TABLE II—SAMPLE FROM NEAR PT. LOMA

No.	Time exposed	Per cent K ₂ O	Per cent sol. salt	Per cent org. matter	Per cent ash	Per cent KCl	Per cent N	Per cent I	Remarks
2A.....	None	15.82	40.88	47.32	11.80	25.00	2.77	0.18	Wet, sent in jar
2B.....	3 1/2 hrs.	13.43	34.82	56.54	8.64	21.22	2.66	0.19	Wet, sent in jar
2C.....	15 1/2 hrs.	14.96	37.66	52.76	9.58	23.64	2.40	0.15	Wet, sent in jar
2D.....	18 1/2 hrs.	13.05	34.34	60.12	5.54	20.62	1.77	0.15	Wet, sent in jar
2E.....	21 1/2 hrs.	11.00	30.22	62.52	7.26	17.38	1.85	0.16	Wet, sent in jar
2F.....	24 1/2 hrs.	11.59	30.52	59.92	9.56	18.31	2.10	0.14	Wet, sent in jar
2G.....	27 1/2 hrs.	13.68	38.94	50.56	10.50	21.62	2.22	0.24	Wet, sent in jar
2H.....	39 1/2 hrs.	10.31	27.84	62.38	9.78	16.29	1.97	0.14	Wet, sent in jar
2I.....	42 1/2 hrs.	16.39	38.78	53.30	7.92	25.90	2.22	0.21	Wet, sent in jar
2J.....	45 1/2 hrs.	11.77	32.44	60.54	7.02	18.60	1.99	0.22	Wet, sent in jar
2K.....	48 1/2 hrs.	16.69	39.72	54.22	5.96	26.37	1.79	0.19	Wet, sent in jar
2L.....	51 1/2 hrs.	15.77	40.80	45.88	13.32	24.92	2.29	0.22	Wet, sent in jar
2M.....	63 1/2 hrs.	16.74	42.58	45.36	12.06	26.45	2.49	0.21	Wet, sent in jar
2N.....	66 1/2 hrs.	16.75	38.64	55.28	6.08	26.46	1.91	0.19	Wet, sent in jar
2O.....	72 1/2 hrs.	14.67	35.58	58.12	6.30	23.18	1.88	0.19	Wet, sent in jar
2P.....	75 1/2 hrs.	12.61	34.74	55.46	9.80	19.93	2.66	0.22	Wet, sent in jar
2Q.....	87 1/2 hrs.	15.54	40.84	48.92	10.24	24.56	2.47	0.19	Wet, sent in jar
2R.....	90 1/2 hrs.	17.62	41.42	49.48	9.10	27.84	2.16	0.16	Wet, sent in jar
2S.....	93 1/2 hrs.	17.03	42.44	44.50	13.06	26.90	2.28	0.20	Wet, sent in jar
2T.....	96 1/2 hrs.	15.61	37.80	54.42	7.78	24.67	2.16	0.10	Wet, sent in jar
2U.....	99 1/2 hrs.	20.28	46.28	43.96	9.76	32.05	2.12	0.20	Wet, sent in jar
2V.....	111 1/2 hrs.	15.72	38.80	51.44	9.76	24.84	2.26	0.23	Wet, sent in jar
2W.....	135 1/2 hrs.	15.60	39.24	48.74	12.02	24.65	2.57	0.27	Wet, sent in jar
2.....	111 1/2 hrs.	15.27	34.52	57.42	8.06	24.13	1.80	0.18	Dried and sent by mail
M. P.....		30.00	57.10	32.76	10.14	47.41	1.66	0.06	Sea lettuce from Marrowstone Point

presence in harvested kelp is not detrimental to the value of the kelp.

BUREAU OF SOILS
U. S. DEPARTMENT OF AGRICULTURE
WASHINGTON

A POSSIBLE COMMERCIAL UTILIZATION OF NELSONITE

By WILLIAM H. WAGGAMAN¹

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INTRODUCTION

In Nelson County, Va., there are large bodies of rock locally known as Nelsonite which consist essentially of the two minerals ilmenite and apatite. The material is not only of scientific interest but in the light of some recent experiments performed in this laboratory may be of considerable commercial importance.

