

**RECOVERY OF A HIGH GRADE PYROPHYLLITE  
CO-PRODUCT FROM BAGHOUSE DUST**

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**ABSTRACT**

The North Carolina State Minerals research Laboratory is studying the potential of column flotation to produce a high grade refractory product from baghouse dust collected from a pyrophyllite operation. The baghouse dust is considered a waste product and consists primarily of quartz, feldspar, andalusite, and pyrophyllite. Column variables investigated include froth depth, reagent types, and dosages. The product is evaluated for pyrometric cone equivalent, chemical analysis, particle size distribution, and surface area.

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### **INTRODUCTION**

Waste production is a costly and unavoidable result of ore deposits. The North Carolina State University Minerals Research Laboratory (MRL) works to economically utilize mine waste, reduce the waste stream, and create viable products for consumption. MRL has a program designed to find methods to economically utilize this mine waste to create marketable products, thus reducing the total waste stream. Baghouse dust is often difficult to contain and to dispose because of its small particle size and low bulk density. In effort to reduce the baghouse dust stream in a North Carolina pyrophyllite operation the MRL is studying the capability of column flotation to separate high alumina materials and produce a viable high grade refractory product. The baghouse dust is presently removed from two points during the production of pyrophyllite. Only a portion of this material can be sold due to its low alumina values. The remainder is impounded.

Pyrophyllite is a hydrous aluminum silicate ( $\text{Al}_2\text{O}_3 \cdot 4\text{Si}_2\text{H}_2\text{O}$ ) and is found in monoclinic and massive rhombohedral crystalline patterns (Cornish 1983). Its theoretical composition is 66.7%  $\text{SiO}_2$ ,

28.3%  $\text{Al}_2\text{O}_3$ , and 5.0% LOI. Pyrophyllite is mined in Japan, Australia, and North Carolina. Pyrophyllite originates in acid volcanic rocks or tuffs where alumina is high. Pyrophyllite has a high alumina content which is extremely effective for refractory utilization. Commonly found in formations with pyrophyllite is andalusite. Andalusite is another aluminum silicate with a high content. A combined pyrophyllite and andalusite product has properties of relatively high pyrometric cone equivalent, low hot load deformation, thermal shock resistance, reheat stability and low thermal conductivity (Cornish 1983). Pyrophyllite is used in ceramics, cosmetics, insecticides, paint, roofing, rubber, and refractory products.

Column flotation technology is a relatively recent innovation in the mining industry. Hollingworth (1967) filed the first U.S. patent for a countercurrent flotation column. Since then a large portion of the research done on the column flotation process has focused on very fine to ultrafine particle operations. With the recognition of the successes of the column flotation process by Reddy et al (1988), Luttrell et al (1988), Ynchansti et al (1988) and others with ultra fine particles, the MRL has researched the use of column flotation to recover and reduce waste in many non-metallic minerals produced in North Carolina, including pyrophyllite.

The basic concept is very similar to traditional cell flotation. Air is introduced in the form of bubbles at the bottom of the column. The bubbles attract the ultrafine, hydrophobic particles and carry them upwards. the length of the column allows for a finer separation by discouraging the hydrophillic particles from washing out with the hydrophobic ones. Key to proper results are the froth depth, wash water rate, column height, feed rate, and reagent types and dosages.

## EXPERIMENTAL

### Materials

**Sample:** The sample used in the project was collected directly from the baghouses at a pyrophyllite mining operation. The front baghouse material was from the crushing and screening operations and was identified as MRL #6039. The dust collected in the rear baghouse was collected from the drying and bagging process and was identified as MRL #6038. The samples were placed in 50 lb. bags. The samples were split from both materials and the particle size distribution was determined by screen analysis on a 200 mesh screen. Eighty percent of the sample was found to be less than 200 mesh.

**Reagents:** Tests were done with two frothers: Nottingham Econofroth 925, a water-soluble polyglycol ether and Cyanamid Aerofroth 65, a pure polyglycol. Fuel oil was included as a promoter. Petrosulfonates Nottingham M70 and Nottingham HM70 as well as amines Nottingham A-60 and Nottingham A-50 were used as collectors. NaOH and H<sub>2</sub>SO<sub>4</sub> were used to control the pH.

**Experimental Setup:** For the benchscale test, a standard two liter Denver flotation cell was employed at 1200 rpm. The column utilized in this project has a height of 226 cm and a diameter of 9.5 cm and is constructed of a clear acrylic. The total column area is 71.3 cm<sup>2</sup> and the volume is 16.1 liters. The feed location for this column is located 66.7 cm from the top and the bubble generator is located 18 cm from the bottom of the column. The reagents input units were peristaltic pumps. The bubble sparger is a porous polyethylene tube of a 20 micron mean pore size and a 1.59 cm diameter manufactured by Porex Corporation. The details of the flow and the column are shown in the index to this paper. Each following figure will be sequentially placed in the index.

## PROCEDURES

**Material Split:** A 50 lb. bag of both MRL #6038 and MRL# 6039 were split using a conventional manual splitter into approximately 32 samples weighing between 500 and 600 grams. One of the 32 samples for both 6038 and 6039 was taken and split again into approximately 100 gram sample. This sample was placed on a 200 mesh screen and run on the rotap for twenty minutes. The split above and below 200 mesh was measured and a percentage of weight calculated. The splits from both 6038 and 6039 were then processed by heavy liquid analysis.

**Bench Scale:** The standard flotations were done for this project to provide a comparative measure of the results of the column floats and to also optimize the reagents used. The two materials were split into approximately 500g samples. Scrubbing and conditioning were done at 60% solids for 5 minutes at 700 rpm in the standard benchtop scrubbers with a three tiered blade. At this time, the pH was taken and recorded. The material then was placed in a standard Denver flotation cell and floated for 3 minutes. The float concentrate was placed in a cleaner flotation process similar to the first for a period of 3 minutes. The products and tailings were filtered, dried, and weighted. Three standard floats were done with combinations of NaOH, fuel oil, Nottingham 925, A-50, A-60, and H<sub>2</sub>SO<sub>4</sub>. Ten more tests were run with one or two reagents to attempt to optimize the reagents to be used in the column flotations. A flow sheet is attached in the index to illustrate this process.

**Column flotation:** Before beginning to run the column flotation, the reagent pumps, wash water flow rate, air pressure, feeder, and column water flow rate were calibrated. Once calibrated, the column was stated up and the readings were checked again and adjusted. The column was allowed to run for an hour before samples were taken to achieve accurate measures of the separation. The column was fed by an automatic feeder that was checked periodically to insure a constant feed rate.

After reaching equilibrium, a first set of samples were taken from the concentrate discharge and the tailings discharge for a period of five minutes. A sample was also taken from the conditioner discharge for two minutes directly after the concentrate and tailings samples were taken. The column was then allowed to run for 30 minutes before a second set of samples were taken from the

concentrate and the tailings discharges and the conditioner discharge. The samples were then weighed wet, filtered, and dried. The dry weight was taken and the sample split for heavy liquid analyses.

Analytical procedures: The procedure used for heavy liquid mineral separation is detailed in Sullivan (1927) and Browning (1961). The head feeds as well as the products and tailings from both the standard flotation and the column flotation were separated by heavy liquids to estimate the mineral content. The divisions of the material were labeled as quartz for any material that had a density of less than 2.71, pyrophyllite for any between 2.71 and 2.96, and andalusite for any above 2.96. The samples were then sent for chemical analysis for LOI, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Na<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, and Fe<sub>2</sub>O<sub>3</sub> and for pyrometric cone equivalent. There is a flow sheet in the index that illustrates the heavy liquid analysis.

