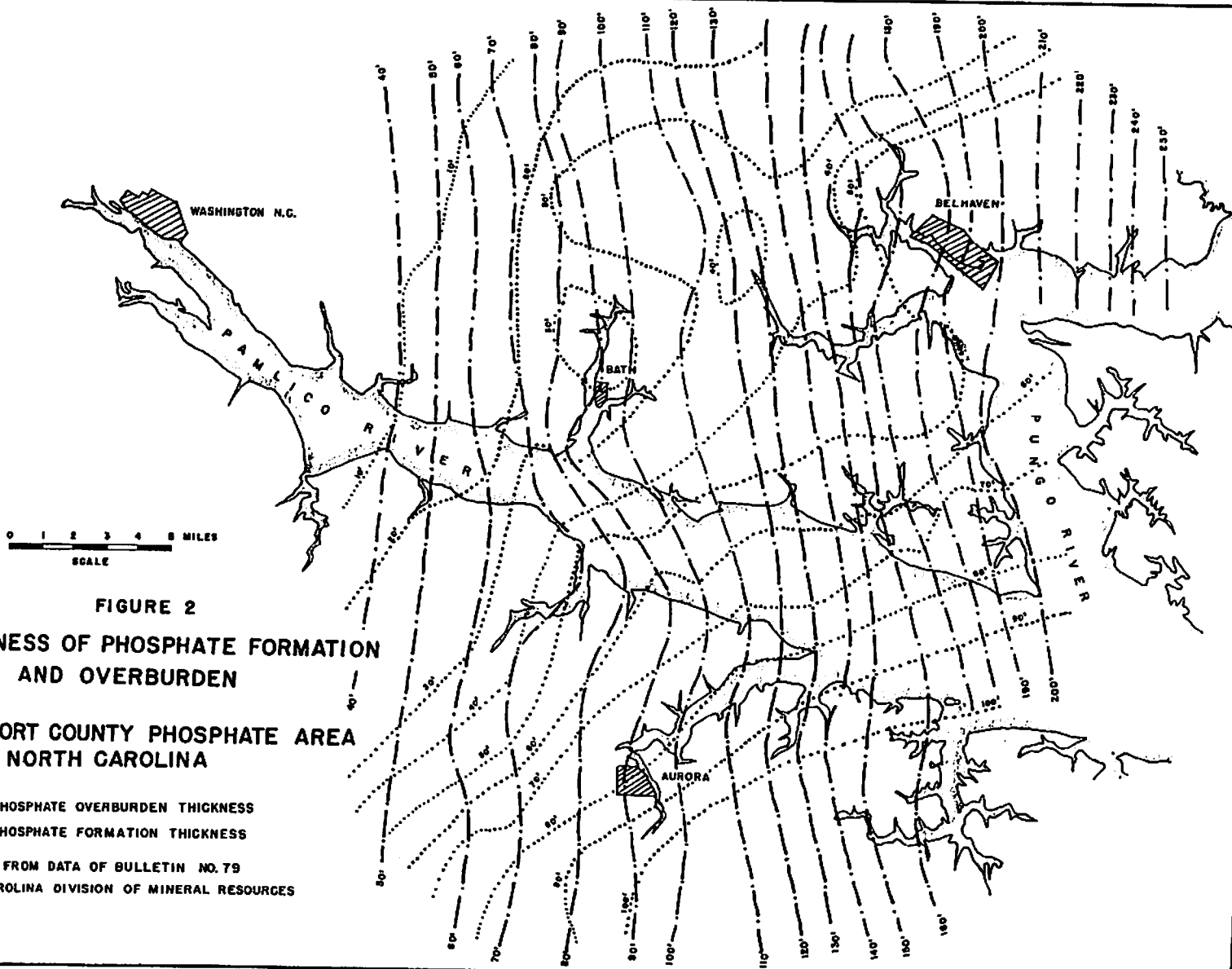
