

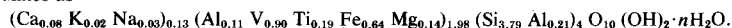
# VANADIUM–TITANIUM-BEARING MIXED-LAYERED CLAY FROM POTASH SULPHUR SPRINGS, ARKANSAS

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**Abstract**—A unique vanadium-titanium-bearing mixed-layered clay is currently being mined as an ore of vanadium at Potash Sulphur Springs, AR. The clay contains 80% expandable portion and has both a 14.22 and 18.24 Å hydration state in addition to the 9.49 Å dehydrated state; ethylene glycol expands this material to 16.69 Å. The 060 reflection for all hydration states is between 1.50 and 1.51 Å indicating the material is dioctahedral. The “mean chemical formula” calculates as



**Key Words**—Mixed-layer, Titanium, Vanadium.

## INTRODUCTION

The Union Carbide Company has been operating a vanadium mine in the fenitized zone of the Potash Sulphur Springs alkali intrusion since 1960. Chemical analyses on bulk samples of the ore, which is composed predominantly of an expandable clay plus minor amounts of other included minerals, principally alkali feldspar, rutile and apatite, yield average values of 2%  $\text{V}_2\text{O}_5$  and 1%  $\text{TiO}_2$ . As of this time there is no reported occurrence of a clay containing vanadium or titanium as major cation constituents. Minor amounts of titanium have been reported such as in the Amory montmorillonite (Grim, 1968; Ross and Hendricks, 1945). This current study was two-fold: (1) to determine if vanadium or titanium are in the clay structure and (2) if vanadium and titanium are in the clay, to determine the chemical analysis and lattice parameters of the clay.

## GEOLOGY OF THE POTASH SULPHUR SPRINGS COMPLEX

The Potash Sulphur Springs igneous intrusion is located in sections 17 and 18, T3S, R18W in Garland County, AR, and is situated on the nose of a southwest plunging anticlinal fold in the Zig Zag Mountains. It is a discordant alkalic igneous intrusion 1.2 k in diameter and roughly circular in shape, which is surrounded on the north, east and west predominantly by Arkansas novaculite and on the south by Stanley shale. The intrusion has generally been considered to be a ring-dike similar in composition to the Magnet Cove complex; chemical and petrographic investigations, however, indicate that the intrusion is a “differentiated syenite stock which was later intruded by mafic bodies” (Howard, 1974). The core and western rim of the complex consists of feldspathoidal syenite while the remainder of the rim consists of leucopulaskite and pulaskite. Carbonatite, ijolite, malignite, naujaite and numerous lamprophyre dikes are disseminated within the complex (Figure 1). Geophysical evidence indicates a south-

eastward dip to the complex (Pollock, 1965). An accurate age determination for the Potash Sulphur Springs intrusive is lacking; however, the date is generally assumed to be analogous to that of the Magnet Cove intrusion which has been determined as being  $98 \pm 10$  million yr before present (late Cretaceous) (Erickson and Blade, 1963; Miller, 1969; Naeser and Paul, 1969).

Fenitization (alkali metasomatism), which followed the emplacement of the intrusion, altered the eastern rim of the nepheline syenite to a pulaskite and leucopulaskite. This fenitization extended outward into the surrounding sediments and is most noticeable in the Arkansas novaculite, which originally had an average composition of 99% silica (Purdue and Miser, 1923). Mineralogic studies of the fenitized novaculite zones have shown a replacement of the quartz grains by alkali feldspar (probably orthoclase), with the boundary between the fenite zone and the pulaskite–leucopulaskite marked by the presence of large lath-shaped alkali feldspar grains (Heathcote, 1976). Once the quartz is completely replaced by the alkali feldspar, further fenitization “involves a recrystallization of the alkali feldspar and simultaneous development of fluorapatite” (Heathcote, 1976). Heathcote (1976) believes the fenitizing agent to be a highly fluid phase of the original nephelinitic magma composed chiefly of those elements generally thought to be concentrated in the fluid phase as a magma cools and differentiates. At Potash Sulphur Springs, the particular elements introduced during fenitization are potassium, sodium, aluminum, calcium, iron, phosphorus, fluorine and sulphur. The fenitization is believed to be followed by a carbonate phase. Evidence for this is interpreted from the fact that the “carbonate stringers (carbonate–pyrite veinlets) are always found cutting fenitized rock, but fenitized rocks are not always cut by carbonate stringers” (Heathcote, 1976). This carbonate phase is believed to be the medium through which the vanadium and titanium were introduced into the area.

