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Producing Synthetic Rutile From Ilmenite by Pyrometallurgy

Pilot-Plant Studies and Economic Evaluation

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Producing Synthetic Rutile From Ilmenite by Pyrometallurgy

Pilot-Plant Studies and Economic Evaluation

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PRODUCING SYNTHETIC RUTILE FROM ILMENITE BY PYROMETALLURGY

Pilot-Plant Studies and Economic Evalution

by

G. W. Elger,[1] D. E. Kirby,[2] and S. C. Rhoads[1]

ABSTRACT

This report describes pilot-plant studies and an economic evaluation of a process developed by the Federal Bureau of Mines, Albany Metallurgy Research Center, to produce synthetic rutile from a rock-type ilmenite concentrate. In this process, ilmenite blended with coke and lime is smelted in an electric arc furnace to separate most of the iron as salable pig iron and to form a titania-enriched slag of low iron content, which is treated with oxygen and titanium pyrophosphate. The titanium oxides are converted to crystalline rutile; the phosphate glass contains most of the associated impurities. The treated slag is ground and leached with sulfuric acid to liberate rutile crystals, which are concentrated by physical methods.

In pilot testing conducted to obtain data for a cost estimation, a synthesized product containing about 88 wt-pct TiO_2 and less than 2 pct FeO was extracted from a rock-type ilmenite concentrate containing about 45 wt-pct TiO_2.

The estimated fixed capital cost (on a mid-1975 basis) of a plant producing 500 tpd of synthetic rutile is $28,290,100. Based on 330 operating days per year, the estimated operating cost for this process is $351 per ton of synthetic rutile. The estimated selling price of synthetic rutile made from rock-type ilmenite is $344 per ton, based on a credit of $160 per ton of pig iron and a 20 pct interest rate of return on investment after taxes.

INTRODUCTION

Research performed by the Federal Bureau of Mines on titanium extraction is directed toward developing technology for beneficiating domestic ilmenite to produce a substitute for imported natural rutile with a minimum amount of water pollution. This report discusses smelting of ilmenite and synthesis of rutile carried out in pilot plant equipment and includes an economic evaluation of the recovery of rutile from a rock-type ilmenite concentrate.

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[1]Research chemist.
[2]Metallurgist.

Ores containing titanium, principally rutile (TiO_2) and ilmenite ($FeTiO_3$), are widely distributed in many parts of the world. The United States has abundant reserves of ilmenite and titaniferous magnetite minerals, but is dependent on Australia for most of its rutile used in producing TiO_2 pigment, welding rod coatings, and titanium metal.

In the United States, TiO_2 pigments are manufactured by either the sulfate or the chloride process. In the sulfate process, titania-enriched smelter slag or ilmenite concentrates are dissolved in sulfuric acid followed by separation of the iron sulfate and subsequent hydrolysis of titanium oxides, which are calcined to TiO_2. Because of the high iron oxide content in the feedstock, the sulfate route produces large quantities of byproduct iron salts and acid wastes, which may cause serious environmental problems when discarded in the ocean and other waterways (3).[3]

The newer chloride process accounts for about half of the domestic TiO_2 production and offers fewer potential environmental problems. It is generally based on the reaction of rutile-coke mixtures with chlorine in fluid-bed reactors to produce titanium tetrachloride, which is oxidized to TiO_2 or reduced to sponge metal by reaction with sodium or magnesium. Presently, one major domestic TiO_2 pigment producer uses high-grade ilmenite concentrates as feed material for chlorination. The feedstock consists of mixed altered titanium minerals containing approximately 65 wt-pct TiO_2 and is essentially free of the troublesome impurity oxides, which cause problems with operation of fluid-bed reactors. However, a pollution problem exists in the disposal of large quantities of coproduct iron chlorides produced by the direct chlorination of feedstock containing nonrutile minerals, principally ilmenite.

Increased consumption of rutile in pigment manufacture by the chloride process has placed increased demands on the limited reserves of rutile, thereby driving prices up. This has stimulated worldwide interest in upgrading ilmenite to produce a substitute for natural rutile that is acceptable feedstock to fluid-bed chlorinators (6). Efforts are mainly directed toward separating the iron from the less abundant high-quality ilmenite concentrates to produce a synthetic rutile and a usable iron oxide byproduct (2, 9). Most of the upgrading processes require a thermal reduction and/or oxidation step prior to leaching. Besides acid leaching (9), other ilmenite upgrading processes include selective chlorination (2, 6), direct chlorination, sulfidization (8), and electric smelting (14).

Quebec Iron and Titanium Corp., Sorel, Quebec (10), employs electric smelting of ilmenite mixed with coal to separate most of the iron as pig iron and prepare titania-enriched slag containing about 72 wt-pct TiO_2, 13 wt-pct FeO, and other oxide impurities. The titania smelter slag is used mainly as feedstock to sulfate-process plants. It is unacceptable for direct chlorination because some of the associated metal oxide impurities form liquid chlorides, which accumulate and cause defluidization of the bed.

[3]Underlined numbers in parentheses refer to items in the list of references preceding the appendix.

The Albany Metallurgy Research Center has developed two pyrometallurgical approaches for extracting rutile substitutes from titanium minerals. In one approach, perovskite or ilmenite concentrate blended with coke and lime is smelted to form titanium-calcium carbides. Reaction of the carbides with water liberates titanium carbide, which is chlorinated (7). The second approach entails the extraction of synthetic rutile from high-titania slag produced by smelting ilmenite. Iron is separated as a valuable pig iron coproduct. The high-titania slag is oxidized and fluxed with titanium pyrophosphate additive to form discrete rutile crystals, which are separated from the glassy matrix by leaching. Previous results obtained in laboratory tests show that high-quality synthetic rutile containing 88 to 97 wt-pct TiO_2 can be extracted from ilmenite concentrates containing 33 or 45 wt-pct TiO_2 (4). More than 90 percent of the titanium was extracted in a synthetically prepared feedstock acceptable for chlorination (5).

ACKNOWLEDGMENTS

The authors wish to acknowledge the cost evaluation studies performed by Frank A. Peters and his process evaluation group at the Bureau of Mines College Park Metallurgy Research Center, College Park, Md. Special recognition should be given to John F. Hogan, chemical engineer, who performed the cost study and prepared the tables showing capital costs, material balance, and daily thermal and utility requirements.

ILMENITE STARTING MATERIAL

The ilmenite concentrate treated in pilot-plant equipment to extract synthetic rutile was obtained from a massive rock-type deposit located near Tahawus, N.Y. Ilmenite concentrates from this deposit are normally used as feedstock to sulfate-process pigment plants. Rock-type ilmenite concentrate was selected for testing because it was representative of low-grade material available from one extensive domestic deposit. The less abundant, high-quality ilmenite concentrates are also amenable to rutile synthesis via the slag route, but they are more valuable as feedstocks for direct chlorination purposes.

The sample of ilmenite concentrate received from the Tahawus mine contained approximately 45 wt-pct TiO_2, along with iron oxides, alumina, silica, and magnesia as major impurities as shown by the chemical analysis given in table 1.

Mineralogical examination of a sample revealed that the ilmenite grains were intergrown with magnetite with small amounts of the accessory minerals, garnet, pyroxene, feldspar, and trace amounts of pyrite and calcite. Table 2 lists the size distribution of Tahawus ilmenite concentrate. Approximately 66 wt-pct of the material was plus 200 mesh in size.

TABLE 1. - Composition of Tahawus ilmenite concentrate

Constituents	Weight-percent
TiO_2	45.2
FeO	37.5
Fe_2O_3	7.1
MnO	.2
SiO_2	3.7
Al_2O_3	2.1
CaO	.1
MgO	2.6
S	.3
C	.02

TABLE 2. - Screen analyses of Tahawus ilmenite concentrate

Mesh range	Weight-percent
Minus 20, plus 35	0.2
Minus 35, plus 48	2.3
Minus 48, plus 65	7.7
Minus 65, plus 100	20.7
Minus 100, plus 150	16.8
Minus 150, plus 200	17.9
Minus 200, plus 325	16.9
Minus 325	17.5

PILOT-PLANT STUDIES

Data from bench-scale studies were used to design procedures for synthesizing and recovering rutile from ilmenite concentrate in pilot-scale equipment. Figure 1 shows three principal steps of the mineral synthesis process. First, pelletized concentrate blended with coke and lime is smelted in an electric arc furnace to separate most of the iron oxides as a pig iron coproduct and leave TiO_2 enriched in a slag fraction. Most of the other metal oxide impurities present in the ilmenite charge transfer to the slag fraction, which contains reduced titanium oxides that form a pseudobrookite-type structure (orthorhombic oxides) under a low oxygen partial pressure (4). In the second step, molten slag is tapped from the furnace, blown with oxygen, and treated with titanium pyrophosphate. The slag can be treated with titanium pyrophosphate before oxidation as was done in preliminary laboratory studies. Titanium pyrophosphate reacts with the slag impurities to form a phosphate glass matrix. Rutile is the principal titanium-bearing phase to crystallize in oxidized slag (4). In the final step, the treated slag is leached and attritioned in dilute sulfuric acid to separate the matrix, thus liberating the rutile crystals which are then concentrated by physical methods.