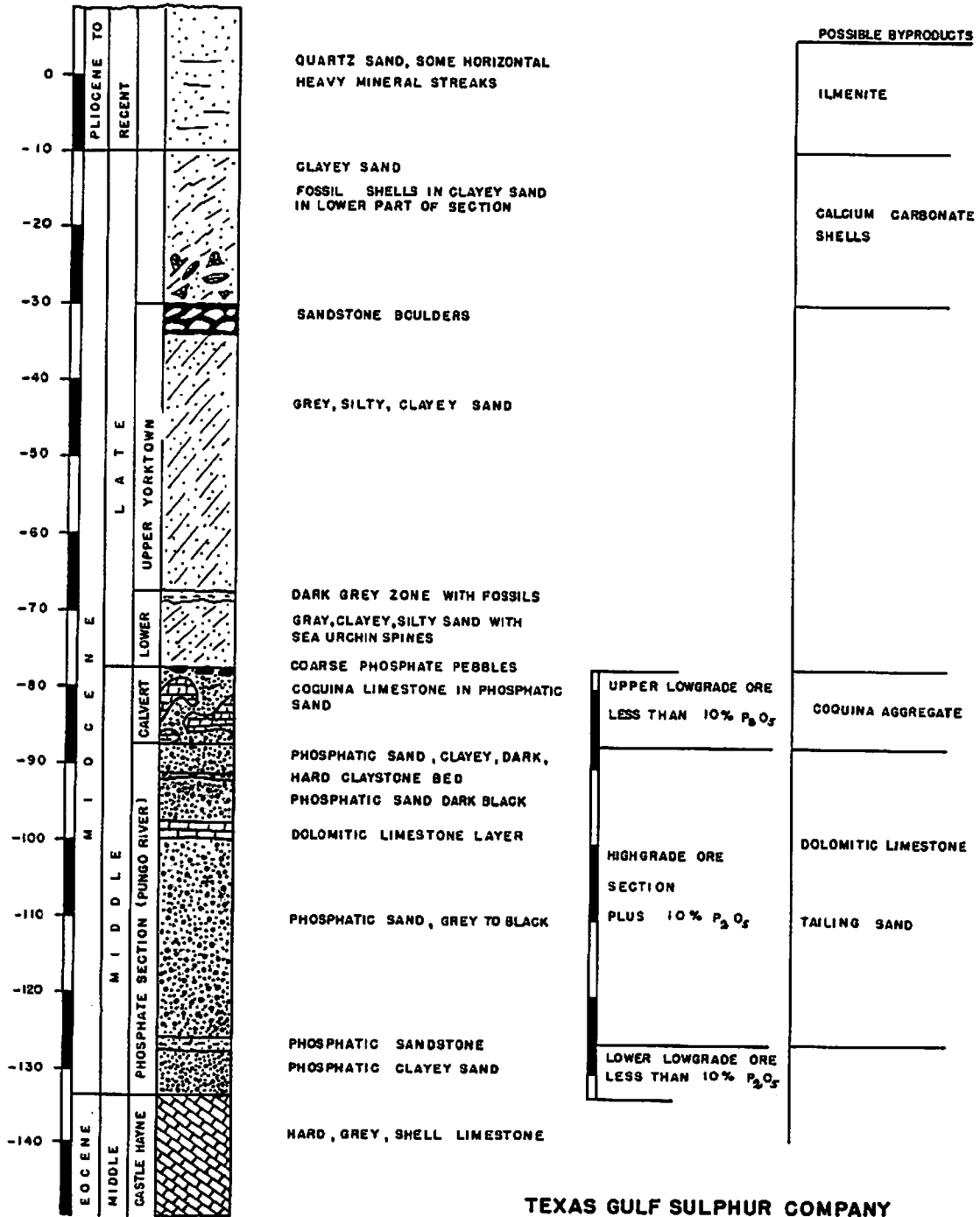