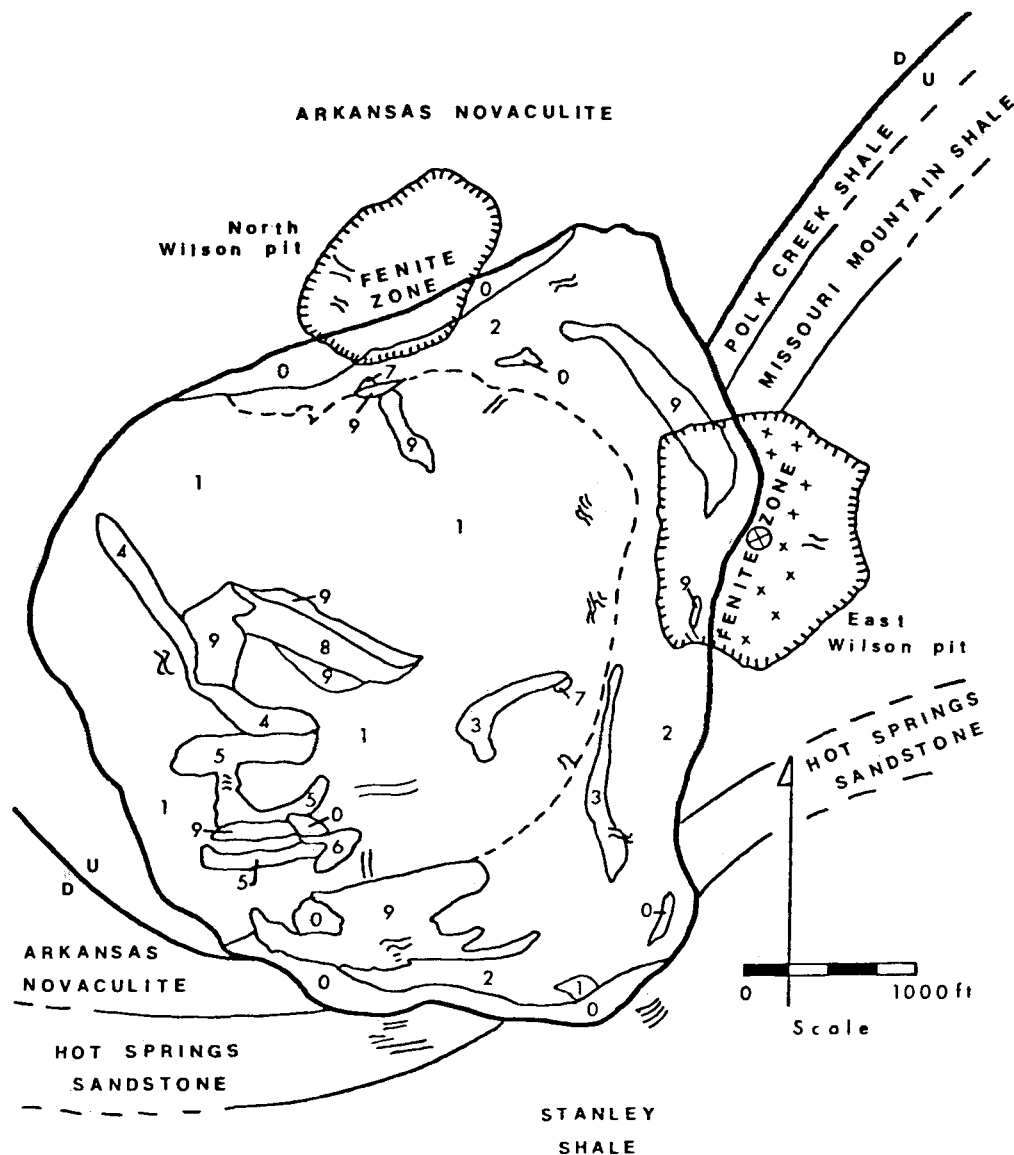


Fig. 1. Map of Potash Sulphur Springs igneous complex (modified from Howard, 1974). Explanation: (0) metamorphosed contact rocks and xenoliths, (1) feldspathoidal syenite, (2) pulaskite-leucopulaskite, (3) ijolite, (4) malignite, (5) fasinite, (6) naujaite, (7) nordsojite, (8) carbonatite, (9) lamprophyres, (x) breccia, (x) sample collection site.