## **RESULTS AND DISCUSSION**

### **Bench Scale Reagent Optimization Test**

For the sample designated as MRL#6038, the bench scale test showed that the highest grades were obtained by floating with a combination of fuel oil and polyglycol frother at a pH close to 9.0. However, the recoveries for pyrophyllite at this condition were relatively low. At approximately 15-20% the natural pH of between 5.2 and 6.0, the pyrophyllite recovery increased to 30-40% while the grade remained relatively high at approximately 60%.

For the sample designated as MRL #6039, the highest pyrophyllite grade (77.1%) was obtained using only a frother. However, this resulted in the lowest pyrophyllite recovery (32.9%) of all the conditions tested. The best overall test came from using a small amount of fuel oil (along with 0.2#K frother) in the rougher float and an additional 0.1#14 frother in the cleaner step. The pyrophyllite grade for this test was 71.6% with a pyrophyllite recovery of 45.9%. The yield was 14.9%. Several different reagents were tested in the bench test on both MRL #6038 and #6039 in the major distinctions of amines and petroleum sulfonates. The tests show the best results for both 6038 and 6039 with the petroleum sulfonate reagent used. This information was carried over to the column tests.

For all of the bench tests, a cleaner stage was necessary to produce a high grade pyrophyllite concentrate. The bench tests had poor yields because the froth depth in a standard Denver flotation cell is relatively small, approximately one inch. This does not allow the small particles of the pyrophyllite dust enough opportunity to associate themselves with a bubble to be floated. The large bubble size of the Denver cell also restricts the number of particles that can attached and floated out of the cell. These two factors combined necessitate the use of a second cleaner float to concentrate the alumina. This ultimately leads to lower overall yields due to losses of the fines during filtration as well as more intensive use of energy and reagents to create the desired product.

The headfeed analysis done on MRL #6038 and #6039 shows a distinct difference in the percentage of pyrophyllite and overall alumina content. The sample MRL# 6039 had a 22.9% pyrophyllite and 28.3% overall alumina content according to the heavy liquid analysis. This was much higher than MRL #6038 which had 11.9% pyrophyllite and 21.7% alumina according to the heavy liquid analysis. The feeds remained the same in the column tests. A complete breakdown of

the feed analysis is found in the index of this paper.

### **Column Test Results**

The froth depth tests were run on the MRL #6038 sample. With consistent feed and reagents on all the tests, the alumina content and the percent pyrophyllite on the samples taken from the varying froth depth. Both the alumina content and percent pyrophyllite rose considerably with an increase in froth depth up to 30 inches (see graphs in the index). At this point, the weight the froth started to cause the collapse of the bubbles which defeats the purpose of flotation. With this information, the remainder of the tests were run at the optimal depth of 30 inches.

A number of tests were conducted using different amounts of fuel oil to gain a better understanding of its effect as a collector and a conditioner. For both of the tests on MRL #6038 and #6039, the alumina content in the samples taken remained fairly constant, varying between 27 and 28% for MRL#6038 and 26 and 28.5% for MRL#6039. This was what was generally expected to happen due to the nature of the interaction that fuel oil has during flotation.

For MRL #6038, the addition of fuel oil appears to have hurt the pyrophyllite recovery and yield as illustrated by the graphs in the index. The best recoveries at 28.3% were made without using fuel oil and there is a substantial drop in recoveries with the first additions of fuel oil. The yield grade shows the same decrease at 0.1 lb. per ton of fuel oil. However, both graphs do show an increase at 0.2 lb. per ton of fuel oil which suggests that the fuel oil does aid in concentrate production. The reason for the drop is believed to be the isopropyl alcohol that is used to cut the fuel oil so that it may accurately pumped. Isopropyl alcohol is known to cause the bubbles in a froth to become brittle and collapse. The alcohol might have negatively impacted the recoveries and yields for MRL #6038. More tests will be needed to figure out the appropriate dosage of fuel oil and to determine the extent of its beneficial effects.

For the MRL #6039 sample, the tests showed a very typical recovery- yield- grade relationship with response to the addition of fuel oil. The graphs found in the index show the percent pyrophyllite recovery increases from 25.3% to 29.2% and the yield increases from 6.0% to 12% with the increase of fuel oil dosage. In response, the third graph showing the grade drops from 80.1% to 70.8% as the fuel oil dosage is increased. The fuel oil at 0.2 lb. per ton did increase the yield to close



to an acceptable yield, but the grade dropped below an acceptable grade for industry. More tests will be need to attempt to bring the grade and the yield to appropriate and fiscally acceptable standards.

## CONCLUSIONS

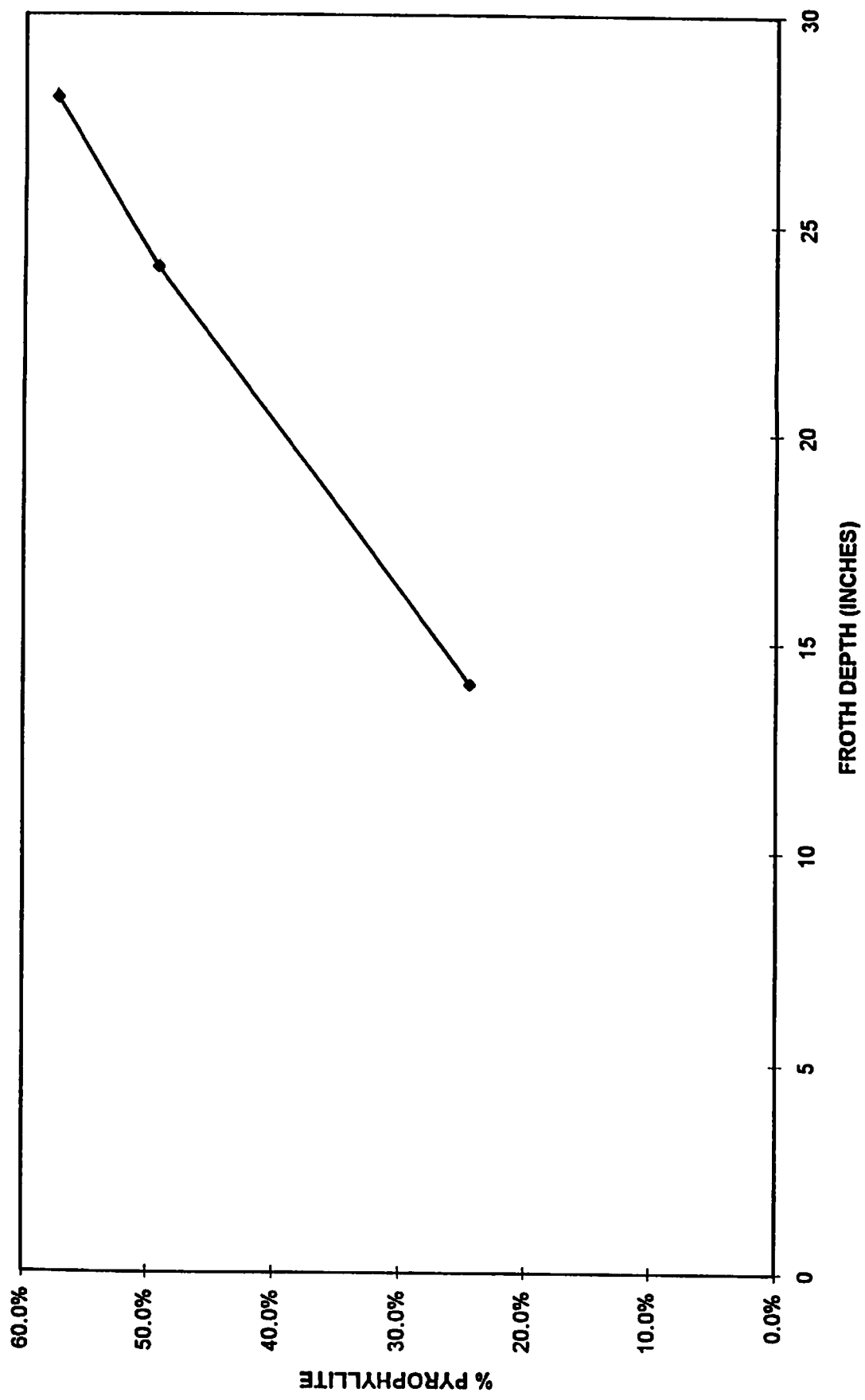
For this study, the column tests produced higher grade pyrophyllite products with higher recoveries in a single stage when compared to a two-stage bench float. In the column tests, a deep froth depth of 30 inches gave the highest grade of products because it allowed the froth to drain and cleanse itself in the wash water while maintaining the bubble structure integrity. The column tests did produce show that a high alumina content refractory product , with a percent alumina as high as 28.8%, can be made from this dust stream. However, more work needs to be done to determine how to increase the yields above 10% while maintaining a grade of better than 27% alumina content. Increasing the fuel oil in the column test increased the yield while maintaining a relatively constant alumina grade. Further tests are needed to determine the optimal fuel dosage due to the negative effect of the isopropyl alcohol on the bubble resilience. Adding petroleum sulfonate as a collector to create a combined pyrophyllite/andalusite product did increase the yield substantially, as high as 30.3% on MRL #6039, but the average grade was only 24% alumina. Further tests with petroleum sulfonate are needed to raise the grade of the combined pyrophyllite/andalusite product to above 27% alumina. Currently, these results are not efficient enough to be used in industry to produce a high grade alumina refractory product.