FIGURE 3
GEOLOGIC SECTION N.C. PHOSPHATE AREA



TEXAS GULF SULPHUR COMPANY
LEE CREEK TEST PIT JANUARY 1964

FIGURE 4
TEXAS GULF SULPHUR COMPANY PHOSPHATE
PILOTPLANT AT ASHEVILLE MARCH 1963

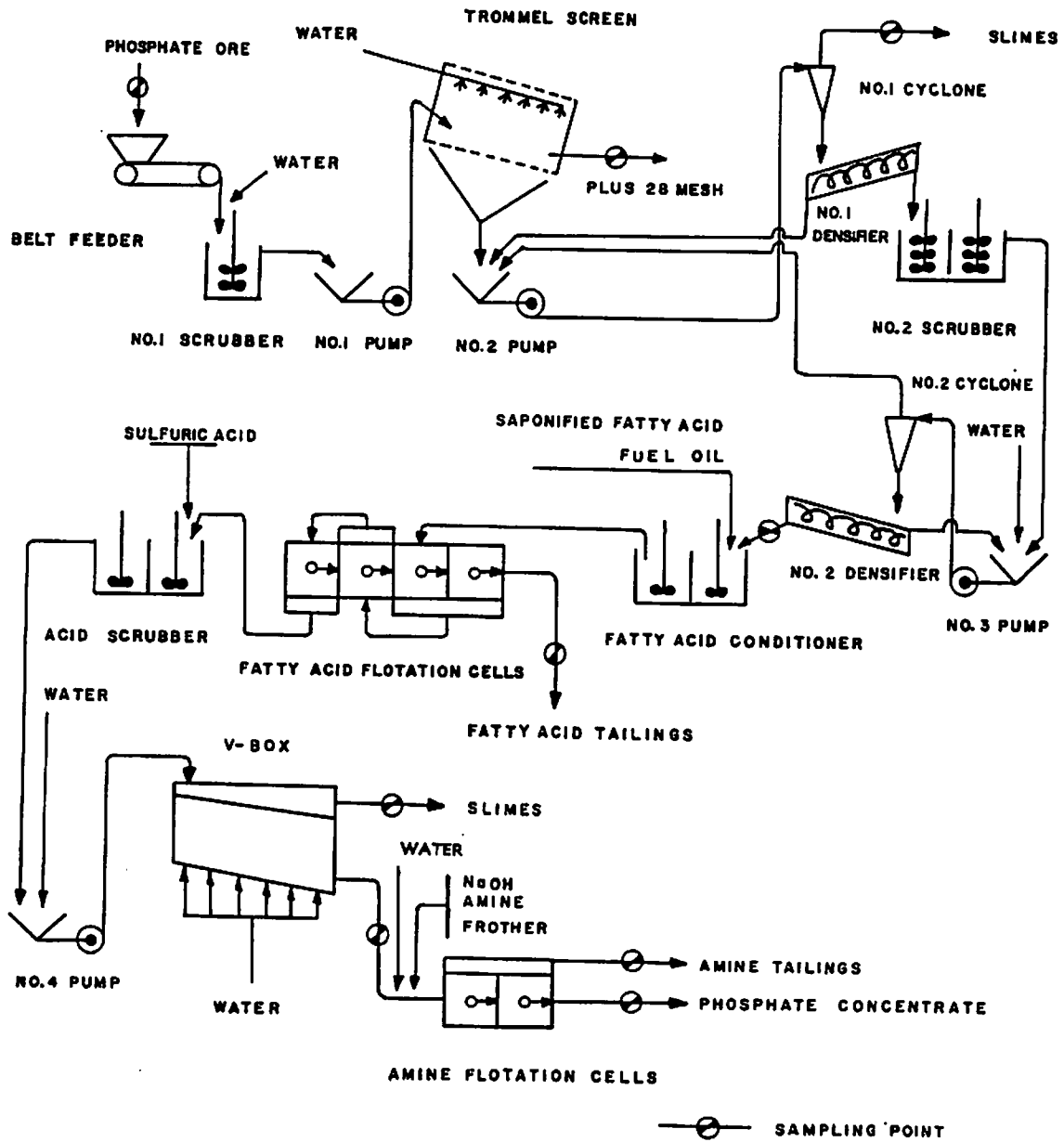
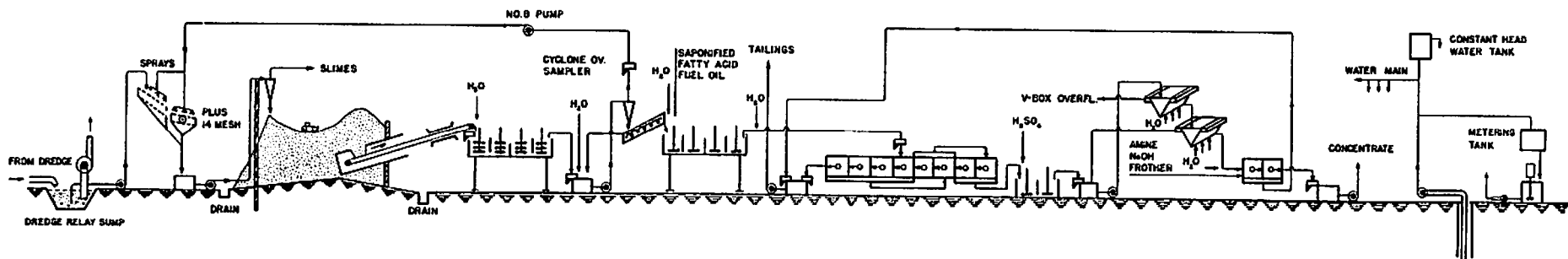