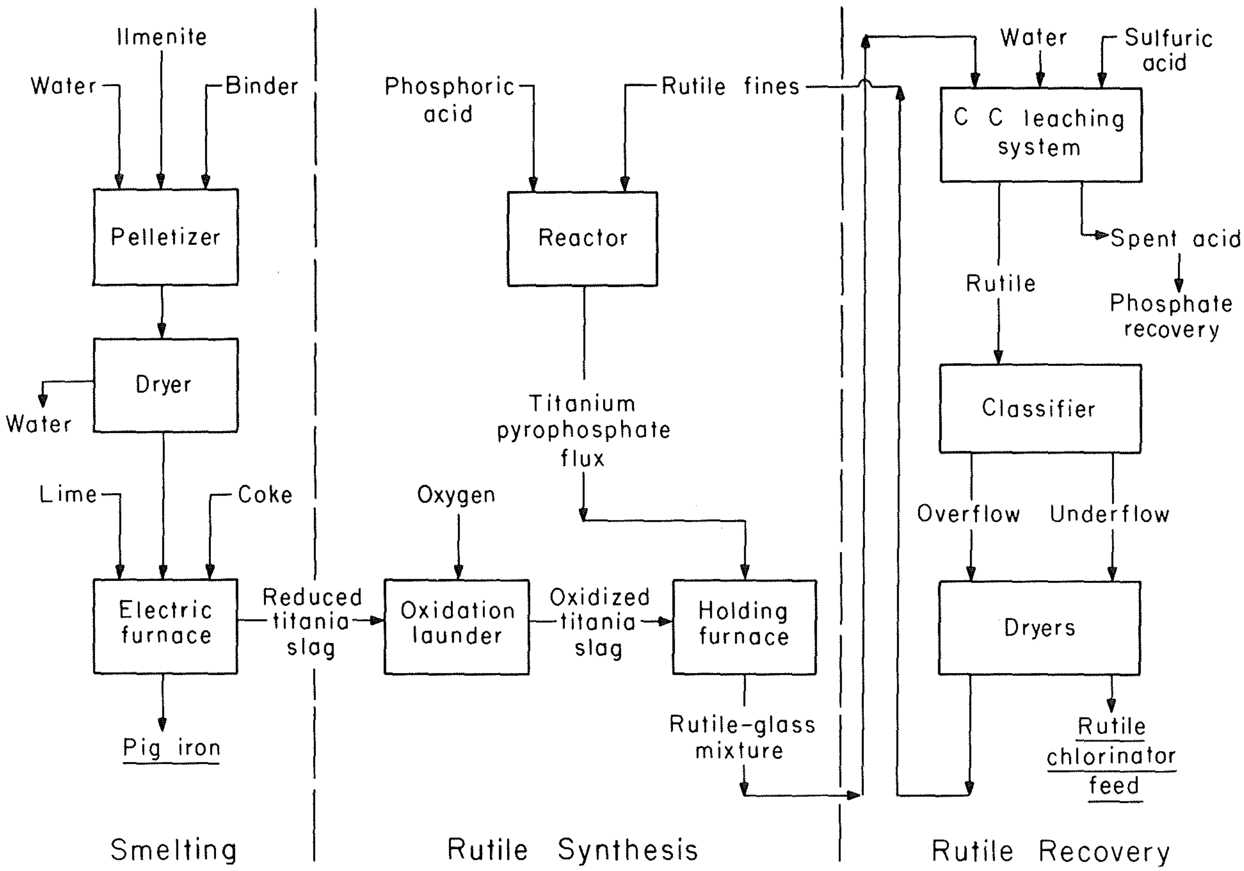

FIGURE 1. - Rutile from ilmenite.

Ilmenite Smelting

Smelting ilmenite in pilot tests was carried out in a single-phase furnace and rated at 500-lb steelmaking capacity. The furnace shell had been modified by removing the combination charging door and tapping spout and enclosing the resulting space with steelplate. Two tapholes were provided, one at hearth level for tapping molten iron and the other 3 inches above the hearth for tapping slag. The furnace was lined with 9 inches of magnesia brick next to the shell and 9 inches of carbon brick inside the magnesia brick. The hearth and sidewall were lined with rammed carbon material. The inside diameter of the furnace was 18 inches and the depth was 27 inches.

The furnace cover consisted of a rammed high-alumina refractory, 8 inches thick. The cover was provided with four holes; two for electrodes, one for feeding charge, and one for venting carbon monoxide. The two graphite electrodes were 3 inches in diameter and were operated manually. The charge material was stored in a hopper on the upper furnace deck and was discharged onto a belt feeder that regulated the flow to a chute extending downward through the furnace cover.

In the smelting step, coke reductant selectively reacts with iron oxides in ilmenite to separate iron and leave the titanium oxides enriched in a slag of low-iron content (3 to 5 wt-pct FeO) essential for rutile synthesis. Reductant requirement was based on the reactions

$$Fe_2O_3 + 3C \rightleftarrows 2Fe + 3CO \tag{1}$$

and

$$FeO + C \rightarrow Fe + CO, \tag{2}$$

and on saturation of the iron product with 3.5 to 4 wt-pct carbon. Some carbon is consumed in reducing part of the TiO_2, but it is not included in reductant calculations. Carbon additions were held to the range of 105 to 115 pct of stoichiometric requirements. Some additional carbon was furnished by the carbon liner inside the furnace.

In actual practice, a slag skull would probably form a sidewall liner, and a higher carbon addition equivalent to 125 to 135 percent of stoichiometric requirements would be necessary.

For the smelting tests, ilmenite concentrate blended with 2 wt-pct bentonite (binder) was agglomerated into pellets approximately 1/2 inch in diameter on a disk pelletizer 5 feet in diameter that was rotated at 30 rpm. Water equivalent to 15 wt-pct of the ilmenite charged was added to blended constituents during pelletization. The pelletized ilmenite was dried in an oven at 140° C for 24 hours.

The charge consisted of 340 lb of pelletized ilmenite blended with 37.2 lb of coke (minus 1/4 inch, plus 20 mesh) and 12.5 lb of metallurgical pebble lime. The CaO content of the lime addition, equivalent to 3.5 wt-pct of the ilmenite charged, fluxed the low-iron titania slag smelted at approximately 1,600° C. High titania slags of low-iron content have been produced by the Bureau without using a lime additive. Higher operating temperatures of 1,675° to 1,725° C were necessary, however, which required higher power input levels and resulted in increased refractory consumption and operating costs.

Before starting the actual smelting tests, the melting of about 150 lb of iron scrap under a slag cover of 15 lb each of lime and quartz preheated the furnace to a maximum temperature of about 1,675° C. After approximately 2 hours, the wash heat was tapped from the furnace and discarded. To initiate smelting, about a 20-lb charge was added to cover the hearth before arcing was resumed, and a molten pool was formed. The charge was then fed into the pool at the rate of 2.5 to 3.6 lb min^{-1}. The balance between charging rate and power input level was critical to minimize frothing or foaming of the bath (14). Although the arc furnace transformer of 1,000-kva capacity was operated at the lowest tap setting, the power input level of 165 to 225 kw was too high for optimum smelting. Previous investigators (14) had established that this size furnace should operate at 100 kw when smelting rock-type ilmenite.

During smelting, the FeO content of the slag was monitored by means of bath samples taken at 15-min intervals and analyzed by means of an optical-emission technique. The FeO content in the slag ranged between 1.3 and 2.5 wt-pct during feeding of the charge. Generally, the desired 3- to 5-wt-pct-FeO level in the slag was not obtained because of the high smelting temperature. Laboratory studies had shown the slag with this range of iron content to be the most suitable for rutile synthesis. Attempts were made to increase the ilmenite charging rate to 4 or 5 lb min^{-1}, but the bath foamed excessively owing to unknown circumstances. Decreasing the proportion of coke in the charge failed to lower the rate of iron removal from the slag fraction because of the carbon liner. These problems led to formation of a slag containing a higher percentage of Ti_2O_3 than in the slag product of tests made in a smaller sized furnace operated at a lower power input level (table 3). Table 4 shows the composition of the iron product made from Tahawus ilmenite smelted in a pilot furnace.

TABLE 3. - Composition of reduced titania slag made from Tahawus ilmenite, weight-percent

Series	TiO_2	Ti_2O_3	FeO	Fe°	SiO_2	MgO	CaO	Al_2O_3	MnO	C
1[1]	19.3	54.0	2.5	0.6	5.4	5.6	6.9	5.2	0.05	0.6
2[2]	46.0	20.4	2.7	.8	6.0	4.5	5.1	4.4	.5	1.6

[1]Tests performed in a pilot-scale furnace operated at a power input level of 160 to 225 kw while smelting 350-lb charges of Tahawus ilmenite blended with coke and lime.
[2]Tests performed in a small laboratory furnace operated at a power input level of 45 to 52 kw while smelting 100- to 125-lb charges of Tahawus ilmenite blended with coke and lime.

TABLE 4. - Composition of pig iron made from Tahawus ilmenite

Constituents	Weight-percent
Fe	94.8
Mn	.2
Si	.8
C	3.6
Ti	.5
S	.06
P	.04

Rutile Synthesis

The degree to which titanium in the slag is converted to rutile is dependent on the iron oxide and titanium oxide contents. High-iron smelter slags, which contain 10 to 13 wt-pct FeO, did not respond to rutile synthesis effectively (12). Ideally, the optimum slag should contain between 3 and 5 wt-pct FeO, and the minimum TiO_2 content should be at least 60 wt-pct. Efforts were only partially successful in treating slag containing lower quantities of titanium oxides owing to the presence of a stable spinel structure (titanium calcium silicate).

Cooled specimens of reduced titania slag of low-iron content, a product of smelting ilmenite blended with coke and lime (2.5 to 5 wt-pct of ilmenite in charge), usually contained two titanium-bearing phases: pseudobrookite-type (60 wt-pct) and calcium titanate crystals (15 wt-pct). Other phases formed consisted of a noncrystalline glass matrix (24 wt-pct) and entrapped iron prills (1 wt-pct) (5). In the Bureau process, both titanium-bearing phases are converted to rutile: pseudobrookite by oxidation, and calcium titanate by reaction with titanium pyrophosphate.

Oxygen required to form rutile is based on the following empirical reactions:

$$Ti_2O_3 + 1/2\ O_2 \rightarrow 2\ TiO_2 \tag{3}$$

$$2FeO + 1/2\ O_2 \rightarrow Fe_2O_3 \tag{4}$$

$$2Fe^\circ + 3/2\ O_2 \rightarrow Fe_2O_3 \tag{5}$$

$$C + 1/2\ O_2 \rightarrow CO \tag{6}$$

Conversion of calcium titanate can be represented by the following empirical equations:

$$2TiP_2O_7(s) \underset{\Delta}{\rightleftarrows} 2TiO_2(s) + P_4O_{10}(g) \tag{7}$$

$$3CaTiO_3(\alpha) + 1/2(P_4O_{10})g \rightleftarrows Ca_3(PO_4)_2(\alpha,\beta) + 3TiO_2(\text{Rutile}) \tag{8}$$

and/or $$2CaTiO_3(\alpha,\beta) + 1/2P_4O_{10} \rightleftarrows Ca_2P_2O_7(\alpha,\beta,1) + 2TiO_2(\text{Rutile}) \tag{9}$$

Previous studies made in laboratory crucibles established that rutile crystals formed in oxidized slags at temperatures as low as 650° C, but that 1,500° to 1,650° C is preferred from the standpoint of increased size and purity of the recovered crystalline product (5).

In pilot studies, the method of treatment usually used was to tap molten slag at 1,600° C into a preheated launder and treat it directly with oxygen at a flow of 16 cu ft min^{-1} (fig. 2). Bath temperatures were measured by expendable immersion thermocouples. No difficulty was encountered in keeping the slag molten during oxidation.