The study of recycling baghouse dust from pyrophyllite mines is an ongoing mission of the MRL as a service to the mining industry of North Carolina. The column flotation study is very much a preliminary study of this situation. While the tests of this study give an idea of the results of column flotation of pyrophyllite waste fines, more tests need to be done to optimize the recovery and grade in order for this venture to be profitable to these mines.

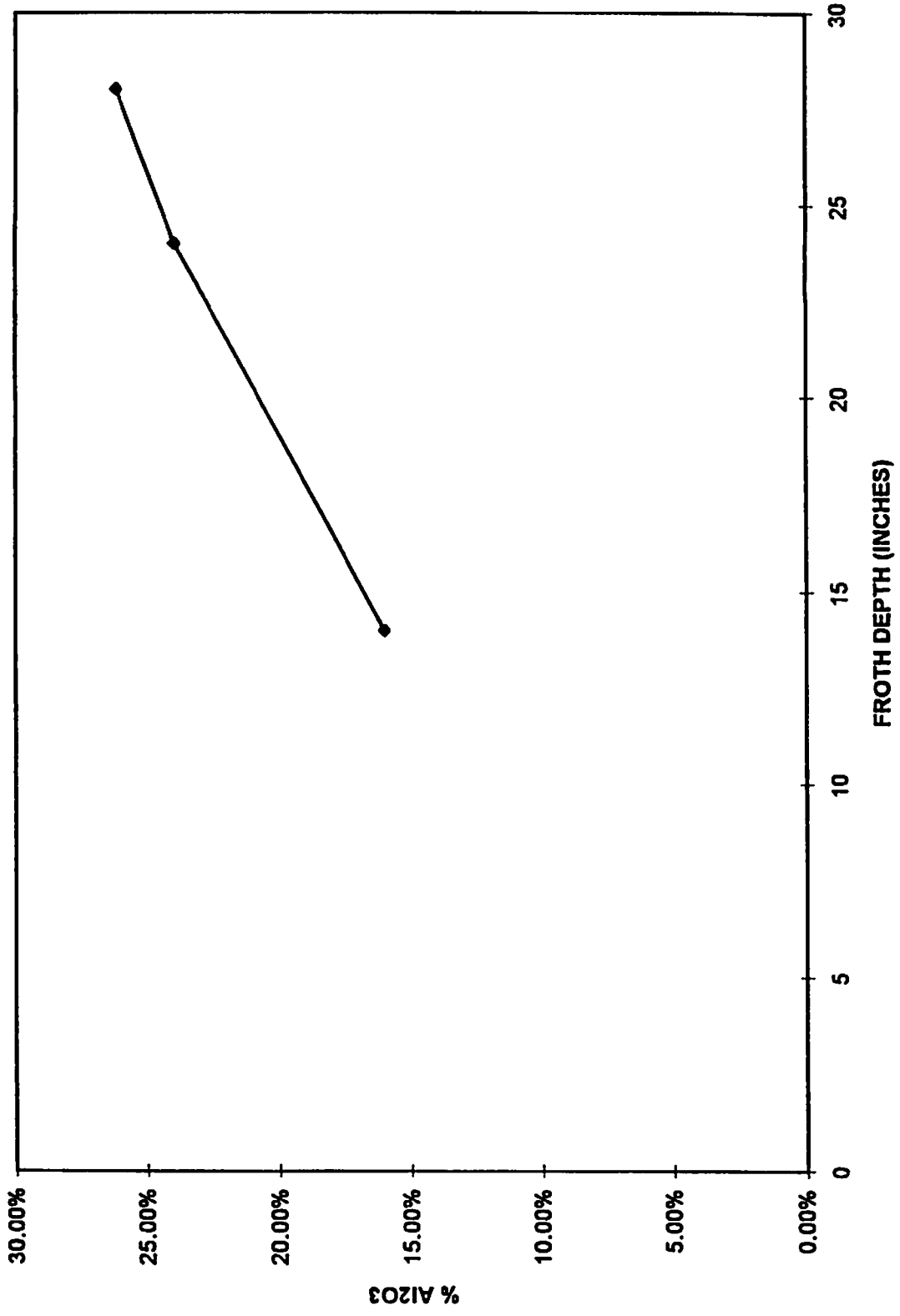
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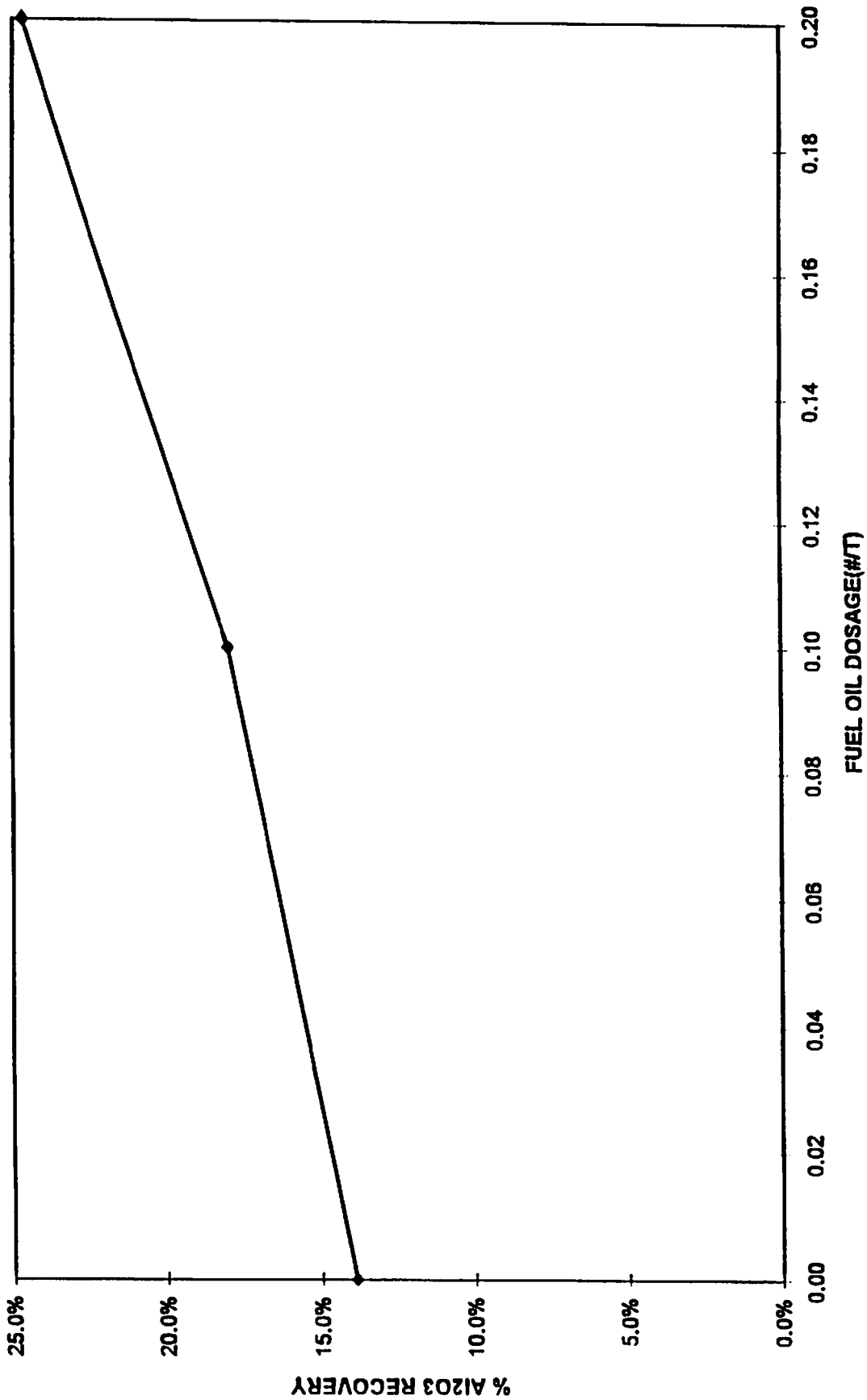
COLUMN FLOATS ON MRL #6038 - FROTH DEPTH VS. % PYROPHYLLITE