FIGURE 5

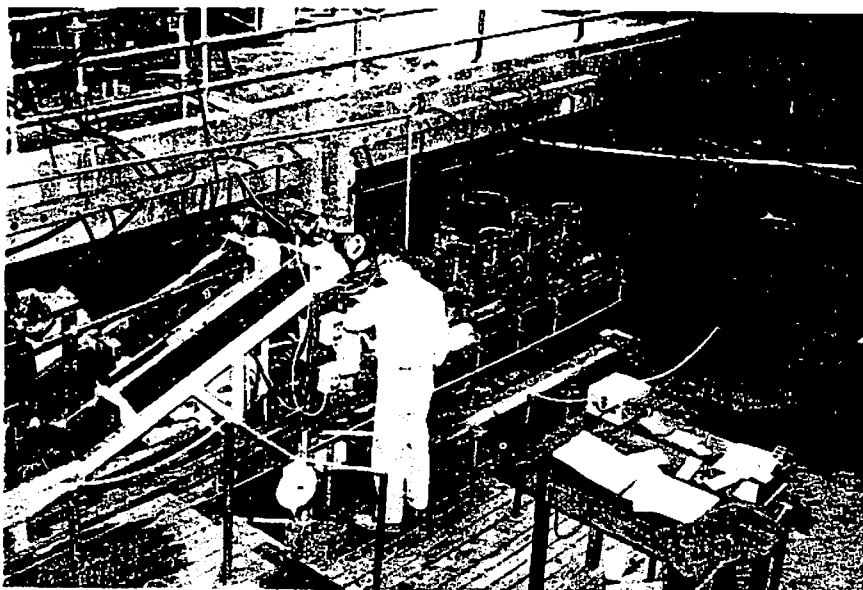
SUGGESTED FLOWSHEET FOR TEXAS GULF SULPHUR COMPANY PHOSPHATE PILOTPLANT AT LEE CREEK



	NO. 1 PUMP	STATIONARY SCREEN 1/2 INCH 14 MESH	VIBRATING SCREEN 1/2 INCH 14 MESH	H ₂ O	NO. 2 PUMP	NO. 1 CYCLONE OV. UF.	BELT/TUNNEL	WEIGHT-METER	H ₂ O	SCRUBBER	H ₂ O	NO. 3 PUMP	NO. 2 CYCLONE OV. UF.	SCREW DENSIFIER OV. UF.	H ₂ O	FATTY ACID CONDITIONER	H ₂ O	NO. 4 PUMP TAILINGS	FATTY ACID FLOTATION MACHINE	ACID SCRUBBER	NO. 5 PUMP	H ₂ O	NO. 1 V-BOX OV. UF.	H ₂ O	NO. 2 V-BOX OV. UF.	H ₂ O	AMINE FLOTATION MACHINE	MIDDLING	NO. 6 PUMP CONCENTRATE	NO. 7 PUMP WATER	REAGENT PUMPS	REAGENT MIXING TANK	
TPH DRY	12	—	12	0	10.8	1.6	2.7	27	0	2.7	0	2.7	0.2	25.0	25.0	25	0	11	25	1.4	14	0	0	14	0	0	14	0	1.4	0.1	1.3	0	
% SOLIDS	38	—	60	0	10	10	80	80	0	65	0	10	0.01	50	80	63	0	15	25	35	10	0	0	30	0	0	30	25	10	25	0		
GPM	96	—	—	34	410	360	0.9 CUFT/MIN	—	3	97	65	100	85	15	6.8	62.2	90	25	27	6.6	32	25	62	15	45	45	15	27	187	1.2	18	500-600	