Titanium pyrophosphate was added to the oxidized slag discharged into the holding furnace; P_2O_5 in the flux addition was equivalent to 5 pct of the estimated slag weight. The procedure for preparing titanium pyrophosphate, containing approximately 60 wt-pct P_2O_5 and 40 wt-pct TiO_2, was discussed in a previous report (5). Figure 3 shows oxygen blowing of molten slag in a launder.

The degree of slag oxidation was estimated from the Ti_2O_3 content of the slag prior to tapping and after treatment. The Ti_2O_3 content in the reduced slag averaged about 56 wt-pct and, after treatment in the launder, averaged 27 wt-pct. Residence time of the slag in the launder was only 2 to 5 seconds,

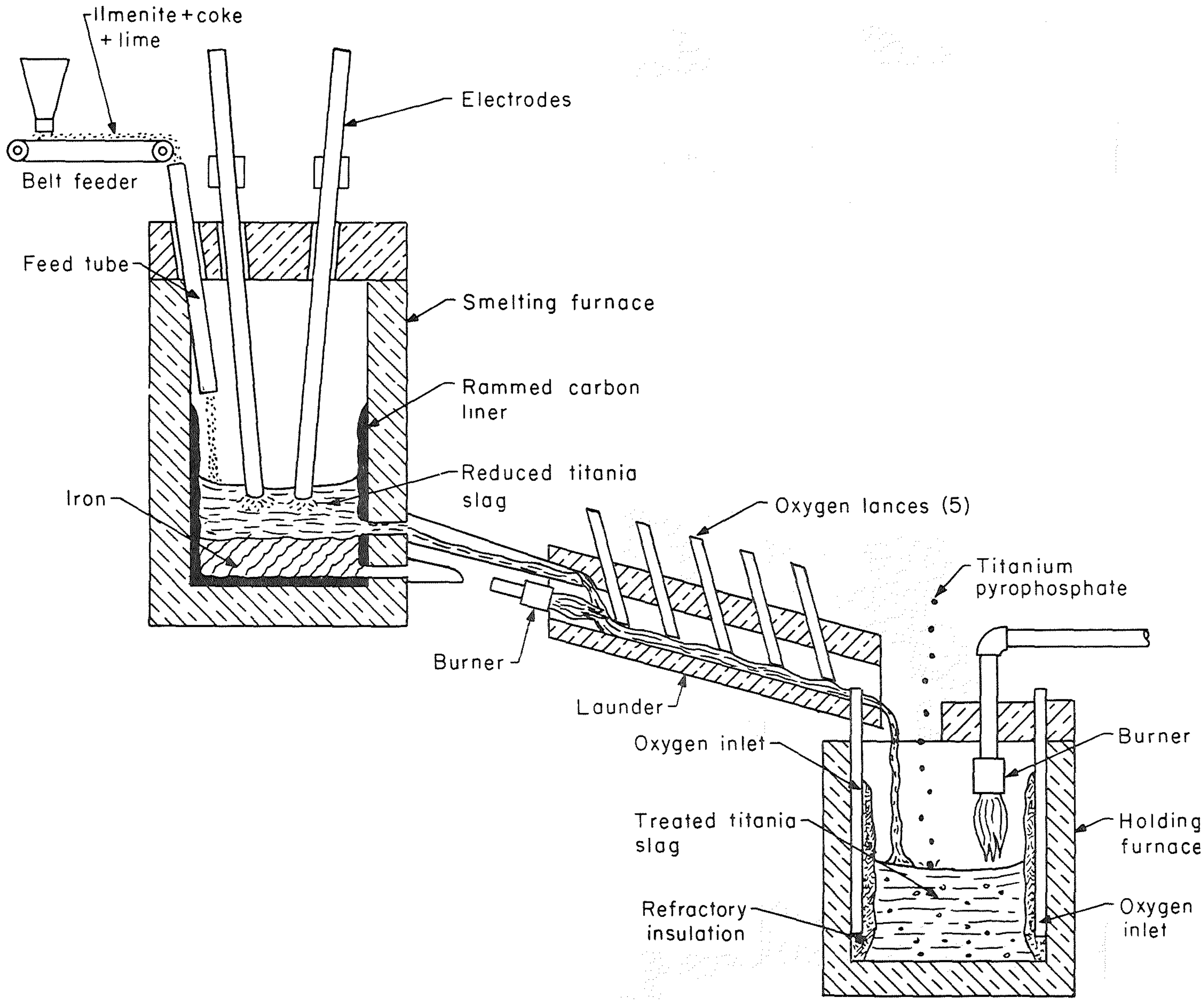

FIGURE 2. - Rutile synthesis equipment.

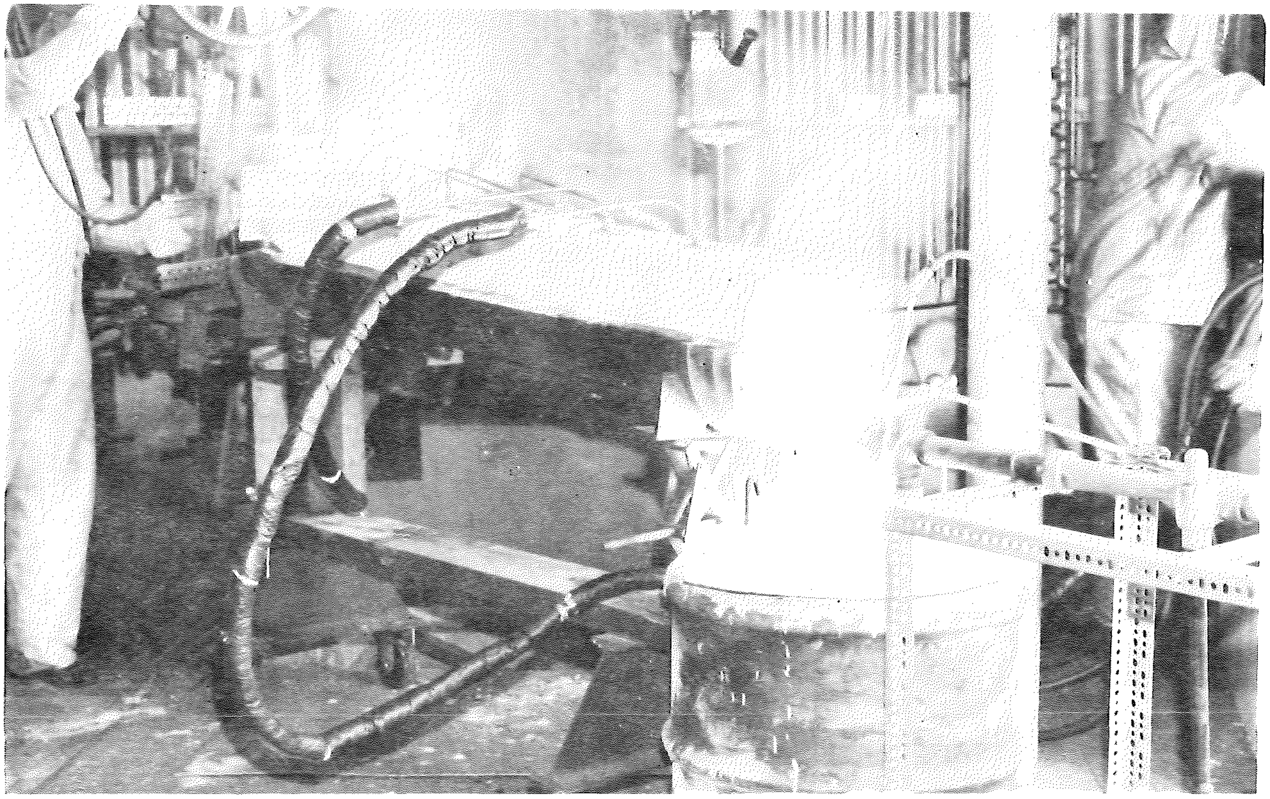

FIGURE 3. - Rutile synthesis by oxygen blowing of molten titania slag.

and more than half of the contained Ti_2O_3 was oxidized to TiO_2. The slag launder of 5 feet in length provided insufficient residence time for complete oxidation of slag. Indications are that the launder should be 25 to 50 feet in length, but limited time prevented modification of the equipment. An oxidizing flame was, therefore, impinged on the surface of the pool of slag in the holding furnace, and the Ti_2O_3 content in the slag was lowered to about 20 wt-pct. In cooled specimens of slag produced under these conditions, about 40 pct of the titanium phases were converted to rutile crystals.

Improved slag oxidation was achieved in the final test by inserting two gas spargers in the holding furnace. Two alumina tubes, each 1/2 inch in diameter and enclosed within a 1-1/2-inch thickness of phosphate-bonded alumina plastic refractory was used to inject oxygen beneath the surface of the slag pool 12 inches deep. Exit ends of the tubes were enclosed within the porous refractory and were approximately 2 inches from the bottom of the furnace. The slag was sparged with oxygen at a flow of 1 cu ft min^{-1} for 10 min. This provided excellent oxidation efficiency; the treated slag contained only 2.4 wt-pct Ti_2O_3. The slag was cooled in the holding furnace.