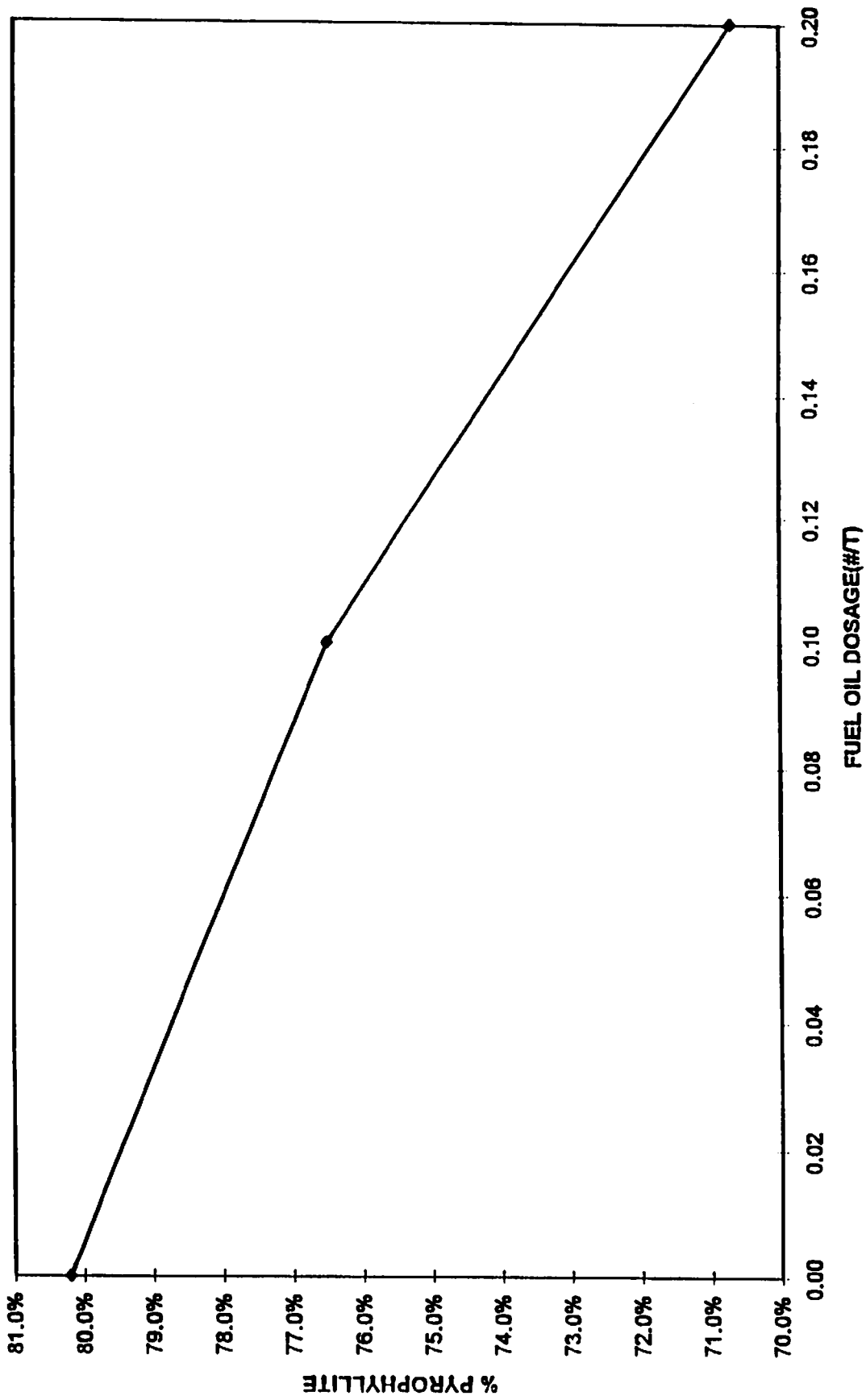
**COLUMN FLOATS ON MRL #6038 - FROTH DEPTH VS. ALUMINA CONTENT**



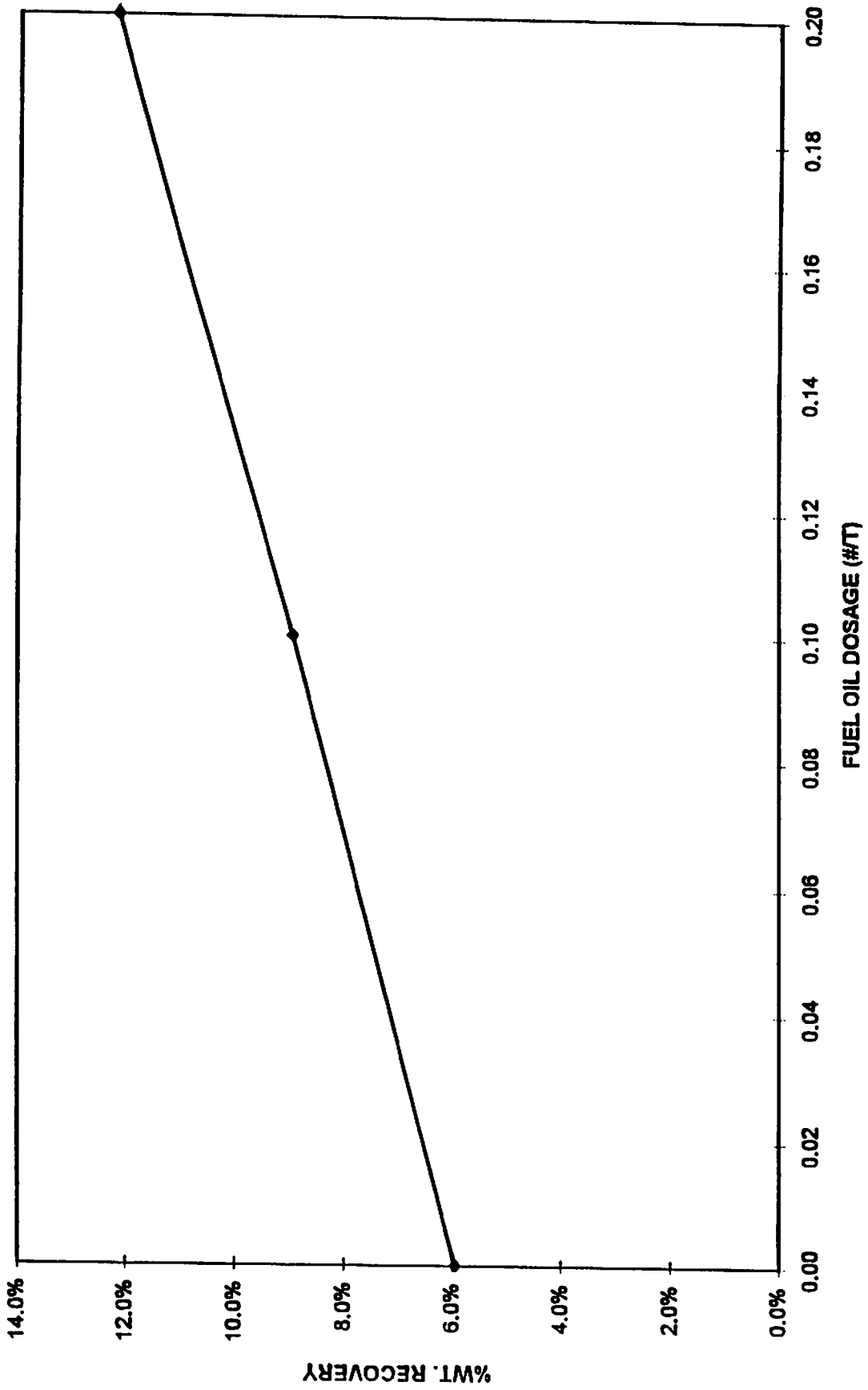
# COLUMN FLOATS ON MRL #6039 - FUEL OIL DOSAGE VS. ALUMINA RECOVERY



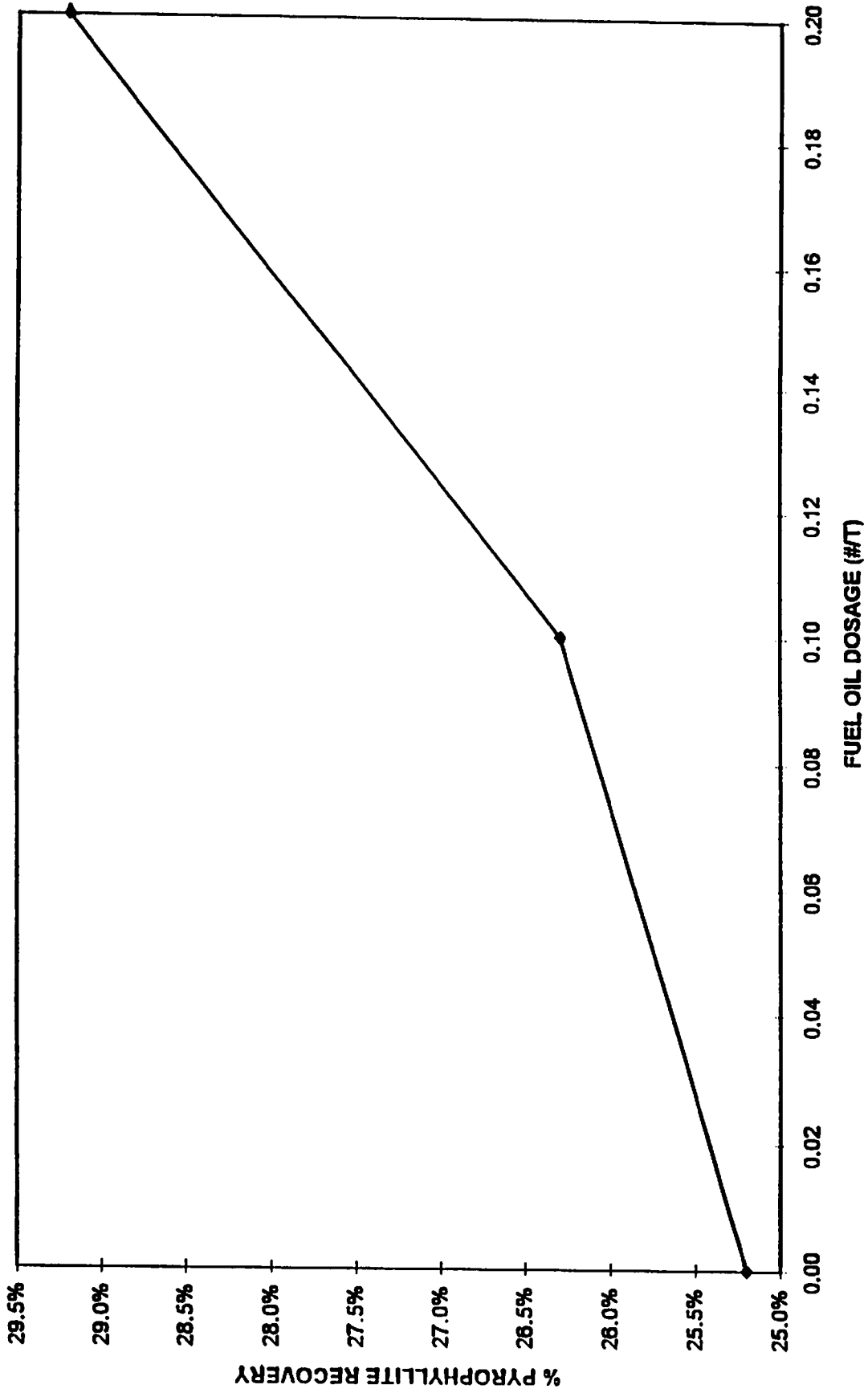
# COLUMN FLOATS ON MRL #6039 - FUEL OIL DOSAGE