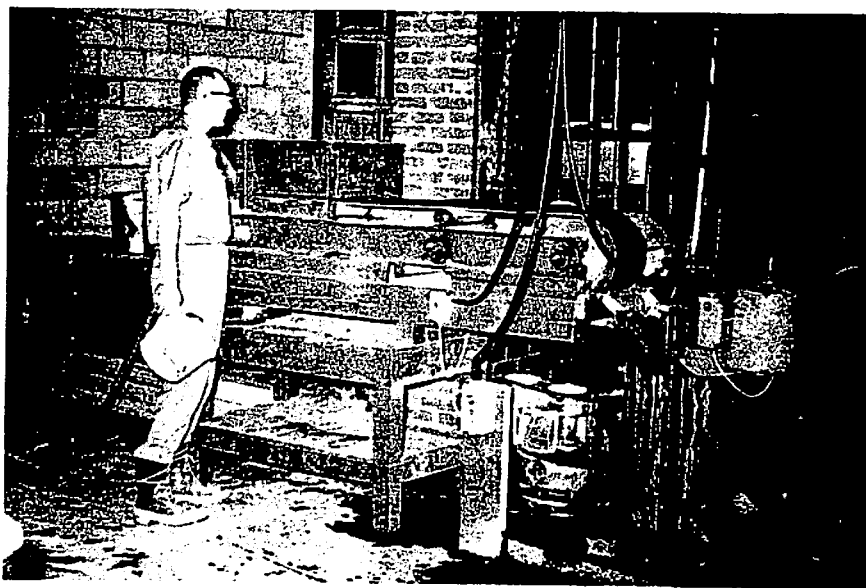
MINERALS RESEARCH LABORATORY
ASHEVILLE MAY 1963

APPENDIX
Pilot Plant Photos



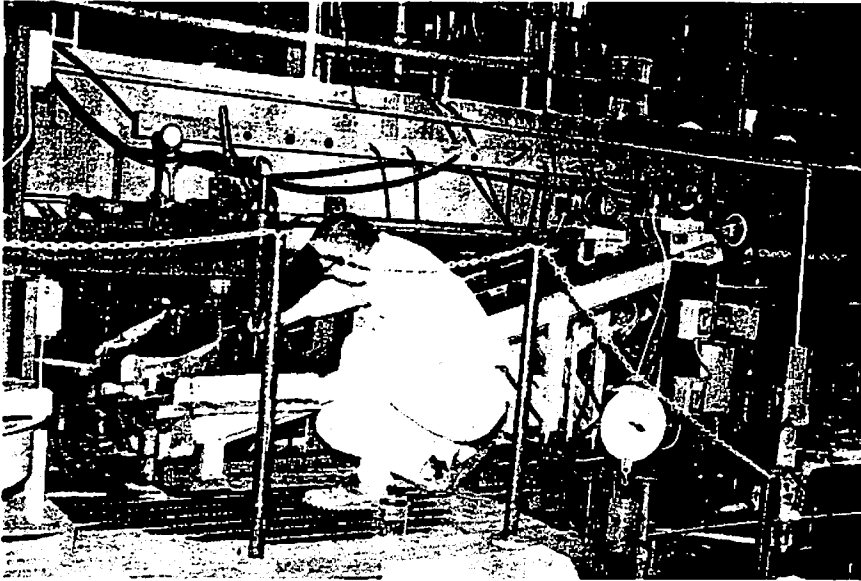
General Layout

Cyclone at left discharging into screw densifier, densifier discharging into fatty acid conditioner, conditioner discharging into flotation cells.