Figure 4 shows the microstructure of reduced titania slag and treated slag specimens from the final test. Reduced slag contained black pseudobrookite-type crystals acicular in shape that were enclosed in a glassy matrix. The treated slag contained more small (<100 μm) rutile crystals than were observed in crucible tests (fig. 5). In some tests the FeO content

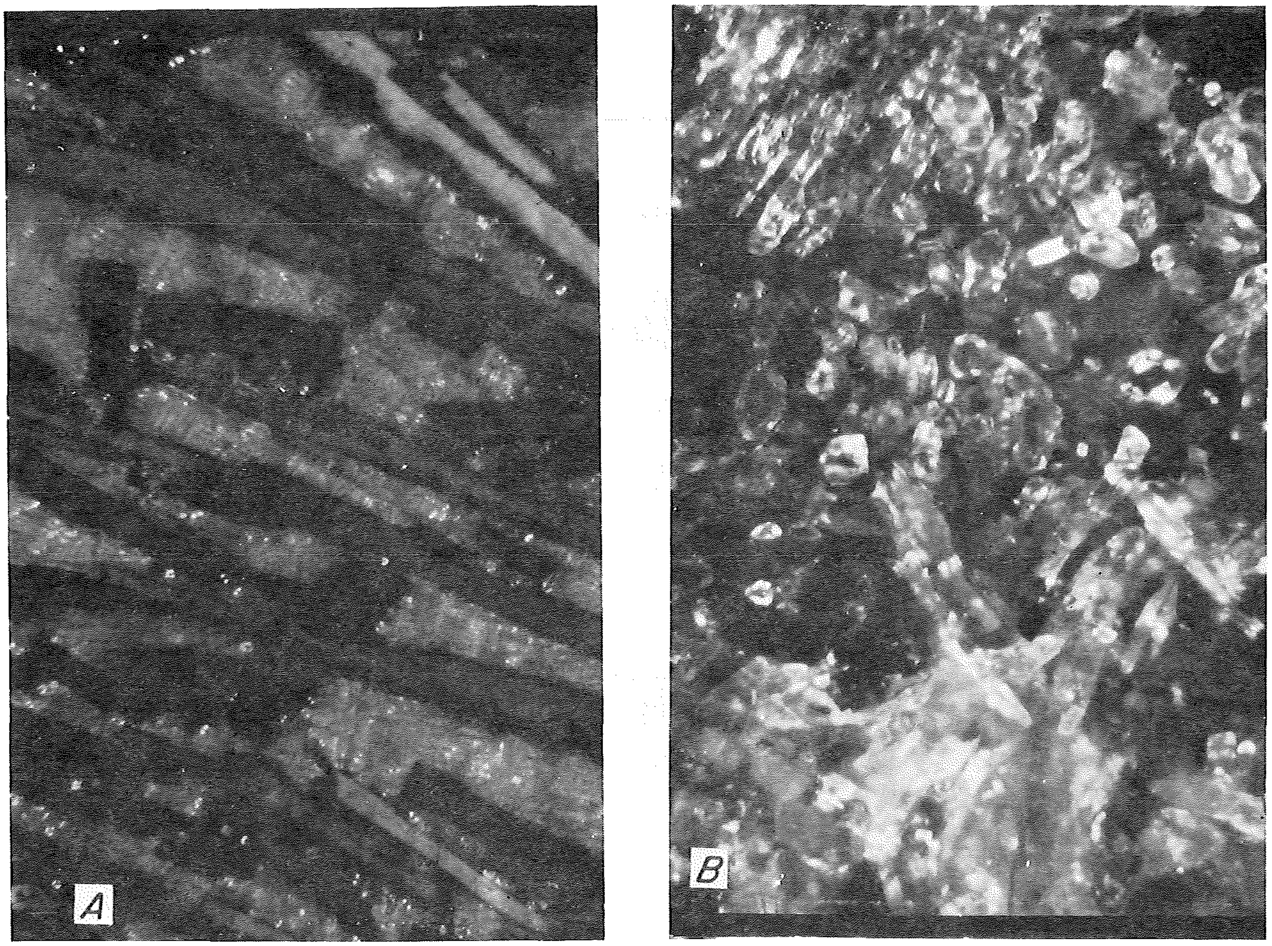

FIGURE 4. - Polished sections from a titania slag made from Tahawus ilmenite in pilot-scale equipment. *A*, Large primary crystals of a pseudobrookite-type structure in a silicate glass matrix as seen in a reduced slag; *B*, rutile crystals in a section from a slag oxidized at approximately 1,600° C and fluxed with titanium pyrophosphate, P_2O_5 equivalent to 5 pct of slag weight. Polarized light (X 160).

of the slag increased from 3.6 to 8.0 wt-pct, due to reoxidation of iron prills entrapped in the slag.

Rutile Recovery

Synthetic rutile was recovered from the glassy matrix using a procedure previously devised in bench-scale testing (5). Treated slag from pilot testing was ground to pass through a 28-mesh screen preparatory to leaching in dilute sulfuric acid. Samples of the ground slag were leached for 60 min in a 8 wt-pct sulfuric acid solution at 85° C and the resulting slurry attritioned for 5 min. Most of the glass matrix separated from the crystals by chemical dissolution; the remaining impurities were mechanically abraded from the surface of rutile crystals by attrition grinding. Only 0.2 to

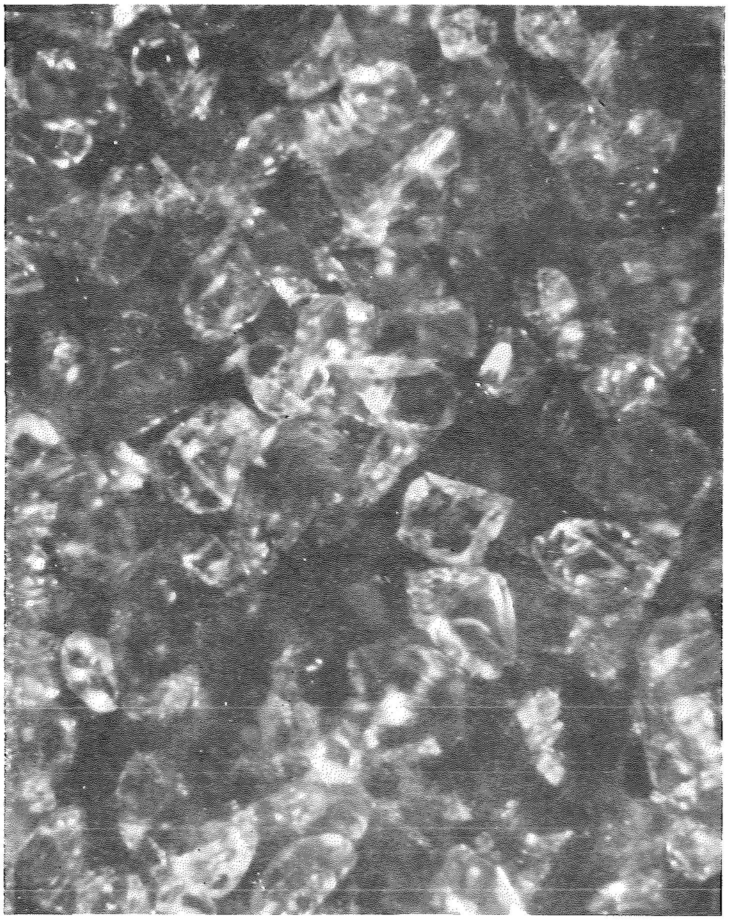

FIGURE 5. - Rutile crystals in a polished section from a slag specimen oxidized at approximately 1,430° C for 1 hour and fluxed with titanium pyrophosphate; P_2O_5 equivalent to 5 pct of slag weight. Test made in bench-scale equipment. Polarized light (X 160).

0.3 part sulfuric acid was required per part treated slag to separate most of the slag impurities. Synthesized rutile containing approximately an 88-wt-pct equivalent TiO_2 was produced, and the titanium recovered represented 88 pct of that contained in the treated slag. A partial analysis of the slag and synthetic rutile product is shown in table 5.

In pilot tests, the TiO_2 content in the synthetic rutile product was slightly lower than the product containing 92 wt-pct TiO_2 made in crucible tests in which an equivalent quantity of titanium pyrophosphate was added to slag. Slag treatment on a larger scale should result in improved oxidation efficiency through better control of slag flow to launder and increased residence time of slag oxygen-blown in a launder of sufficient length. Actually, slag oxidation may be more efficient in a transfer ladle or a converter than in a launder. Insufficient time remained in our testing program to study these alternate routes of oxidation.

TABLE 5. - Chemical analyses of treated slag and synthetic rutile product

Sample	Constituent, weight-percent					
	TiO_2	Total Fe	FeO	C	P_2O_5	Other[1]
Treated slag.....	72.3	1.5	1.6	0.08	5.5	19.0
Synthetic rutile.	87.6	1.9	(2)	(2)	.2	(2)

[1]Includes CaO, MgO, SiO_2, Al_2O_3, MnO_2.
[2]Analyses not made.

ECONOMIC EVALUATION

Proposed Process

The process evaluated in this report is based on smelting rock-type ilmenite concentrate with lime and coke to separate a pig iron byproduct and produce titania-enriched slag, which is treated further to extract rutile. The low-iron slag is treated by oxidation and fluxing with titanium pyrophosphate to remove the impurities and form crystalline rutile. Following treatment, the rutilized slag is leached in dilute acid to liberate the upgraded titanium product. The resultant slurry is filtered to extract synthetic rutile containing approximately 92 wt-pct TiO_2. The sequence of operations is shown in figure 6.

The plant described in this report is designed to produce 500 tpd of synthetic rutile. The proposed process entails four principal steps: feed preparation, smelting and slag treatment, leaching, and product recovery.

Feed Preparation

The starting material consists of a rock-type ilmenite concentrate having a particle size range of 65 to 325 mesh. Its chemical composition is shown in table 1. It is brought to the plant by truck, dumped into a hopper, and transported by belt conveyor to a storage pile.

Petroleum coke used to reduce iron oxides in ilmenite, lime, and a bentonite binder are brought into the plant by rail and dumped into hoppers. From these hoppers, the constituents are conveyed to separate storage silos.

Ilmenite concentrate and binder are conveyed from storage to a zigzag blender where they are mixed with tailings from the leaching section and dust recovered from the baghouse used to clean electric arc furnace offgases. The mixture is pelletized with a water addition in a disk agglomerator and conveyed to a rotary dryer. The fuel used in the dryer is carbon moxoxide recovered from the smelting furnace. Petroleum coke and lime are conveyed from storage to a zigzag blender where they are mixed with pelletized ilmenite from the dryer. The blended furnace charge is conveyed to 3-hour-capacity storage bins.

Smelting and Slag Treatment

The pellets are conveyed from storage to surge bins located above the three electric arc smelting furnaces. The surge-bin gravity feeds six belt conveyors, two for each furnace. The belt conveyors, located above the electric arc furnaces, which operate at 1,600° C, continuously charge the furnaces through a number of feed ports along their sides. This feeding arrangement is used to allow the charge to protect the sidewall refractories of the electric arc furnaces from the effects of the corrosive titania slag.