VS. PYROPHYLLITE GRADE



# COLUMN FLOATS ON MRL #6039 - FUEL OIL DOSAGE VS. YIELD

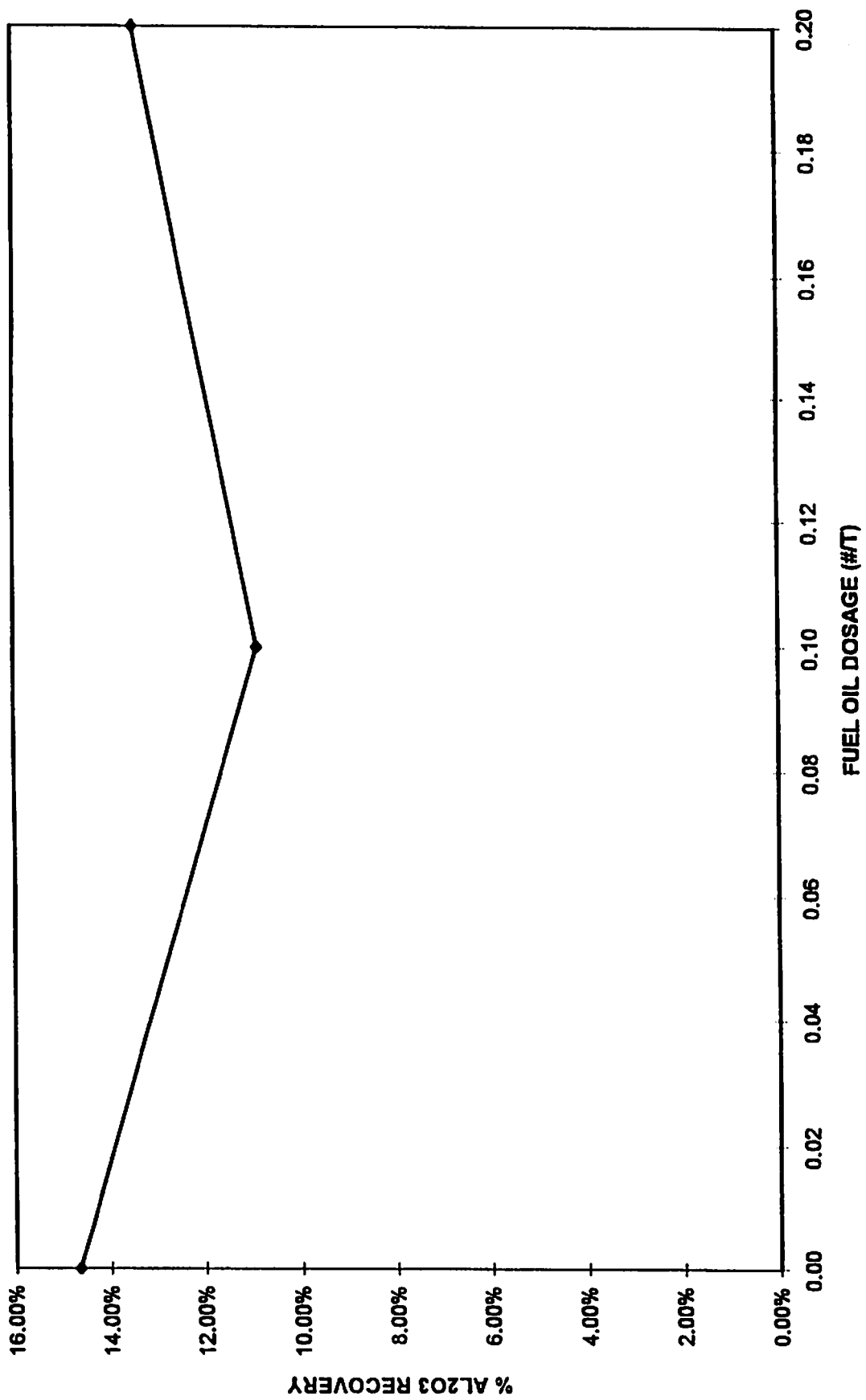


# COLUMN FLOATS ON MRL #6039 - FUEL OIL DOSAGE VS. PYROPHYLLITE RECOVERY

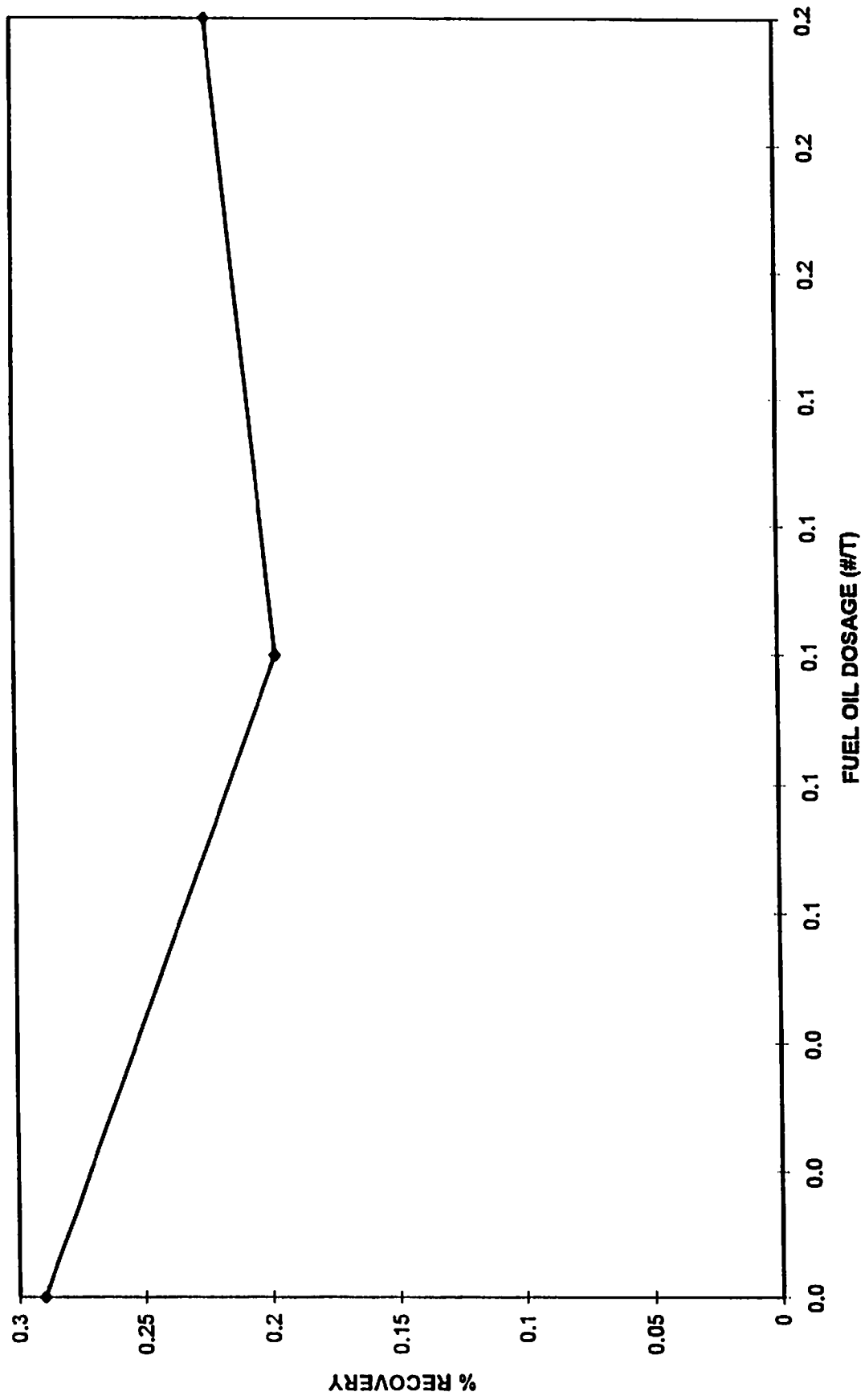




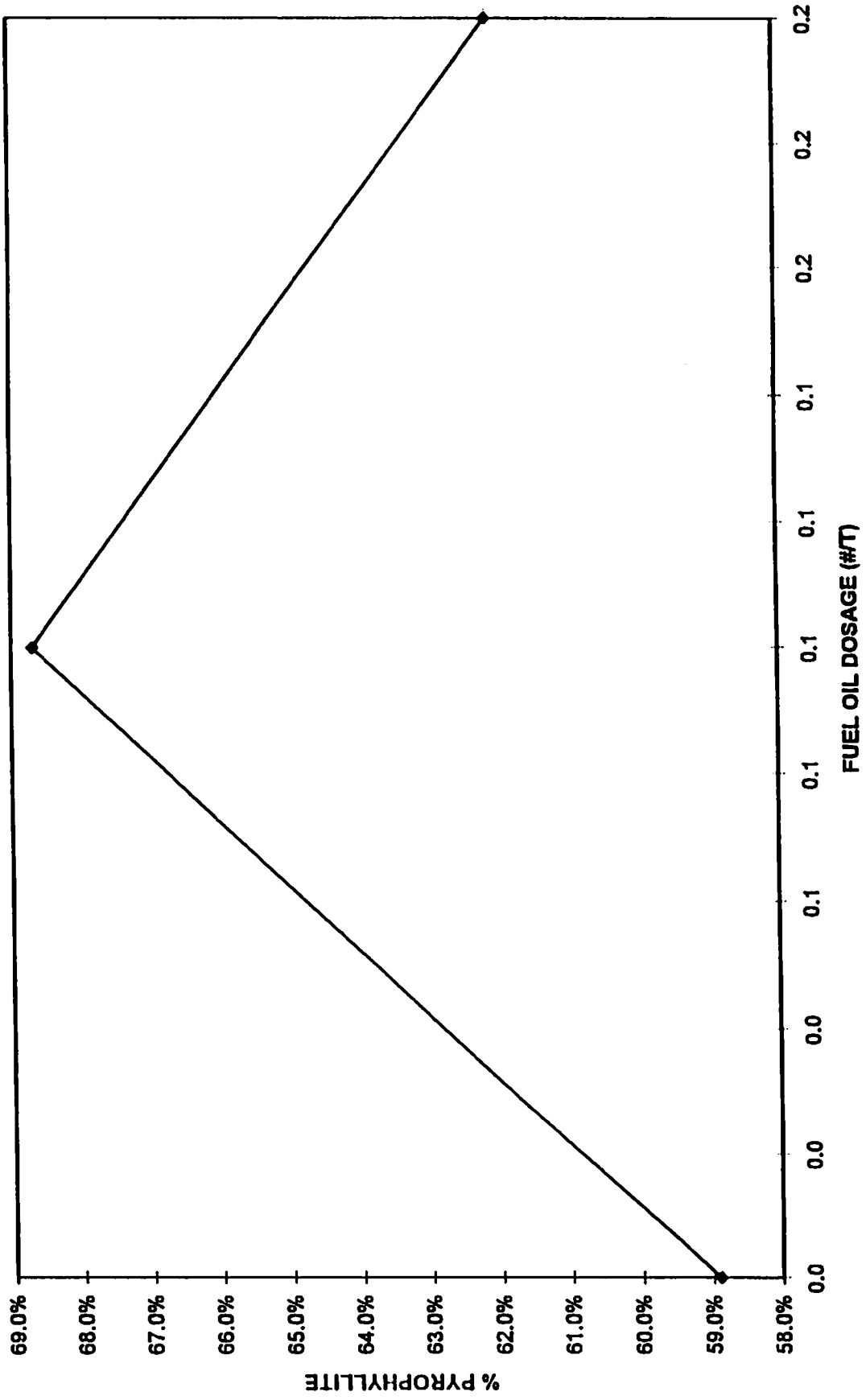