The rock occurs in the foothills of the Blue Ridge near Roseland, Va., about 7 miles northwest of Arrington, a station on the Southern R. R., and 24 miles northeast of Lynchburg. According to Watson² it is probably of pre-Cambrian age and occurs in dikes which are in places over 65 feet thick and 2,100 feet in length as exposed on the surface. The formation can be traced in a general northeast southwest direction for about 7 miles. Watson described it as "a hard rock composed of granular white apatite and black ilmenite." After weathering, however, the rock can be readily disintegrated and a more or less clean separation made of the two minerals by either of the methods described further on in this paper.

ILMENITE

Ilmenite or titanite iron is a black crystalline mineral having feebly magnetic properties. It has a specific gravity when pure of 4.5 to 5.0 and the following formula and composition:

FeTiO ₃	Titanium oxide (TiO ₂).....	52.7 per cent
	Iron protoxide (FeO).....	47.3 per cent

According to Dana,³ however, the ratio of iron to

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² "Mineral Resources of Virginia," 1907, p. 300; "Economic Geology," U. S. Geol. Survey, 1909, pp. 206-7.

³ "A System of Mineralogy," 1892, pp. 217-219.

titanium varies widely corresponding to the general formula $MFeTiO_3 \cdot nFe_2O_3$.

Ilmenite is rather widely distributed in nature, occurring in beds in gneiss and other crystalline rocks; also in small particles in many crystalline rocks and frequently associated with magnetite. It is used in the manufacture of ferro-titanium for the steel industry. In 1911 there were 51.17 tons of ferro-titanium imported into this country valued at \$22,700, which gives an average price of \$443.61 per ton.

APATITE

Apatite or phosphate rock is a brittle crystalline mineral which varies in color from almost white to dark green-red and brown. There are two main varieties of this mineral; namely, chlor-apatite and fluor-apatite, the latter being much more plentiful than the former. The specific gravity of apatite varies from 3.17 to 3.23 and the formula and composition of the two varieties according to Dana, are as follows:

Chlor-apatite, $CaCl_2 \cdot Ca_4(PO_4)_3$	P_2O_5 =	41.0 per cent
	CaO =	53.8 per cent
	Cl =	6.8 per cent
Fluor-apatite, $CaF_2 \cdot Ca_4(PO_4)_3$	P_2O_5 =	42.3 per cent
	CaO =	55.5 per cent
	F =	3.8 per cent

Apatite, however, is seldom found absolutely pure and though widely distributed in nature does not often occur in minable quantities. On treatment with sulfuric acid it yields a very high-grade superphosphate, and if it were not for the fact that the better deposits are rather inaccessible and that careful picking or "cobbing" is necessary before a product sufficiently high-grade for the market is obtained, this mineral would be more extensively used in the fertilizer industry.

SEPARATION OF APATITE AND ILMENITE

At first sight the association of apatite and ilmenite in a rock would seem to seriously affect the commercial value of both of these minerals. In the manufacture

of steel, phosphorus is the most dreaded impurity which can occur in the iron or iron ore used, so that ilmenite containing over 1 or 2 per cent of this element would be rejected as dangerous.

On the other hand, the presence of comparatively small percentages of iron in phosphate rock nearly always causes trouble in the acid phosphate produced therefrom, and when this element is present in large amounts it often renders the acid phosphate unfit for use.

According to Watson¹ the ratio of apatite to ilmenite in nelsonite varies widely from rock consisting largely of the former mineral to that in which ilmenite predominates. Before such a deposit can be economically worked, therefore, it seems essential that a method be devised for making a clean separation of the two minerals.

There are two methods which suggest themselves for effecting such a separation. The first is based on the difference in the specific gravities of the minerals, and the second on the magnetic properties of the ilmenite. The ratio of the specific gravity of apatite and ilmenite is as 3.2 to 4.5. This difference is sufficiently great to warrant a fairly clean separation provided the rock is crushed to uniform size. Unless it is ground very fine, however, a preliminary screening is necessary before suspending the material in water. The various modifications of the methods of separating substances by the difference in their specific gravities are well known and need not be enumerated here.

A separation of the two minerals by means of a magnetic separator has also proved feasible but owing to the rather feeble magnetic properties of the ilmenite it is not entirely removed from the apatite, by this method, and though a preliminary heating enhances somewhat this magnetism, such a procedure would probably prove too expensive in commercial practice.