Every 2 hours approximately 20 tons of titania slag is tapped from each furnace into ladles containing titanium pyrophosphate. When the molten slag

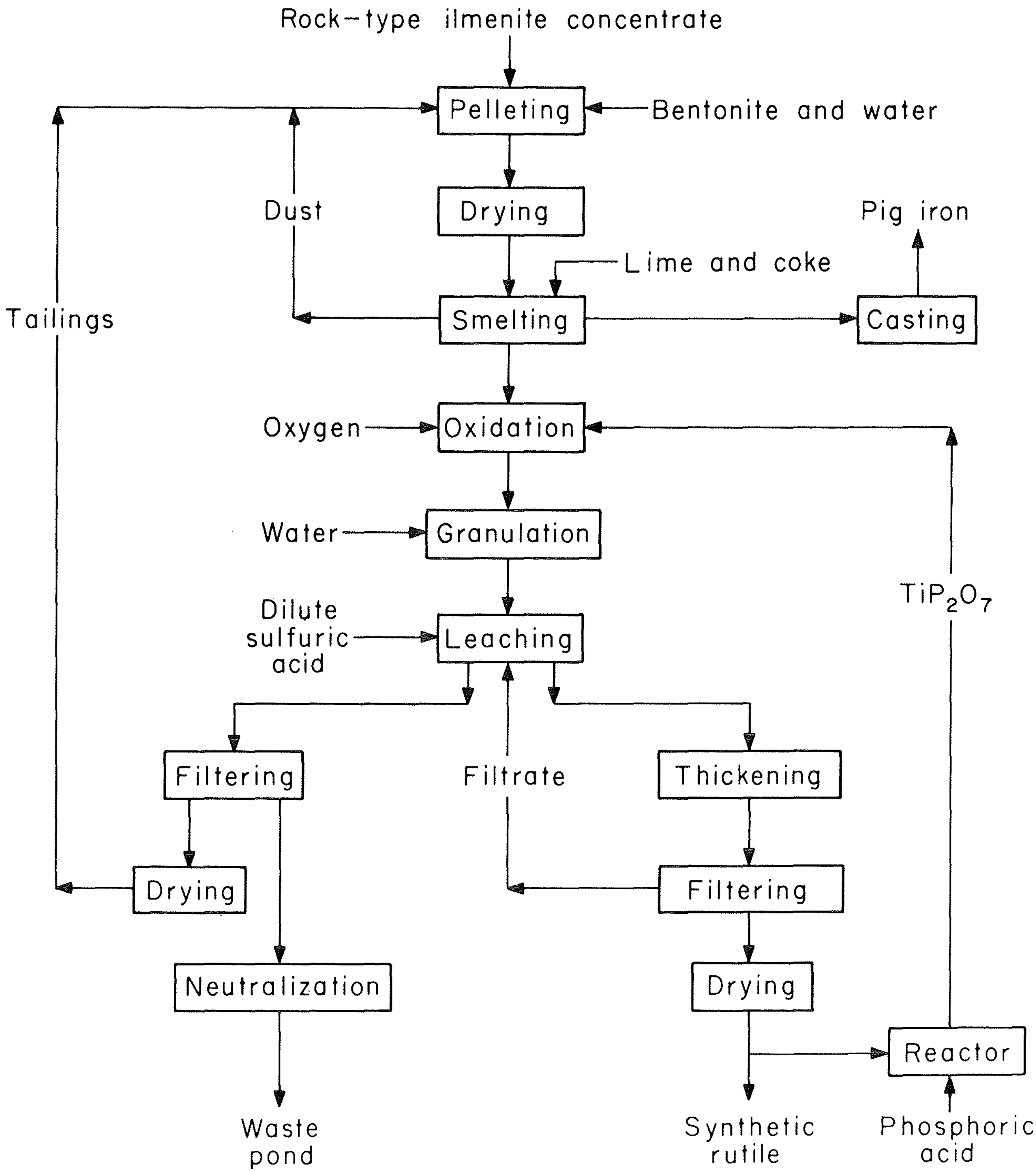

FIGURE 6. - Production of synthetic rutile from ilmenite.

is added, titanium pyrophosphate decomposes to TiO_2 and releases P_2O_5 for reaction with the associated impurities. The slag is then transferred from the ladle to a converter and blown with oxygen to convert Ti_2O_3 to TiO_2. Molten iron is tapped at 6-hour intervals from each furnace into a ladle that transports the iron to pig-casting machines. Once hardened, the 50-lb pigs are dumped by the pig-casting machine and stored before shipment.

Carbon monoxide generated during smelting is collected and used as a fuel for drying the pelletized feed to the smelting furnaces.

Leaching

Oxidized slag from the converter in the smelting and slag-treatment section is poured into a tank containing water, which quenches and granulates the slag. Laboratory tests previously established that quenching has no significant effect on rutile particle size. Indications are that TiO_2 is not very soluble in the molten phase of oxidized slag. The slurry from the tank is pumped to a rod mill, which grinds the slag to minus 25 mesh. Leaving the rod mill, the slurry is pumped to a three-stage countercurrent leaching system operating at about 70° C in which the slag is leached with dilute sulfuric acid. Approximately 1-1/2 hours are required in the agitated leach circuit to dissolve most of the impurities. (This is based on laboratory data derived from countercurrent leaching.)

Underflow from the leaching circuit is pumped to a thickener with the underflow from the thickener pumped to a rotary vacuum filter. The filter separates and washes the synthetic rutile.

Overflow from the leaching circuit and the thickener is filtered to recover the entrained solids, which are dried and conveyed to storage bins before being recycled to the feed preparation section. The filtrate, recovered from both filtering operations in this section, is neutralized with limestone and pumped to a tailings pond.

Product Recovery

Synthetic rutile from the filter in the leaching section is conveyed to a rotary dryer to remove water; the rutile is then conveyed to a 14-day capacity storage silo for bulk shipment. A portion of the synthetic rutile is withdrawn for preparation of titanium pyrophosphate. This is prepared by blending synthetic rutile with phosphoric acid and heating the mixture to 450° C in a rotary kiln. The titanium pyrophosphate produced is conveyed to the smelting and slag treatment section and used to treat the titanate slag.

Cost Analysis

The cost evaluation presented in this report is a study estimate based on published sources brought up to date by the use of inflation indices. Freight costs to the plant location are not considered. It is assumed that the plant is within short trucking distance of the ilmenite deposit.

Capital Costs

The capital cost estimate is of the general type termed "study estimate" by Weaver and Bauman (15). This type of estimate, prepared from a flowsheet and a minimum of equipment data, can be expected to be within 30 pct of the actual cost. The fixed capital cost on a mid-1975 basis is $28,290,100 for a plant designed to produce 500 tpd of synthetic rutile. The fixed capital cost for the process is shown in table 6.

TABLE 6. - Estimated capital cost[1]

Fixed capital:	
Feed preparation section	$2,317,500
Smelting and slag treatment	12,427,200
Leaching section	2,672,400
Product recovery	2,330,600
Oxygen plant	3,267,400
Steam plant	173,500
Subtotal	23,188,600
Plant facilities, 10 pct of subtotal	2,318,900
Plant utilities, 12 pct of subtotal	2,782,600
Fixed capital cost	28,290,100
Working capital:	
Raw material and supplies	3,360,200
Product and in-process inventory	4,823,300
Accounts receivable	4,823,300
Available cash	4,452,100
Working capital cost	17,458,900
Total capital cost	45,749,000

[1]Basis: M and S equipment cost index = 443.8.

Cost-capacity data of the type presented by Bauman (1), Mills (11), and Peters and Timmerhaus (13) are used as the primary sources of equipment costs. The Marshall and Swift Equipment Cost Index is used to convert the equipment costs to a mid-1975 basis. Factors for piping, instruments, and the like are assigned to each section using as a basis the effect that fluids, solids, or a combination of fluids and solids may have on each item such as piping, instruments, etc. Construction materials that withstand corrosion are also considered in assigning the factors. These factors are shown for each section in the equipment cost summary tables in the appendix.

For each section, the field indirect costs, which include field supervision, inspection, temporary construction, equipment rental, and payroll overhead, are estimated at 10 pct of the direct cost. Engineering, administration, and overhead are each estimated at 5 pct of the construction cost. A contingency of 10 pct and a contractor's fee of 5 pct are included in the cost of each section.

The cost of a steamplant is estimated from the steam requirements and is included as a section cost. Also, the oxygen is supplied by an oxygen plant, the cost of which is also included as a section cost.

Cost of plant facilities is estimated as 10 pct of the total process section costs and includes costs such as those for laboratories, shops, roads, administrative buildings, fences, and fire protection equipment. Plant utility costs, which included costs for items such as steam, water, and power distribution systems, and sewers, are estimated at 12 pct of the total process section costs. The costs for plant facilities and utilities include the same field indirect costs, engineering, and administration and overhead costs, contingency, and contractor's fees as are included in the section costs.

Working capital is defined as "the funds in addition to fixed capital, land investment, and startup costs that a company must provide to operate a plant." Working capital, also shown in table 6, is estimated from the following items: (1) raw material and supplies inventory (cost of raw materials and operating supplies for 1 month), (2) product and in-process inventory (total operating costs for 1 month), (3) accounts receivable (total operating cost for 1 month), and (4) available cash (direct expenses for 1 month).

Land investment and startup costs are not included in the process study estimate.

Operating Costs

The estimated operating cost is based on an average of 330 days of operation per year over the life of the plant, thus allowing an average of 35 days downtime per year for inspection, maintenance, and unscheduled interruptions. The operation costs are divided into direct, indirect, and fixed costs.

Direct costs include raw materials, utilities, direct labor, maintenance, payroll overhead, and operating supplies. The raw material costs do not include transportation costs since a plant location is not considered except for the assumption that the plant is within short trucking distance of the ilmenite source. Utilities include electricity, water, and natural gas, which are purchased, and steam, which is produced onsite. Raw material and utility requirements per ton of ilmenite are shown in the appendix.

The direct labor cost is estimated on the basis of manning the plant with 4.2 men for each position that operates 24 hours per day, 7 days per week and 1 man for each position that operates 8 hours per day, 5 days per week. The manning assignments are shown by section in the appendix. Cost of labor supervision is estimated at 15 pct of the operating labor cost.

Plant maintenance is separately estimated for each piece of equipment and for the building, structures, electrical system, piping, plant utility distribution system, plant facilities, etc. Material and supplies represent 40 pct of the maintenance cost, and labor represents the remaining 60 pct.

Payroll overhead, estimated as 35 pct of direct labor and maintenance labor, includes vacation, sick leave, social security, and fringe benefits. The cost of operating supplies is estimated as 20 pct of the plant maintenance cost.

Indirect costs include the expenses of control laboratories, accounting, plant protection and safety, and plant administration. Research and overall company administrative costs outside the plant are not included. Forty percent of direct labor and plant maintenance is used for estimating the indirect cost.

Fixed costs include the cost of taxes (excluding income taxes), insurance, and depreciation. Both taxes and insurance are each estimated as 1 pct of the plant construction cost; depreciation is based on a straight-line 11-year plant life.

The estimated operating cost for a plant producing 500 tons of synthetic rutile per day is $351 per ton of synthetic rutile (table 7).