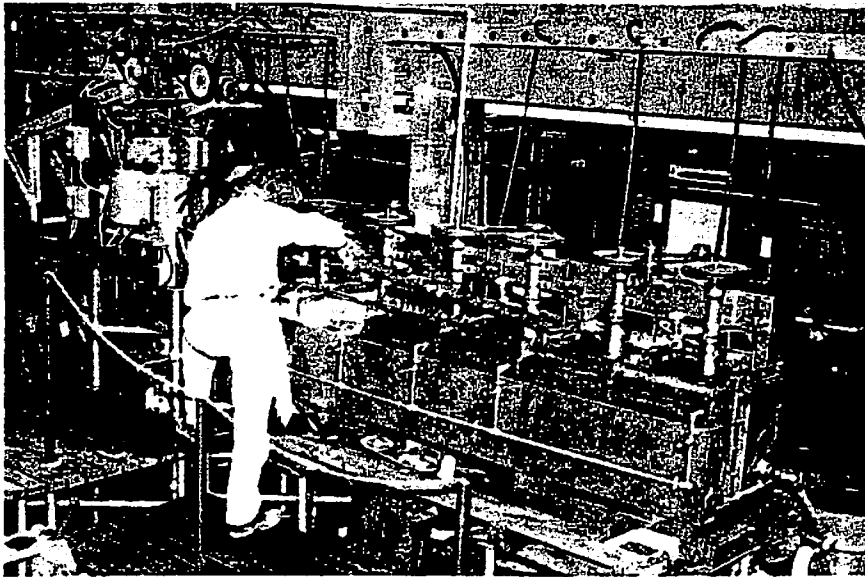
Ore Feeder

Hopper on feed belt feeding into primary scrubber and no. 1 pump.



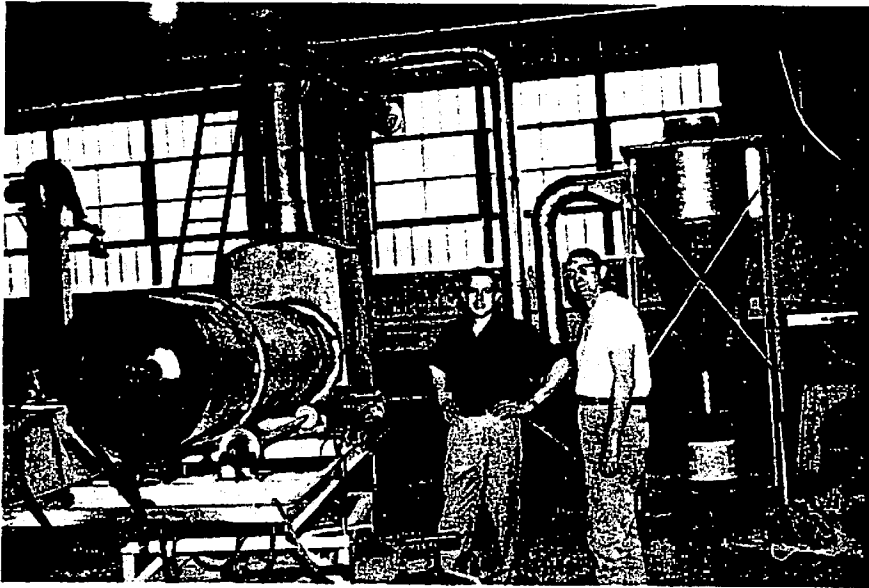
Desliming Circuit

Two cyclones at left discharging into a screw densifier. Fatty acid conditioner on right under densifier. Amine flotation launder overflowing (white froth).



Flotation Section

No. 8 Denver flotation machine in center. Fatty Acid flotation and cleaner cells at right (dark froth). Amine flotation cells at left (white froth). Acid scrubber under flotation cell.



Phosphate Drying
Rotary dryer, oil fired-indirect.

Pilot Plant Equipment Description

Belt Feeder

Denver Equipment Company, 7 feet long, 12 inches wide, speed 2.5 feet per minute, capacity 500 pounds per hour, V-belt, gear reducer and chain drive, $\frac{1}{2}$ hp single phase motor 115/230 volts, 1725 rpm.

#1 Scrubber

Denver Equipment Company, 18 by 24-inch pilot plant conditioner, 11.3, 18.7 and 24.6 gallons capacity, with stand pipe, 4-bladed 9-inch impeller, 400 rpm, V-belt drive, 2 hp single phase motor 115/230 volts, 1715 rpm.

#1 Pump

Denver Equipment Company, 1-inch vertical, 1600 rpm, 10.5 gpm H₂O, V-belt drive, $\frac{3}{4}$ hp 3-phase motor 220/440 volts, 1725/1425 rpm.