The altered zone is developed continuously near the pulaskite contact but becomes patchy and degenerates into stringers after about 45 m (150 ft) from this contact; within this patchy zone, fenitized areas tend to follow fractures. Fenitization is evident for at least 300 m (1000 ft) from the pulaskite contact (Heathcote, 1976), and appears to be associated with a carbonatite phase. The carbonatite is usually injected into the alkaline igneous complex near the end of its cooling sequence (Heinrich, 1966); this process usually takes place under constant volume. The mechanisms by which the rocks are fenitized are still unknown, although mineralogical evi-

dence seems to indicate a moderate temperature (400–600°C) and highly volatile alkali-halide fluids interacting with minerals of the country rock (Tuttle and Gittins, 1966).

Vanadium-bearing clay occurs along the contact between the leucopulaskite-pulaskite and the altered novaculite and extends outward into the novaculite for about 15 m (50 ft). The vanadium concentration in the altered area is zoned with the highest concentration along the contact between the leucopulaskite-pulaskite rocks and the fenitized novaculite. The Union Carbide Company has developed two open pit mines—the

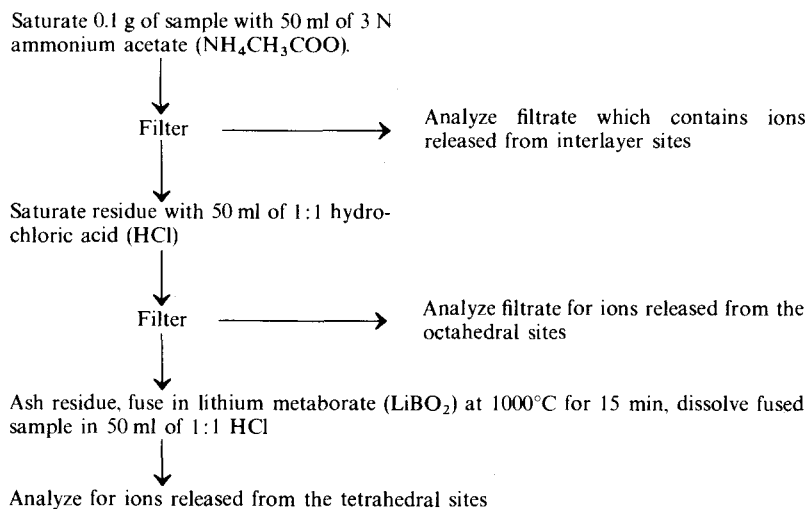


Fig. 2. Flowchart of acid dissolution method of chemical analysis.

North Wilson and East Wilson pits—on the border of the complex in order to mine this vanadium-bearing clay.

## ANALYTICAL METHODS

### Sample preparation

Samples chosen for analyses were collected from the East Wilson pit at the approximate contact between the fenitized zone and the leucopulaskite-pulaskite rocks (Figure 1). The raw samples were disaggregated in 1000 ml of water in an electric mixer and allowed to stand for sufficient time to allow the sand fraction ( $>62 \mu\text{m}$ ) to settle out according to Stoke's law. The clay and silt fractions left in suspension were siphoned off and size fractionated by centrifugal sedimentation. When dry, the sediment was split in half; one half to be used for X-ray analysis and the other half for chemical analysis.

The sample fractions to be used for chemical analysis were dried at  $125^\circ\text{C}$  and weighed, the process being repeated until a constant weight was obtained, then placed in a desiccator over calcium chloride. The sample fractions to be used for X-ray diffraction were dispersed in distilled water, allowed to settle for 15 min and then put on ceramic plates by suction; these samples were then placed in the desiccator with the dried samples. A small amount of the samples dried for chemical analysis were loaded in both glass capillaries for use in a Debye-Scherrer camera and in holders for use on a Guinier camera.

### X-ray analyses

All X-ray powder diffraction analyses were performed on a Philips APD 3500 diffractometer with  $\text{CuK}\alpha$  radiation. This automated diffractometer is equipped with a two-theta compensating slit and a

graphite monochromator which yield a low background down to  $1.5^\circ 2\theta$  and very sharp resolution of diffraction peaks.

Samples were analyzed on the diffractometer in four different environmental states: (1) after heating at  $700^\circ\text{C}$  for 2 hr, (2) room temperature and 15% r.h., (3) room temperature and 100% r.h. (achieved by placing water directly on to the sample just before X-raying), and (4) after exposure to an ethylene glycol atmosphere for 24 hr. All samples were run from  $2^\circ$ – $64^\circ 2\theta$  using a peak search program developed by Philips Electronics.