TABLE 7. - Estimated annual operating cost

	Annual cost	Cost per ton synthetic rutile
Direct cost:		
Raw materials:		
Ilmenite at $60 per ton	$21,225,600	$128.64
Coke at $86 per ton	3,490,700	21.16
Ortho-phosphoric acid at $237 per ton	5,240,100	31.76
Electrodes graphite at $801 per ton	6,872,600	41.65
Lime at $18 per ton	261,400	1.58
Bentonite at $15.50 per ton	133,000	.81
Limestone at $5 per ton	198,000	1.20
Sulfuric acid at $39.70 per ton	2,594,000	15.72
Chemicals for steamplant water treatment	1,400	.01
Total	40,016,800	242.53
Utilities:		
Electric power at 1.2 cents/kwhr	8,504,100	51.54
Water, process at 10 cents/Mgal	13,500	.08
Water, raw at 2 cents/Mgal	27,500	.17
Natural gas at $1/MMBtu	327,100	1.98
Total	8,872,200	53.77
Direct labor:		
Labor at $6 per hour	1,535,000	9.30
Supervision, 15 pct of labor	230,300	1.40
Total	1,765,300	10.70
Plant maintenance:		
Labor	763,200	4.63
Supervision, 20 pct of maintenance labor	152,600	.92
Materials	610,500	3.70
Total	1,526,300	9.25
Payroll overhead, 35 pct of payroll	938,400	5.69
Operating supplies, 20 pct of plant maintenance	305,300	1.85
Total direct cost	53,424,300	323.79
Indirect cost, 40 pct of direct labor and maintenance	1,316,600	7.98
Fixed cost:		
Taxes, 1.0 pct of total plant cost	282,800	1.71
Insurance, 1.0 pct of total plant cost	282,800	1.71
Depreciation, 11-year life	2,571,100	15.58
Total operating cost	57,877,600	350.77

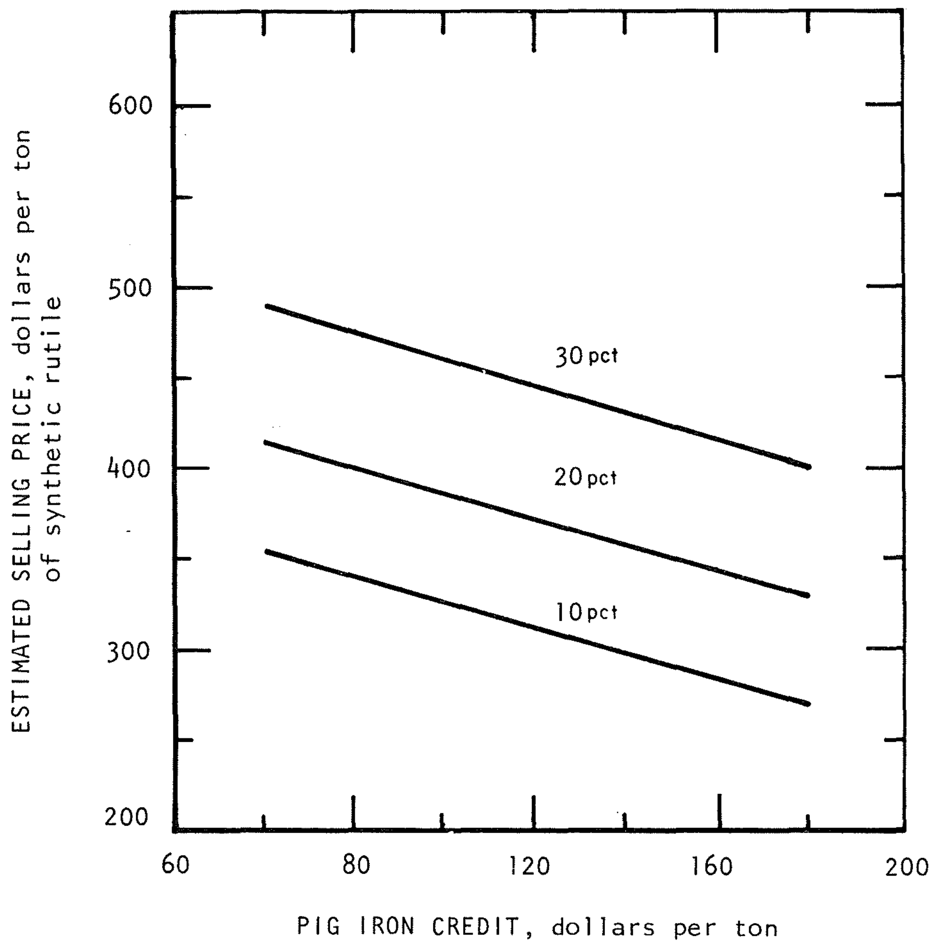

FIGURE 7. - Effect of the pig iron credit on the estimated selling price of synthetic rutile for various interest rates of return on investment after taxes.

Profitability

The synthetic rutile produced in this process is suitable for use as a feed material in the chlorination process. The synthetic rutile would be competing with rutile, which had a selling price quoted at $520 per ton in mid-1975. Figure 7 shows the estimated selling price per ton of synthetic rutile as a function of the pig iron credit for various interest rates of return on investment after taxes. From figure 7 it can be seen that the Bureau-developed process is competitive with natural rutile at its current price without the iron credit. It should be mentioned, however, that because of the large electrical power requirements, the plant should be located where electricity is plentiful and inexpensive.

CONCLUSIONS AND RECOMMENDATIONS

Rutile synthesis was successfully demonstrated in pilot-scale equipment using a Tahawus rock-type ilmenite. An ilmenite-to-carbon weight ratio of 6.2 to 7.2 was sufficient when smelting charges of Tahawus ilmenite blended with coke and lime at approximately 1,600° C to separate high-quality pig iron as a byproduct and leave titania enriched in a slag fraction of low-iron content.

Techniques devised for oxidizing the molten titania slag included use of an oxidation launder and a gas sparger. The gas sparger provided the most efficient method for oxidizing slag to convert titanium oxides to rutile crystals. Titanium pyrophosphate can be added directly to molten slag to react with slag impurities and form a phosphate glass matrix from which crystalline rutile is recovered.

Large-scale tests should be made combining rutile synthesis with the chlorination process. These tests would reveal what problems, if any, might develop with different ilmenite feedstocks, carbon instead of graphite electrodes, and slag-treatment techniques on a large scale.

Based on the economic evaluation, for a 500-tpd synthetic rutile plant, the process developed at the Albany Metallurgy Research Center is competitive with natural rutile without considering the iron credit. Electrical power requirements indicate that the proposed plant should be located where electricity is plentiful and inexpensive.

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APPENDIX

TABLE A-1. - Raw material and utility requirements

	Quantity per ton synthetic rutile
Raw materials:	
Ilmenite........................tons.	2.144
Coke............................ton.	0.246
Orthophosphoric acid.............do..	0.134
Electrodes graphite..........pounds..	104.000
Lime.............................do..	176.00
Bentonite........................do..	104.000
Limestone.......................ton..	0.240
Sulfuric acid....................do..	.396
Utilities:	
Electric power.................kwhr..	4295.066
Process water..................Mgal..	.816
Raw water......................Mgal..	8.319
Natural gas...................MMBtu..	1.983

TABLE A-2. - Direct labor requirements

Section and item	Direct labor assignment			
	Number of men	Shifts per day	Days per week	Total men required
Feed preparation section:				
Front-end loader..........................	1.0	1	5	1.0
Disk agglomerator.........................	1.0	3	7	4.2
Dryer.....................................	2.0	3	7	8.4
Subtotal.............................		-	-	13.6
Smelting and slag treatment:				
Smelting furnaces.........................	9.0	3	7	37.8
Pig-casting machine.......................	2.0	3	7	8.4
Locomotives...............................	4.0	1	5	4.0
Converter.................................	1.0	3	7	4.2
Subtotal.............................	-	-	-	54.4
Leaching section:				
Leaching tanks............................	1.0	3	7	4.2
Dryer.....................................	1.0	3	7	4.2
Subtotal.............................	-	-	-	8.4
Product recovery: dryers (2)................	4.0	3	7	16.8
Oxygen plant: air producer..................	2.0	3	7	8.4
Steamplant..................................	1.0	3	7	4.2
General plant...............................	4.0	3	7	16.8
Total...............................	-	-	-	122.6

TABLE A-3. - Daily utility requirements

Section and item	Electric power, kwhr	Process water, Mgal	Raw water, Mgal
Feed preparation section: disk agglomerator......	11,538	32	0
Smelting and slag treatment......................	2,096,351	0	0
Leaching section:			
Condenser....................................	-	64	3,436
Leaching tanks...............................	-	165	0
Filters......................................	-	120	0
Subtotal.................................	7,622	349	3,436
Product recovery.................................	2,491	0	0
Oxygen plant: air producer.......................	28,993	0	723
Steamplant.......................................	538	27	-
Total....................................	2,147,533	408	4,159

TABLE A-4. - Daily thermal requirements

Section and item	Steam, Mlb psig 15	Cooling water, Mgal	Natural gas, MMBtu
Leaching section:			
Condenser....................................	0.0	3,436.5	0.0
Leaching tanks...............................	179.1	.0	.0
Leaching tanks...............................	115.7	.0	.0
Subtotal.................................	294.8	3,436.5	.0
Product recovery:			
Dryer..	.0	.0	402.6
Dryer..	.0	.0	132.2
Subtotal.................................	.0	.0	534.8
Oxygen plant: air producer.......................	.0	723.0	.0
Steamplant.......................................	-	-	456.5
Total....................................	294.8	4,159.5	991.3

TABLE A-5. - Equipment cost summary, feed preparation section

Item	Cost[1] Equipment	Labor	Total
Hoppers	$29,700	$1,600	$31,300
Belt conveyors	231,800	41,600	273,400
Dust collectors	38,000	3,900	41,900
Feeders	14,600	2,200	16,800
Zigzag blender	13,400	1,300	14,700
Disk agglomerator	193,700	19,400	231,100
Dryer	184,800	29,600	214,400
Rotary cooler	84,800	13,600	98,400
Total	790,800	113,200	904,000
Covered storage			[2]126,700
Hoppers			[2]37,900
Storage silos			[2]92,400
Front end loader			46,500
Total equipment cost X factor indicated:			
Foundations, X 0.090			71,200
Buildings, X 0.080			63,300
Structures, X 0.070			55,400
Insulation, X 0.010			7,900
Instrumentation, X 0.020			15,800
Electrical, X 0.100			79,100
Piping, X 0.050			39,500
Painting, X 0.050			39,500
Miscellaneous, X 0.100			79,100
Total			450,800
Total direct cost			1,658,300
Field indirect, 10.0 pct of total direct cost			165,800
Total construction cost			1,824,100
Engineering, 5.0 pct of total construction cost			91,200
Administration and overhead, 5.0 pct of total construction cost			91,200
Subtotal			2,006,500
Contingency, 10.0 pct of subtotal			200,600
Subtotal			2,207,100
Contractor's fee, 5.0 pct of Contingency subtotal			110,400
Section cost			2,317,500

[1]Equipment costs are based on the M and S index of 443.8.
[2]Installed cost.