Trommel Screen

28 mesh Tyler, Laboratory designed, 24-inch diameter, 36 inches long, 23 rpm, with 6 water sprays, V-belt drive from motor reducer, $\frac{1}{2}$ hp single-phase motor 115/230 volts, 1725/36 rpm.

#2 Pump

Nagle, 1 $\frac{1}{2}$ -inch horizontal, 1800 rpm, 12.6 gpm, V-belt drive, 5 hp 3-phase motor 220/440 volts, 3500 rpm.

#1 Cyclone

Dorr Company, 3-inch diameter, intake opening $\frac{3}{8}$ by $\frac{1}{2}$ -inch, vortex opening $\frac{3}{4}$ -inch diameter, intake pressure 10 psi, adjustable apex.

#1 Screw Densifier

Denver Equipment Company, 12-inch diameter, 9-inch pitch, 9.5-foot length, 12 rpm, V-belt, gear reducer drive, $\frac{1}{2}$ hp motor, 3-phase 220/440 volts, 1725 rpm.

#2 Scrubber

Laboratory-designed, 2 compartments, 3-gallon effective volume each, three 4-bladed impellers of 11-inch diameter on each shaft running at 720 rpm, V-belt drive, two $\frac{1}{2}$ hp motors, single-phase 115/230 volts, 1800 rpm.

#3 Pump

Wilfley, 1-inch horizontal, 12.5 gpm, 1200 rpm, V-belt drive, 3 hp single-phase motor 115/230 volts, 1725 rpm.

#2 Cyclone

Humphrey, 3-inch diameter, intake opening $\frac{3}{4}$ -inch diameter, vortex 1 $\frac{1}{4}$ inch diameter, apex adjustable.

#2 Screw Densifier

Denver Equipment Company, 9-inch diameter screw, 6.5-inch pitch, 9.5-foot length, 17 rpm, V-belt, gear reducer drive, $\frac{1}{2}$ hp motor, 3-phase 220/440 volts, 1740 rpm.

Fatty Acid Conditioner

Laboratory-designed, 2 compartments, 15 by 20 inches each, adjustable to 13, 16, or 21 gallons

total capacity, one 4-bladed impeller of 7-inch diameter each, running at 350 rpm, V-belt drive, 3 hp motor, 3-phase 220/440 volts, 1750 rpm.

Fatty Acid Flotation Cells

Denver Equipment Company no. 8, two rougher cells in series and two single cleaner cells, cell volume 2.75 cu.ft. each, single discharge weir control, impeller speed 820 rpm, V-belt driven in tandem by two 1½ hp single-phase motors, 115/230 volts, 1715 rpm, paddles on froth weir running at 12 rpm.

Acid Scrubber

Laboratory-designed, 2 compartments 8 by 11 inches with one 4-bladed impeller each, 5-inch diameter, running at 1100 rpm, V-belt drive, ½ hp single-phase motor 115/230 volts, 1725 rpm.

#4 Pump

Denver Equipment Company, one-inch vertical, 1000 rpm, 6.0 gpm, V-belt drive, 2 hp single-phase motor 115/230 volts, 1715 rpm.

V-Box

Laboratory-designed, 10 inches wide, 30 inches long, 18-21-inches deep, 15 controlled ¼-inch water inlets, one wall transparent for teeter control, discharge controlled by reducing plugs.

Amine Flotation Cells

Denver Equipment Company no. 8, two cells in series, cell volume 2.75 cu.ft. each, single discharge weir control, impeller speed 820 rpm, V-belt driven in tandem by one 1½ hp single-phase motor, 115/230 volts, 1715 rpm, paddles on froth weir running at 12 rpm.

Reagent Feeder

Denver Equipment Company, two double units, 10 cups each, disk running at 4 rpm, belt, gear reducer drive, 1/20 hp single-phase motor 150 volts, 1725 rpm. Reagent feeders were fed from head tank through float valves for constant level control.

Water System

- 1 Totalizing flow meter
- 1 Flowrator, 11.5 gpm maximum
- 1 Flowrator, 5.0 gpm maximum
- 6 Flowrators, 3.5 gpm maximum
- 2 Flowrators, 2.0 gpm maximum
- 2 Flowrators, 1,600 cc/min. maximum

The water system was fed from 160 psi Asheville city water supply.