Samples run in both the Debye-Scherrer and Guinier cameras were subjected to the same four environments as were the diffraction samples.

### Chemical analyses

An acid-dissolution method for separating the structural units of silicates was first suggested by Rader et al. (in Belt, 1967). Variations of this method have been successfully used by Belt (1967), Brindley and Youell (1951), Gastuche and Fripiat (1962), Osthaus (1956), McClelland (1973), and McCormick (1975); a three-step dissolution was used in this study (Figure 2). The ammonium acetate, hydrochloric acid and dissolved lithium borate flux solutions were each analyzed for Si, Al, V, Ti, Fe, Mg, Ca, K and Na by means of atomic absorption using the standard curve method.

Kinetic studies on solution rates were made for all nine elements in the ammonium acetate and hydrochloric acid solutions to determine length of time and number of repetitions of solution treatment needed to remove each ion from its structural site. Ammonium acetate solutions were allowed to stand for 24 hr at room temperature then decanted. The decanted fluid was analyzed for all nine elements and the residue was put in fresh ammonium acetate solution and the cycle

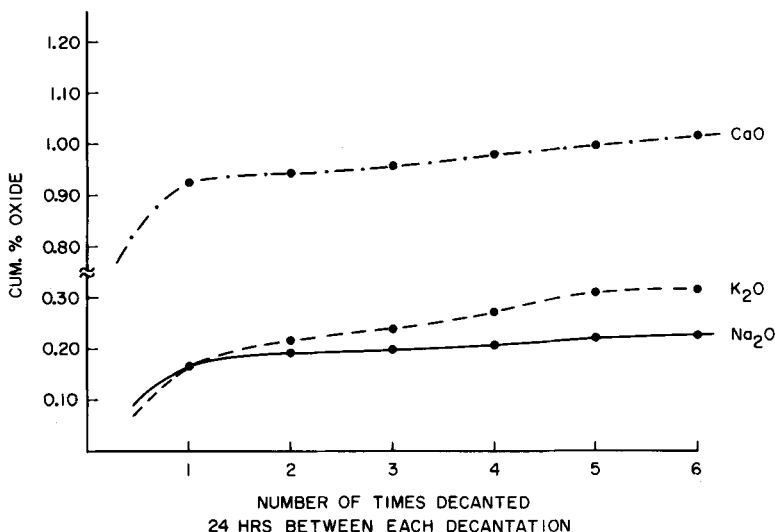


Fig. 3. Rate of solution of Ca, K and Na from sample S3 in 3 N ammonium acetate solution.

repeated six times. Only Ca, K and Na were detected in the solutions (Figure 3).

The hydrochloric acid solutions were heated at 90°C for 2 hr then decanted. The filtrate was analyzed for all nine elements and the residue was put in fresh HCl and the cycle repeated 12 times (Figure 4); only V, Ti, Fe, Mg, Si, Al and Ca were detected in these solutions.

Bulk samples of each split were fused with lithium metaborate and dissolved in 1:1 HCl; the solutions then analyzed for all nine elements. The totals for each element obtained from the separating procedure were compared to the totals of the bulk sample to insure accuracy. A standard (OB-1) basalt supplied by Iowa State University was analyzed and the values we ob-

Table 1. X-ray Data for the 9.49A°, 14.22A°, 18.24A°, and Glycolated phases of the V-Ti-Mixed-Layered Clay at 1 atm. and 25°.