TABLE A-6. - Equipment cost summary, smelting and slag treatment

Item	Cost[1] Equipment	Labor	Total
Belt conveyors	$181,400	$32,700	$214,100
Hoppers	25,900	1,400	27,300
Feeders	13,500	2,000	15,500
Dust collectors	40,900	4,100	45,000
Storage bin	35,800	1,800	37,600
Gas coolers	90,700	9,100	99,800
Fans	10,500	1,000	11,500
Pig casting machine	249,800	37,500	287,300
Total	648,500	89,600	738,100
Minicomputer			[2]298,400
Gas holder			[2]68,800
Cranes			[2]303,900
Smelting furnaces			5,968,700
Molten iron ladle			31,600
Locomotives			675,200
Molten slag ladles			63,100
Converter			173,800
Total equipment cost X factor indicated:			
Foundations, X 0.150			97,300
Buildings, X 0.120			77,800
Structures, X 0.200			129,700
Insulation, X 0.050			32,400
Instrumentation, X 0.100			64,800
Electrical, X 0.100			64,800
Piping, X 0.050			32,400
Painting, X 0.010			6,500
Miscellaneous, X 0.100			64,800
Total			570,500
Total direct cost			8,892,100
Field indirect, 10.0 pct of total direct cost			889,200
Total construction cost			9,781,300
Engineering, 5.0 pct of total construction cost			489,100
Administration and overhead, 5.0 pct of total construction cost			489,100
Subtotal			10,759,500
Contingency, 10.0 pct of subtotal			1,075,900
Subtotal			11,835,400
Contractor's fee, 5.0 pct of Contingency subtotal			591,800
Section cost			12,427,200

[1]Equipment costs are based on the M and S index of 443.8.
[2]Installed cost.

TABLE A-7. - Equipment cost summary, leaching section

Item	Cost[1]		
	Equipment	Labor	Total
Blower	$8,800	$900	$9,700
Condenser	14,800	1,500	16,300
Surge tank	23,800	2,400	26,200
Quenching tanks	2,300	200	2,500
Pumps	37,200	1,200	38,400
Rod mill	76,500	4,600	81,100
Leaching tanks	47,800	2,300	50,100
Storage tanks	48,900	2,400	51,300
Thickener	67,900	3,400	71,300
Filters	295,300	14,700	310,000
Belt conveyors	69,400	12,500	81,900
Hoppers	5,100	400	5,500
Feeders	6,500	1,000	7,500
Dryer	52,400	15,700	68,100
Dust collectors	7,300	700	8,000
Rotary cooler	56,200	9,000	65,200
Storage bins	87,700	4,400	92,100
Mixing tank	22,000	1,100	23,100
Crushers	4,200	600	4,800
Bucket elevator	2,800	400	3,200
Vibrating screen	2,700	300	3,000
Total	939,600	79,700	1,019,300
Total equipment cost X factor indicated:			
Foundations, X 0.100			94,000
Buildings, X 0.180			169,100
Structures, X 0.040			37,600
Insulation, X 0.030			28,200
Instrumentation, X 0.040			37,600
Electrical, X 0.100			94,000
Piping, X 0.350			328,900
Painting, X 0.010			9,400
Miscellaneous, X 0.100			94,000
Total			892,800
Total direct cost			1,912,100
Field indirect, 10.0 pct of total direct cost			191,200
Total construction cost			2,103,300
Engineering, 5.0 pct of total construction cost			105,200
Administration and overhead, 5.0 pct of total construction cost			105,200
Subtotal			2,313,700
Contingency, 10.0 pct of subtotal			231,400
Subtotal			2,545,100
Contractor's fee, 5.0 pct of Contingency subtotal			127,300
Section cost			2,672,400

[1]Equipment costs are based on the M and S index of 443.8.

TABLE A-8. - Equipment cost summary, product recovery

Item	Cost[1] Equipment	Labor	Total
Hoppers	$1,400	$400	$1,800
Feeders	22,000	1,800	23,800
Dryer	442,600	132,800	575,400
Dust collectors	21,800	2,100	23,900
Rotary cooler	89,400	14,300	103,700
Belt conveyors	85,600	15,400	101,000
Storage bins	2,300	100	2,400
Storage tanks	42,300	2,100	44,400
Pumps	1,300	100	1,400
Zigzag mixer	7,500	200	7,700
Cooler	109,000	17,400	126,400
Total	825,200	186,700	1,011,900
Storage silos			[2]218,200
Total equipment cost X factor indicated:			
Foundations, X 0.090			74,300
Buildings, X 0.080			66,000
Structures, X 0.070			57,800
Insulation, X 0.010			8,300
Instrumentation, X 0.020			16,500
Electrical X 0.100			82,500
Piping, X 0.050			41,300
Painting, X 0.010			8,300
Miscellaneous, X 0.100			82,500
Total			437,500
Total direct cost			1,667,600
Field indirect, 10.0 pct of total direct cost			166,800
Total construction cost			1,834,400
Engineering, 5.0 pct of total construction cost			91,700
Administration and overhead, 5.0 pct of total construction cost			91,700
Subtotal			2,017,800
Contingency, 10.0 pct of subtotal			201,800
Subtotal			2,219,600
Contractor's fee, 5.0 pct of Contingency subtotal			111,000
Section cost			2,330,600

[1]Equipment costs are based on the M and S index of 443.8.
[2]Installed cost.

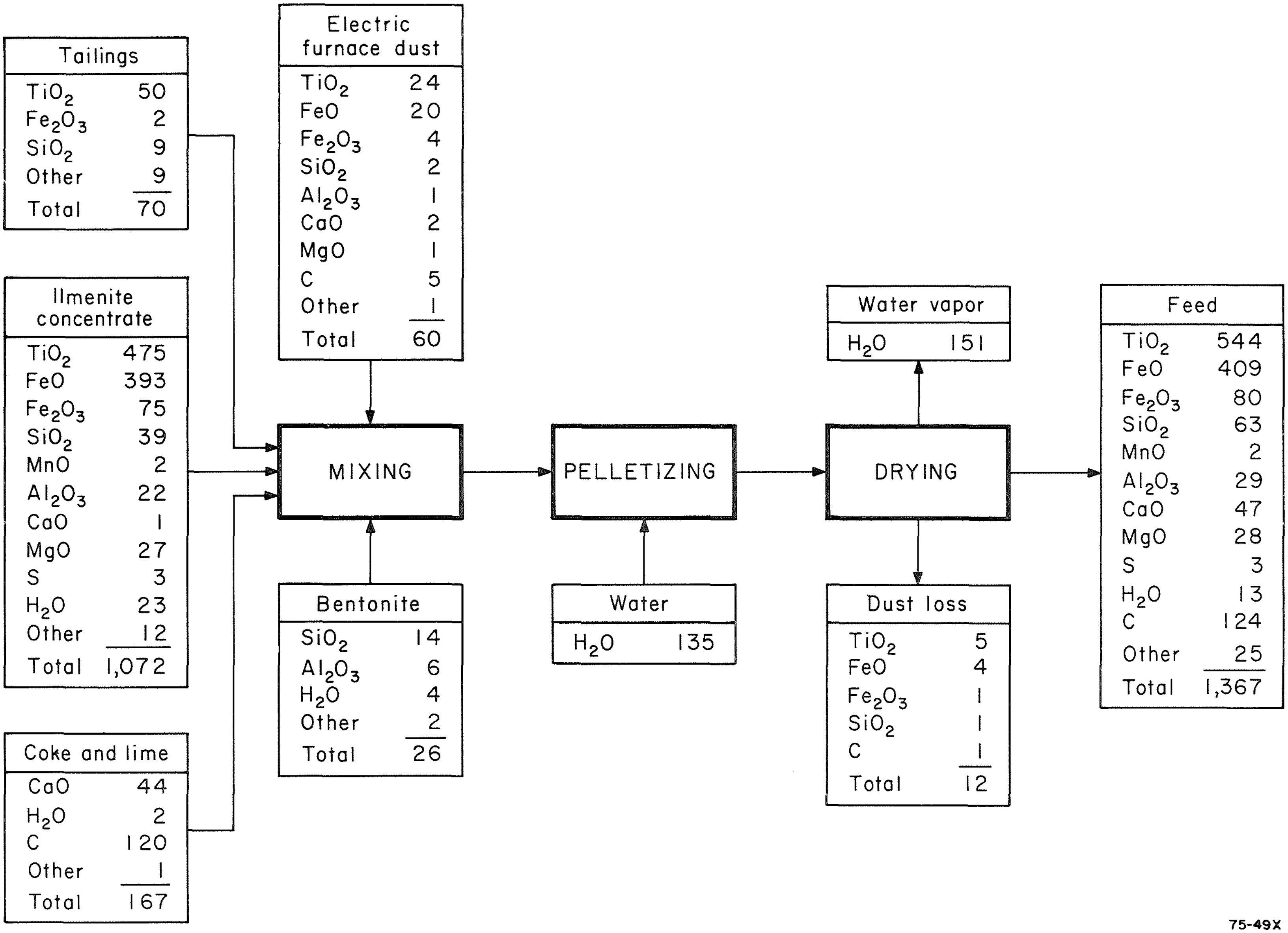

FIGURE A-1. - Feed preparation section. All figures in tons per day.