From a number of experiments with nelsonite it has been pretty well demonstrated that while ilmenite can be fairly easily removed from apatite by either of the methods outlined above it is practically impossible to effect a clean separation by mechanical means, since the nature of the rock is such that there will always be particles of the two minerals which adhere strongly to each other.

We must, therefore, resort to chemical means to bring about this desired end. In the experiments described below it is shown that ilmenite may be almost completely freed from apatite by means of sulfuric acid, with a minimum amount of waste and without involving any great expense. Moreover, all of the products obtained during the process can be utilized commercially.

EXAMINATION AND SEPARATION OF THE MINERALS IN NELSONITE

A typical sample of the rock was submitted to a microscopic examination by Mr. W. H. Fry of this Bureau and found to consist essentially of crystallized ilmenite and partially decomposed apatite. The material was then crushed to pass a $\frac{1}{2}$ mm.

sieve and a separation made by means of a strong magnet. Owing to the rather feeble magnetic qualities of part of the ilmenite this separation was made at the expense of the apatite which was afterwards found to contain a considerable amount of the former mineral in a finely divided state.

The results of this separation are given below:

	Per cent
Nelsonite as separated by magnet in the laboratory contained.....	
Apatite.....	23.8
Ilmenite....	76.2

Both the ilmenite and apatite thus separated were analyzed by Mr. C. F. Miller of this Bureau, with the following results:

	Per cent
Ilmenite as separated by magnet in the laboratory contained.....	
FeO.....	44.2
TiO ₂	44.5
P ₂ O ₅	0.8
Apatite as separated by magnet in this laboratory contained.....	
Fe ₂ O ₃	6.5
P ₂ O ₅	33.3
Ca ₃ (PO ₄) ₂ ...	72.8

Although the amount of phosphoric acid remaining in the separated ilmenite was less than 1 per cent, such a clean separation could hardly be attained in actual works practice. Even this amount of phosphoric acid is objectionable and would probably seriously affect the sale of the material.

Preliminary experiments showed that ilmenite is almost entirely unattacked by dilute sulfuric acid, while it is well known that apatite is acted upon quite energetically by the same acid: so 3 samples of the separated ilmenite of 10 grams each were treated with sulfuric acid of different strengths. The mixtures were warmed slightly and stirred intermittently for 20 minutes. At the end of that time the acid was diluted with water, filtered and washed. The filtrate was made up to 200 cc. and the percentages of iron and phosphoric acid thus dissolved determined in the two solutions. The residual ilmenite was again weighed and also analyzed for phosphoric acid.

The results of these analyses are given in Tables I and II.

TABLE I—ANALYSIS OF SULFURIC ACID EXTRACTS OF ILMENITE

3 samples of 10 grams each treated with 50 cc. of sulfuric acid of various strengths.

Strength of H ₂ SO ₄ used	Amount of P ₂ O ₅ extracted by acid Per cent	Amount of Fe ₂ O ₃ extracted Per cent
50° B.	0.22	0.19
32° B.	0.46	0.29
25° B.	0.41	0.32

TABLE II—ANALYSIS OF ILMENITE AFTER EXTRACTION WITH SULFURIC ACID

Strength of acid used in extraction	Loss in weight of ilmenite Per cent	Amount of P ₂ O ₅ remaining in ilmenite Per cent
50° B.	1.04	0.47
32° B.	1.75	0.31
25° B.	1.58	0.36

From the data given in Tables I and II it will be seen that the apatite remaining in ilmenite after the mechanical separation can nearly all be extracted by means of dilute sulfuric acid (32° B.) without appreciably affecting the ilmenite. The amount of iron

¹ "Mineral Resources of Virginia," 1907.

thus dissolved is too small to lower the value of the acid extract which can be used in treating the separated apatite to produce superphosphate.

While this acid extract is considerably weaker than that employed in the manufacture of acid phosphate it could be readily brought to the desired concentration, either by evaporation, or by mixing it with the sulfuric acid which is shipped in tank cars; this latter acid has a strength of 60° B.

The apatite as separated in the laboratory contained a considerable amount of iron, but this impurity should cause no trouble in the making of superphosphate since only the compounds of this element which are soluble in sulfuric acid cause the objectionable reversion to take place or render the acid phosphate too sticky for use.