9.49A°				14.22A°				18.24A°				GLYCOLATED			
hkl	calc	obs	I**	hkl	calc	obs	I	hkl	calc	obs	I	hkl	calc	obs	I
001*	9.415	9.49	S	001*	14.195	14.2	S	001	18.742	18.2	VS	001*	16.743	16.7	VS
002*	4.708	4.80	BW	003	4.732	4.75	VW	002	9.371	9.3	BM	002	8.372	8.4	M
020	4.544	4.55	M	020*	4.500	4.50	M	003	6.247	6.3	WB	003	5.581	5.6	BW
111	3.895	3.79	M	111	4.229	4.23	W	004	4.686	4.7	VVBW	020	4.525	4.51	M
112	3.514	3.56	M	022	3.801	3.77	M	020	4.533	4.51	M	004	4.186	4.25	W
022	3.267	3.34	W	004	3.549	3.55	M	112	4.290	4.29	W	113	3.509	3.51	M
003*	3.138	3.17	VSB	113	3.465	3.36	M	005	3.748	3.70	VW	005*	3.349	3.36	M
112	3.078	2.93	W	114	2.950	2.95	W	113	3.532	3.56	M	114	3.050	2.95	W
201	2.602	2.60	M	005	2.839	3.18	W	114	3.347	3.34	M	006*	2.791	2.81	W
131	2.464	2.46	W	202	2.585	2.59	W	006*	3.124	3.13	BM	131	2.602	2.58	BW
114	1.990	1.98	M	118	1.713	1.71	W	115	2.953	2.95	W	007*	2.392	2.39	W
222	1.920	1.92	M	206	1.672	1.67	W	201	2.589	2.59	W	028	1.900	1.90	W
134	1.828	1.83	M	060*	1.500	1.51	M	116	2.458	2.46	W	311	1.803	1.82	W
311	1.720	1.72	M	064	1.382	1.38	W	206	1.917	1.91	W	151	1.714	1.71	W
151	1.670	1.67	W					050	1.813	1.82	W	0010	1.674	1.67	M
225	1.5419	1.55	W					311	1.708	1.71	M	060*	1.508	1.51	M
060*	1.515	1.51	BW					242	1.674	1.67	M	1112	1.357	1.36	W
332	1.388	1.38	W					060*	1.511	1.51	M				
								249	1.380	1.38	W				

a <sub>o</sub> (A°) = 5.240 ± 0.009	a <sub>o</sub> (A°) = 5.341 ± 0.002	a <sub>o</sub> (A°) = 5.349 ± 0.008	a <sub>o</sub> (A°) = 5.525 ± 0.01
b <sub>o</sub> (A°) = 9.090 ± 0.009	b <sub>o</sub> (A°) = 9.001 ± 0.003	b <sub>o</sub> (A°) = 9.065 ± 0.006	b <sub>o</sub> (A°) = 9.051 ± 0.007
c <sub>o</sub> (A°) = 9.537 ± 0.043	c <sub>o</sub> (A°) = 14.292 ± 0.004	c <sub>o</sub> (A°) = 18.862 ± 0.023	c <sub>o</sub> (A°) = 16.775 ± 0.009
β angle = 99° 41' ± 12'	β angle = 96° 40' ± 2'	β angle = 96° 45' ± 7'	β angle = 93° 32' ± 10'

\* Pre-indexed spacings for cell refinements.

\*\* Intensity of peaks: S = strong, M = medium, W = weak, V = very, B = broad.

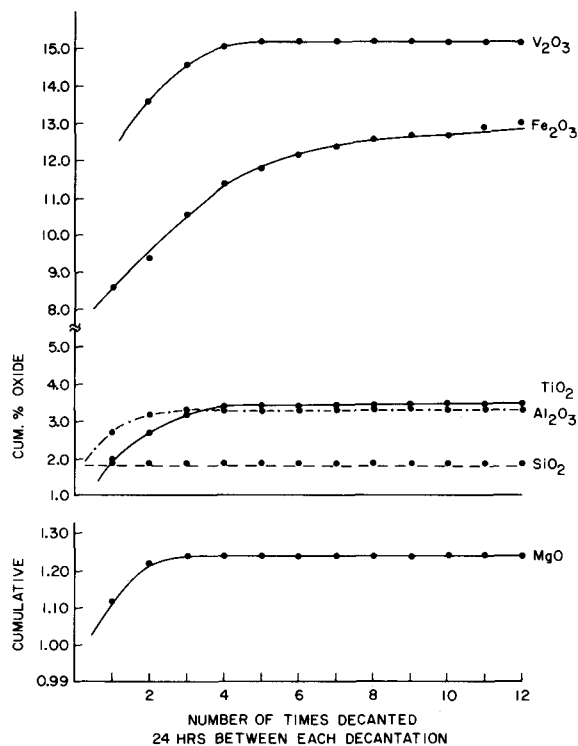


Fig. 4. Rate of solution of V, Fe, Ti, Al, Mg and Si from sample S3 in 1:1 HCl solution.

tained were checked against their values to insure analytical accuracy.