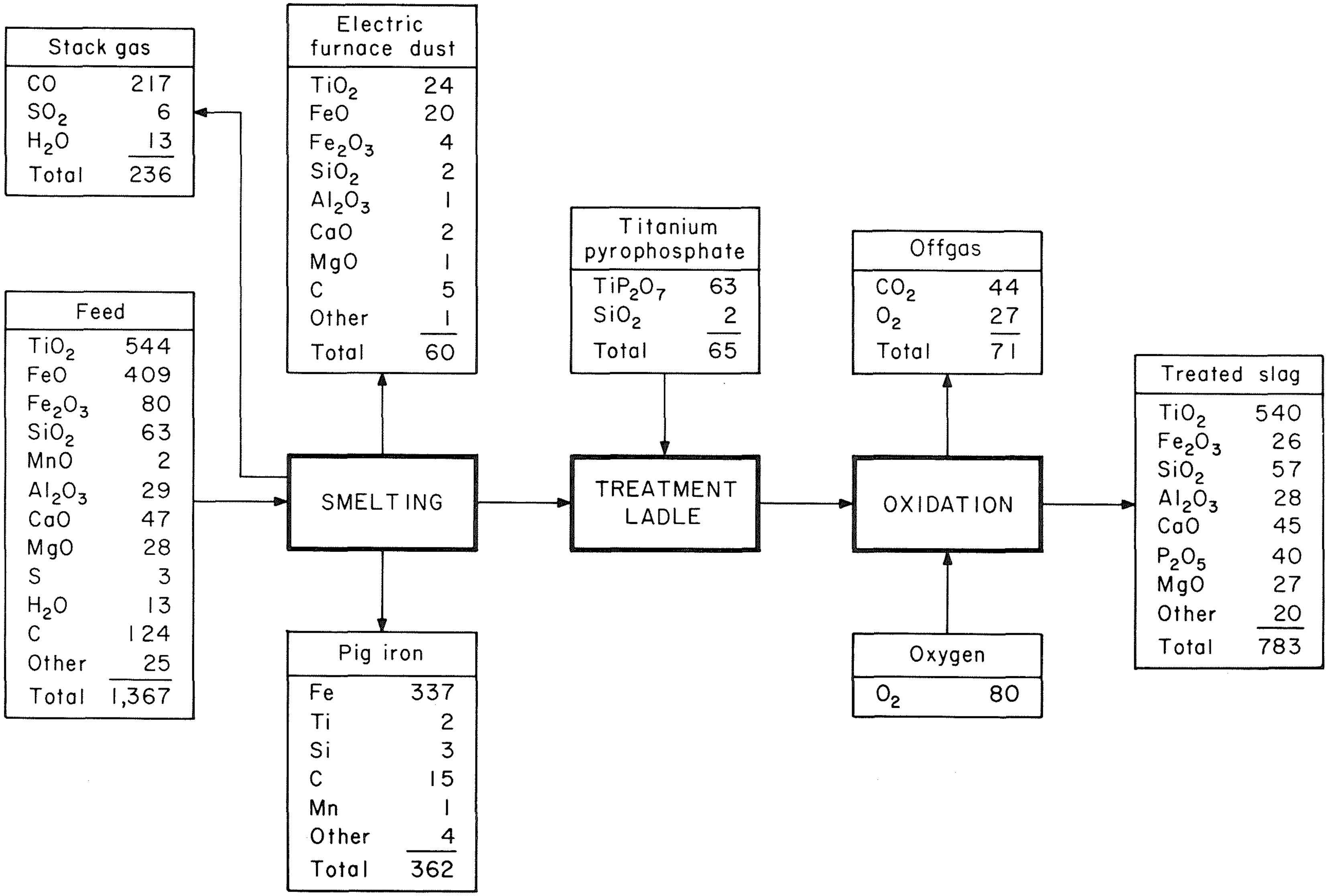

FIGURE A-2. - Smelting and slag-treatment section. All figures in tons per day.

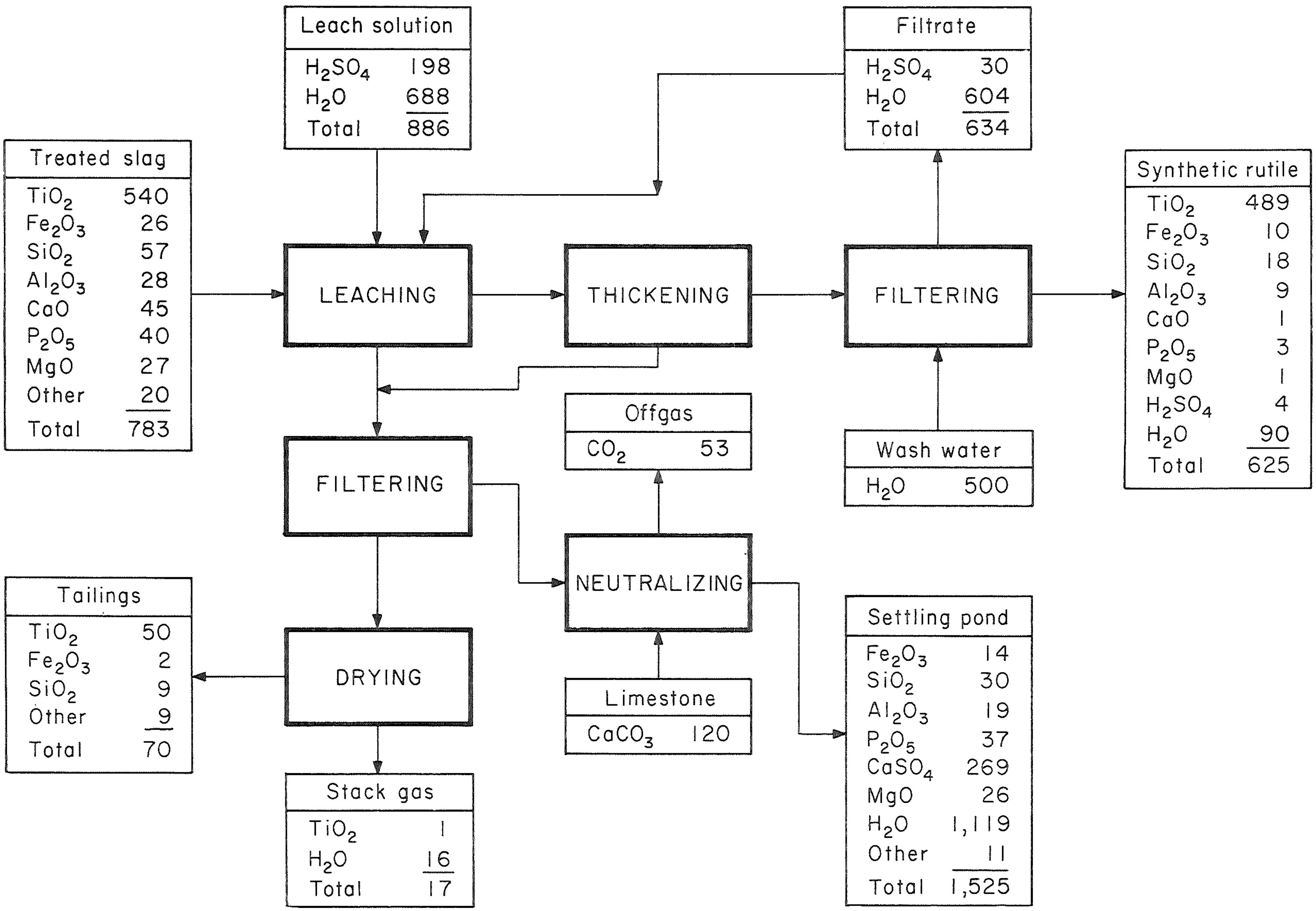

FIGURE A-3. - Leaching section. All figures in tons per day.

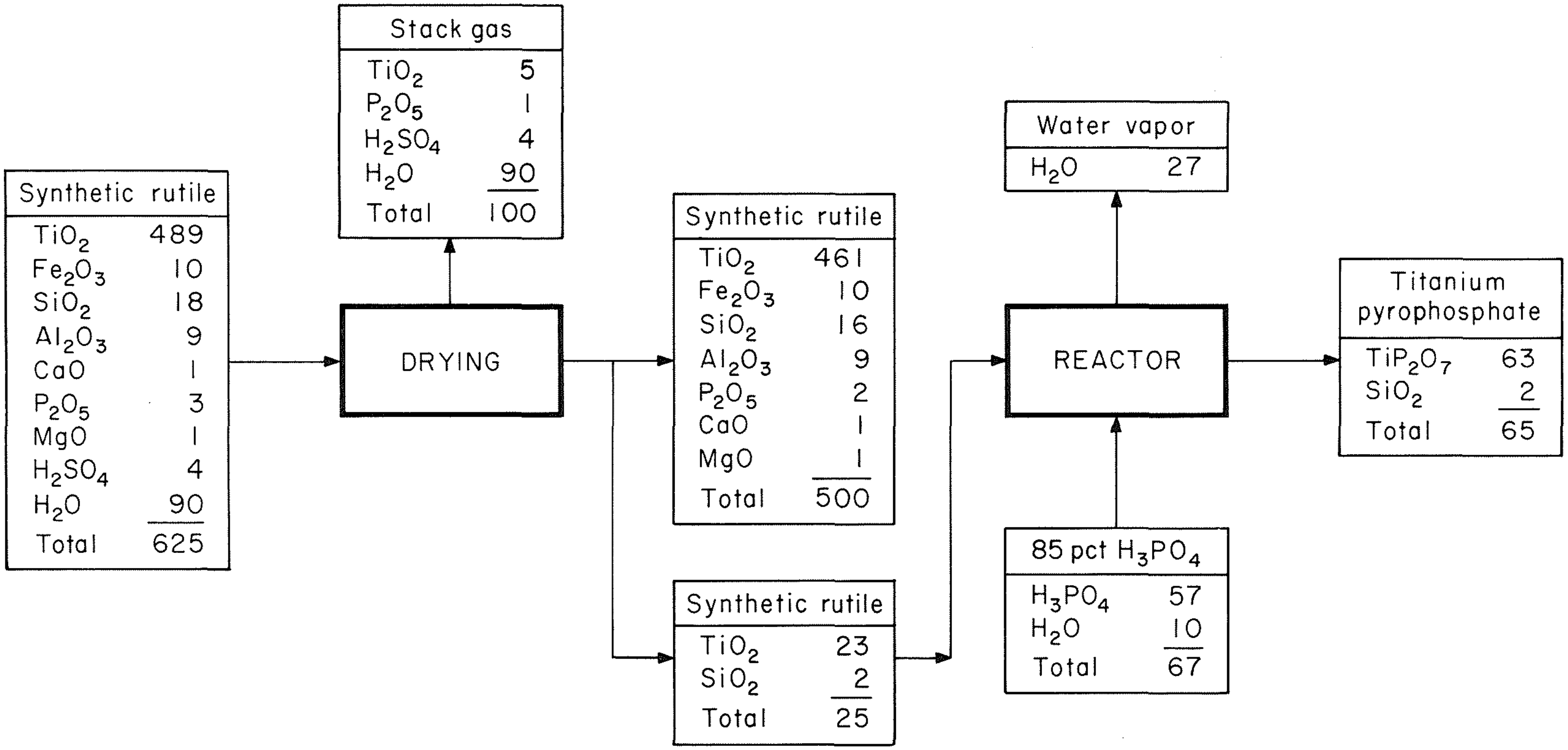

FIGURE A-4. - Product-recovery section. All figures in tons per day.

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