ECONOMIC CONSIDERATIONS

If both the ilmenite and apatite can be disposed of at a fair price the mining of nelsonite should prove quite profitable. Unfortunately the market for ilmenite is rather limited at present, and it is doubtful whether the steel industry would handle the large tonnage which extensive development could produce. If nelsonite could be depended upon to yield an average of 50 per cent of apatite the exploitation of the rock for this mineral alone would be well worth while, but it is doubtful if the general mine run contains such a high percentage of apatite.

The production of apatite from nelsonite would undoubtedly be more expensive than the mining and preparation of Florida phosphate, since the rock is quite hard and would require rather fine grinding before a separation could be made. On the other hand, the average content of phosphate rock in nelsonite is considerably higher than it is in the Florida deposits. The amount of phosphate rock actually marketed from this latter state is probably not more than 15 per cent of the total material taken from the mines; in other words, every ton of phosphate produced involves the handling of over 6 tons of waste material.

The following figures, while approximate, are conservative and give some idea of the cost of mining and separating the two minerals in nelsonite:

TABLE III—COST OF MINING NELSONITE AND MAKING A MECHANICAL SEPARATION OF THE TWO MINERALS, APATITE AND ILMENITE PER TON

Cost of mining.....	\$1.00
Cost of grinding.....	0.50
Cost of mechanical separation.....	0.25
Total cost.....	\$1.75

Assuming that 1 ton of nelsonite will yield 1/2 ton each of ilmenite and apatite then the cost of preparing this amount of ilmenite for the market will be as follows:

TABLE IV—COST OF PREPARING ONE-HALF TON OF ILMENITE FOR THE MARKET

1/2 expense of mining and separating minerals.....	\$0.88
1/2 ton of sulfuric acid (60° B.) at \$8.00 per ton.....	4.00
Cost of washing ilmenite (1/2 ton).....	0.30
Cost of drying ilmenite (1/2 ton).....	0.15
Total cost of 1/2 ton ilmenite.....	\$5.33

The sulfuric acid extract after concentration can

be used in making acid phosphate by mixing it with an equal weight of the separated apatite.

The cost of manufacturing this product would be approximately as follows:

TABLE V—COST OF MAKING ONE TON OF ACID PHOSPHATE FROM ONE-HALF TON OF APATITE SEPARATED FROM NELSONITE

1/2 expense of mining and separating minerals.....	\$0.88
1/8 ton of sulfuric acid (60° B.) (cost included in expenses for cleaning ilmenite).....	...
Cost of drying apatite (1/2 ton).....	0.15
Cost of mixing and handling.....	0.50
Total cost of 1 ton acid phosphate.....	\$1.53

The total cost of mining 1 ton of nelsonite (having the above composition) and producing therefrom 1/2 ton of ilmenite (practically free from phosphorus), and 1 ton of acid phosphate (16 per cent P₂O₅) would be (exclusive of interest on investment, insurance and taxes) about \$6.86 per ton—an amount which would be more than covered by the value of the acid phosphate produced.

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QUANTITATIVE DETERMINATION OF ROSIN IN PAPER

By C. FRANK SAMMET
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As the necessity for securing record papers so free from chemicals and deleterious substances that they may be preserved indefinitely becomes more apparent, the importance of the determination of the sizing materials in the papers, particularly rosin, increases. Obviously, the resistance of the cellulose fibers to the action of light, heat, moisture and fumes is greatest in paper free from harmful materials. In carefully prepared specifications for durable papers it is customary to limit the percentage of rosin which may be present, as this substance, especially when used in large quantities, materially hastens discoloration and deterioration. Limits for rosin are now included in government paper specifications.

ALCOHOL METHODS

Of the numerous methods used for estimating the amount of rosin in paper, many are based on its extraction with alcohol, modifications being introduced in the subsequent treatment of the solution containing the extracted rosin.

Turbidity Method.—The turbidity produced by the precipitation of rosin from solution, on diluting the alcoholic extract with water, is compared with the turbidity produced in a like manner in an alcoholic solution, the rosin concentration of which is known. Although in many cases 0.0002 gram differences in the same solution may thus be detected, it was found that in some papers the determinations varied 25 or even 50 per cent from the actual amount of rosin present. Since more than 0.006 gram of rosin precipitated in 100 cc. of water interferes with the delicacy of the comparison, the quantities used must be so small that the percentage error is greatly affected by the uniformity of precipitation, which depends upon such factors as the sequence of operation, time allowed for precipitation and subsequent comparison,