#### SEM analysis

A number of clay samples were examined by SEM (Figure 5) and mapped for V, Ti, Fe, Mg and Ca using an Ortec energy dispersive system attached to the SEM. Mapping for each of these elements yielded only general scatter and gave no area of concentration for any of the elements.

#### DISCUSSION OF RESULTS

X-ray powder diffraction studies showed the clay to have three states of hydration (18.24, 14.22 and 9.49 Å) in addition to a 16.69 Å phase when saturated with ethylene glycol (Figures 6a–d). The basal spacings obtained from X-ray powder diffraction of all hydration states are irrational series, thus indicating that the clay is mixed-layered.

Table 1 lists all diffraction lines from 2 to 64° 2θ for each hydration and glycolated state. All *hk0* reflections were obtained from both Debye-Scherrer and Guinier photographs. 001 spacings down to 14 Å were obtained from Debye-Scherrer films; those below 14 Å had to be obtained from the diffraction charts.

All interplanar spacings for each hydration and glycolated state were submitted to a cell refinement and indexing program. The observed *d*-spacings, calculated *d*-spacings, cell edges and interaxial angles obtained from this program are listed in Table 1. This author is fully aware that one cannot accurately index a mixed-layered clay or determine accurate cell dimensions for such a clay in this manner. An additional problem is that many of the reflections are broad and/or weak. The indexed values obtained from the program are very close to the observed values and thus this author feels that the listing in Table 1 gives a better overall picture of the various hydration states than would a mere listing of observed *d*-values. The three decimal place values obtained from the computer have been left in Table 1; however, accuracy certainly cannot be claimed much above the first decimal place.

The  $a_0$  and  $b_0$  values differ little between the four phases and it is the  $c_0$  and  $\beta$  values which vary as one would expect. The 060 reflection in all cases is between 1.50 and 1.51 Å, indicating that the clay is dioctahedral (Warsaw and Roy, 1961).

Kinetic studies described earlier (Figures 2 and 3) yield convincing evidence that all Ca, K and Na are re-

Table 2. Comparison of analyses for separated sample S3 and bulk sample S3. Values expressed as % oxides.

	Separated sample S3				Bulk sample S3
	Interlayer	Octahedral	Tetrahedral	Total	
SiO <sub>2</sub>	0.0	1.92	54.20	56.12	55.66
Al <sub>2</sub> O <sub>3</sub>	0.0	2.72	3.14	5.86	3.96
V <sub>2</sub> O <sub>3</sub>	0.0	14.62	0.0	14.62	16.50
TiO <sub>2</sub>	0.0	3.22	0.0	3.22	3.71
Fe <sub>2</sub> O <sub>3</sub>	0.0	12.71	0.0	12.71	12.51
MgO	0.0	1.37	0.0	1.37	1.39
CaO	0.94	tr	tr	0.94	1.14
K <sub>2</sub> O	0.28	0.0	0.0	0.28	0.27
Na <sub>2</sub> O	0.21	tr	tr	0.21	0.24
<b>Total</b>				<b>95.33</b>	<b>95.38</b>

tr, trace amounts detected.

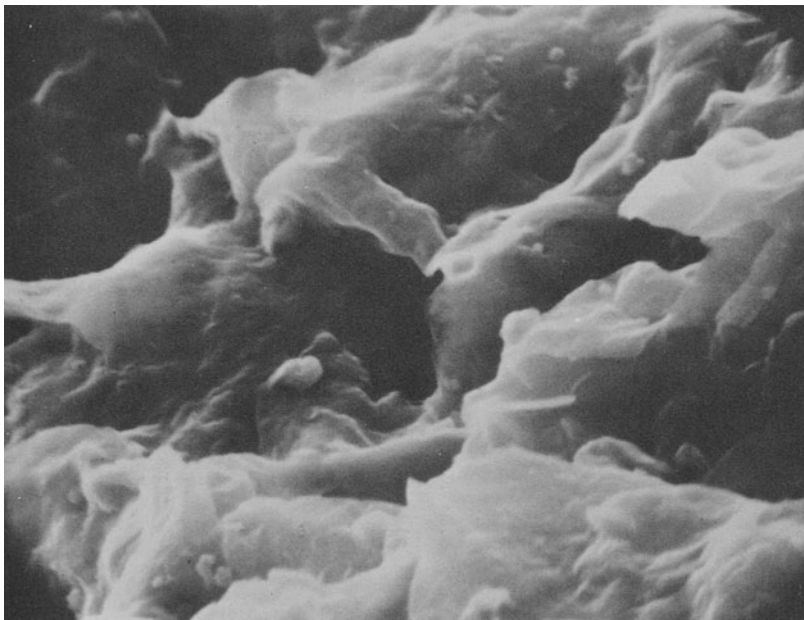


Fig. 5. SEM photograph of the V-Ti-bearing mixed-layered clay magnified 11,000 $\times$ .