COLUMN FLOATS ON MRL #6038 - FUEL OIL DOSAGE VS. ALUMINA RECOVERY



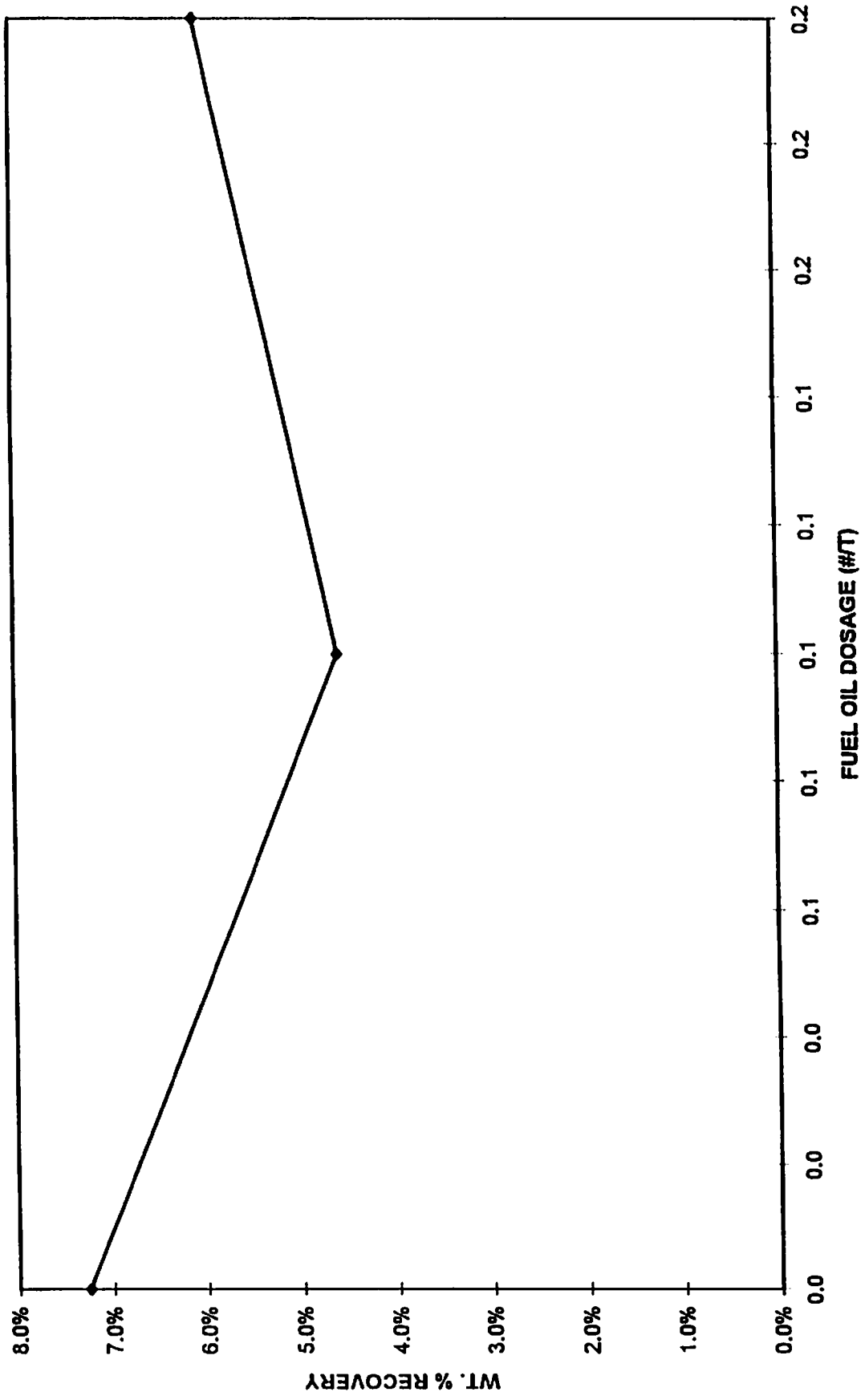
**COLUMN FLOATS ON MRL #6038 - FUEL OIL DOSAGE VS. PYROPHYLLITE RECOVERY**



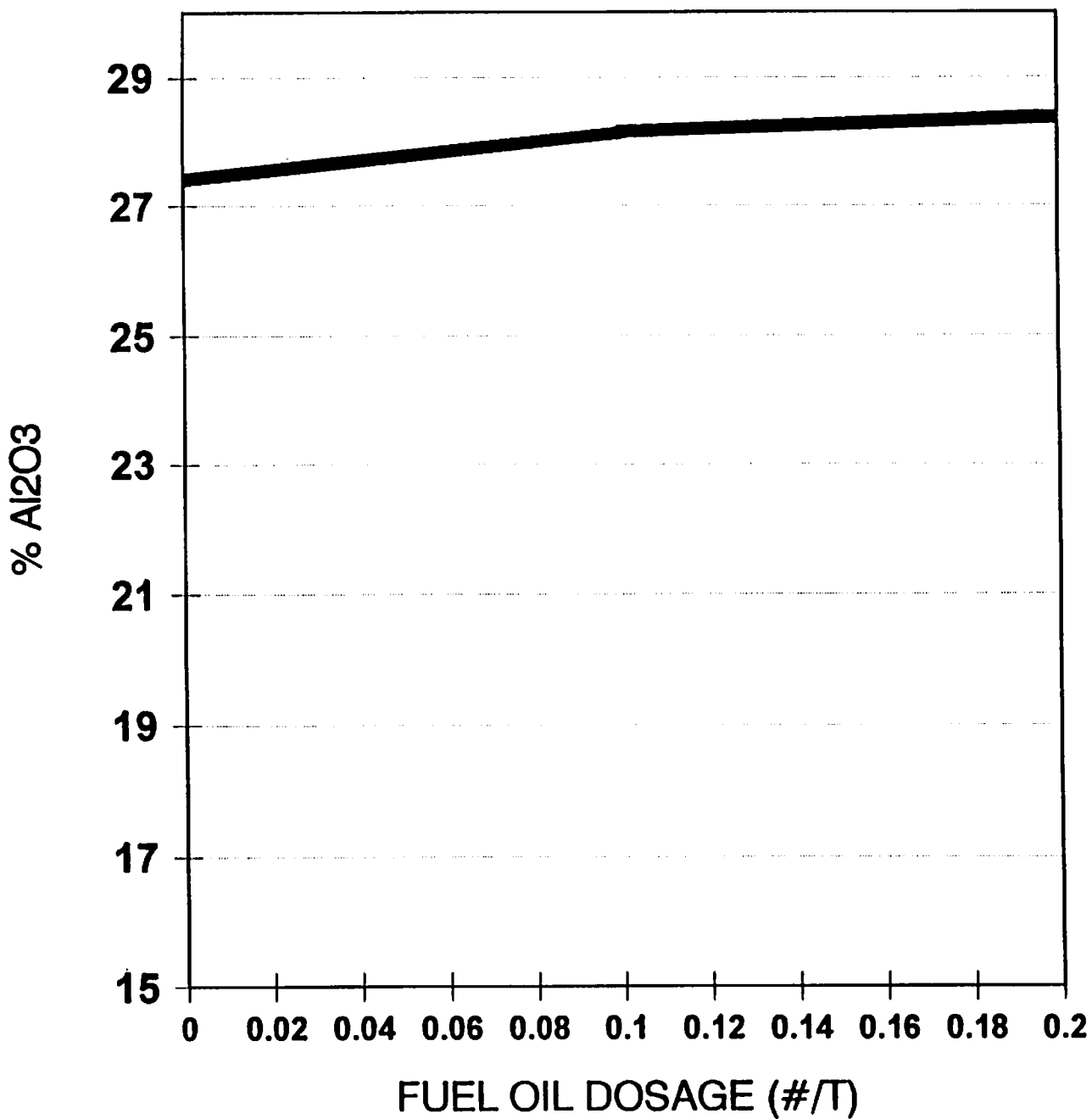
**COLUMN FLOATS ON MRL #6038 - FUEL OIL DOSAGE VS. PYROPHYLLITE GRADE**



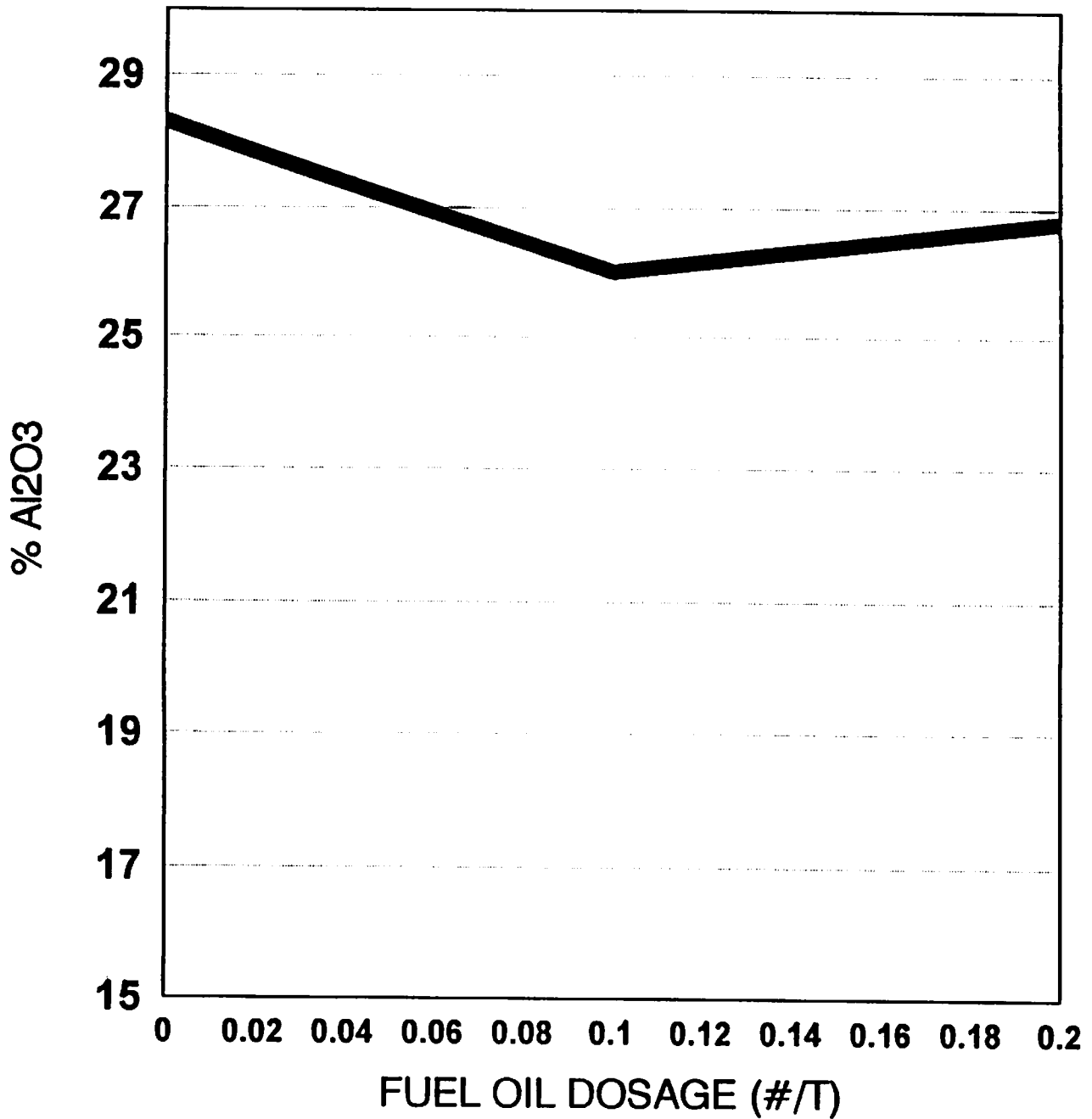
# COLUMN FLOATS ON MRL #6038 - FUEL OIL DOSAGE VS. YIELD



# COLUMN FLOATS ON MRL #6038 FUEL OIL DOSAGE VS. ALUMINA CONTENT



# COLUMN FLOATS ON MRL #6039 FUEL OIL DOSAGE VS. ALUMINA CONTENT



# Feed Analysis

Chemical	MRL#6038	MRL #6039
LOI	3.61	2.88
Al <sub>2</sub> O <sub>3</sub>	13.66	13.06
SiO <sub>2</sub>	79.53	82.26
Fe <sub>2</sub> O <sub>3</sub>	0.868	0.555
Na <sub>2</sub> O	0.178	0.094
K <sub>2</sub> O	0.802	1.158
<u>Heavy Liquids</u>		
Pyrophyllite	11.9	22.9
Andalusite	9.8	5.4
Silicates	78.3	71.6
<u>Theoretical Composition of Pyrophyllite</u>		
SiO <sub>2</sub>	66.7%	
Al <sub>2</sub> O <sub>3</sub>	28.3%	
LOI	5.0%	