leased from interlayer sites by ammonium acetate. The studies also show that when the octahedral layer is destroyed by 1:1 HCl, V, Mg, Ti, Fe and Al are released. Approximately 2% SiO<sub>2</sub> and a slight excess of Al<sub>2</sub>O<sub>3</sub> which theoretically should not be in octahedral positions are also found. Careful X-ray analyses were made on clay samples prior to chemical analysis to insure that no quartz, feldspar, rutile, or apatite were present; none was found. The presence of the silicon and excess aluminium then is most likely due to broken bonds around the edges of the tetrahedral layer.

Tables 2 and 3 contain the results of the chemical

analyses of the three-step dissolution process and bulk sample analyses of sample S2 and S3, respectively. It is apparent that the two bulk sample analyses are in good agreement; however, some of the totals of the separated samples, in particular, V<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub>, are not in good agreement with the bulk sample values. This author has used the oxide values from the separated samples only to determine in which sites the elements occur; the chemical formula has been calculated using the bulk sample values. The difference between the totals in Table 2 and 3 and 100% is assumed to be H<sub>2</sub>O<sup>+</sup>.

Table 3. Comparison of analyses for separated sample S2 and bulk sample S2. Values expressed as % oxides.

	Separated sample S2				Bulk sample S2
	Interlayer	Octahedral	Tetrahedral	Total	
SiO <sub>2</sub>	0.0	2.01	54.07	56.08	55.74
Al <sub>2</sub> O <sub>3</sub>	0.0	2.90	2.55	5.45	4.33
V <sub>2</sub> O <sub>3</sub>	0.0	15.08	0.0	15.08	17.16
TiO <sub>2</sub>	0.0	4.90	0.0	4.90	3.69
Fe <sub>2</sub> O <sub>3</sub>	0.0	13.17	0.0	13.17	12.60
MgO	0.0	1.66	0.0	1.66	1.30
CaO	1.10	tr	tr	1.10	1.24
K <sub>2</sub> O	0.31	tr	tr	0.31	0.30
Na <sub>2</sub> O	0.22	tr	tr	0.22	0.21
<b>Total</b>				<b>95.33</b>	<b>96.57</b>

tr, trace amounts detected.

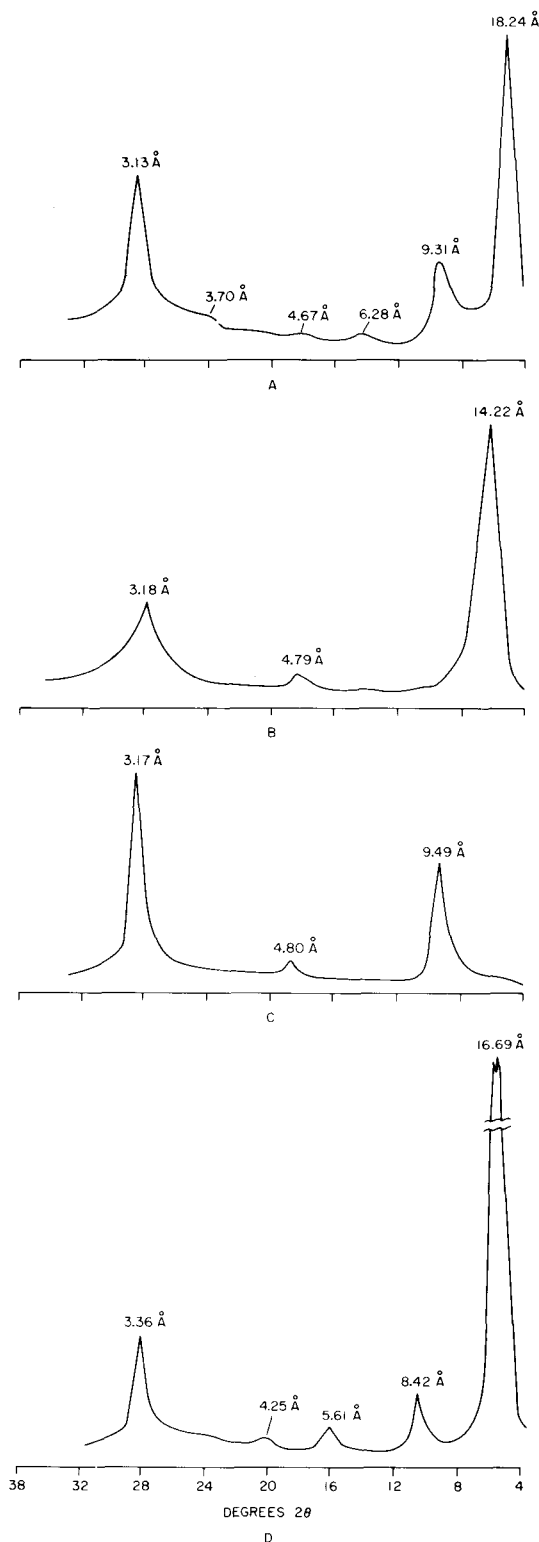


Fig. 6. X-ray powder diffraction tracings of 001 reflections of oriented slides. (a) 18.24 Å phase, (b) 14.22 Å phase, (c) 9.49 Å dehydrated phase, (d) 16.69 Å glycolated phase.

## CONCLUSIONS

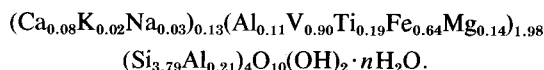
The X-ray analyses of the clay yield the following results:

(1) basal spacings are an irrational series; (2) the glycolated 001 reflection is at 16.69 Å; (3) the dehydrated 001 reflection is at 9.49 Å; (4) two states of hydration (14.22 and 18.24 Å) are present; (5) the 060 reflection occurs between 1.50 and 1.51 Å.

These results show the clay is a randomly mixed-layered clay containing 80% swelling fraction (Weaver, 1956). The presence of only a two- or three-layer hydration state in addition to the dehydrated state indicates that calcium is the predominant interlayer cation (Brown, 1961). The 060 reflection at 1.50–1.51 Å shows the clay to be dioctahedral (Warsaw and Roy, 1961).

Weaver (1965) points out that a single structural formula will not suffice for a mixed-layered structure. Hower and Mowatt (1966) suggest that a "homogeneous" single structural formula represents the mean 2:1 composition. They further state that until the nature of mixed layering can be examined in more detail it is sufficient to write a "mean" structural formula.

The "mean" formula derived from the analyses using the method of Brown and Norrish (1952) is:



## ACKNOWLEDGMENTS

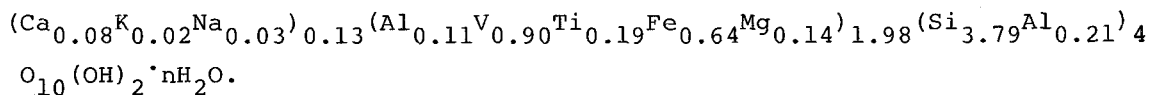
I wish to acknowledge the help of Paula Pritz and Richard Heathcote who performed many of the chemical and X-ray analyses.

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Резюме- Уникальная ванадий-титаносная смешанно-слоистая глина в настоящее время добывается в качестве руды ванадия на месторождении Поташ Сульфа Спрингс, Ар. Глина включает 80% расширяемой части и имеет гидратные состояния 14,22 и 18,24Å в дополнение к дегидратному состоянию 9,49Å. Этиленгликоль расширяет этот материал до 16,69Å. Отражение 060 для всех гидратных состояний находится между 1,50 и 1,51Å, указывая, что материал является диоктаэдрическим. "Средняя химическая формула" вычислена в виде:



Kurzreferat- Ein einmaliger, Vanadin-Titan haltiger, gemischt-Schicht Ton wird im Augenblick als Vanadinerz in Potash Sulphur Springs, Ar. abgebaut. Der Ton besteht zu 80% aus schwellbarem Teil, und hat nicht nur einen 14,22Å und einen 18,24Å Hydratationszustand, sondern auch einen 9,49Å Dehydratationszustand. Äthylenglykol expandiert dieses Material bis auf 16,69Å. Die 060 Reflektion für alle Hydratationszustände ist zwischen 1,50Å und 1,51Å, was andeutet, daß das Material dioktahedrisch ist. Die durchschnittliche chemische Formel wurde ausgerechnet als: