

Wyllieite, $\text{Na}_2\text{Fe}_2^+\text{Al}(\text{PO}_4)_3$, A New Species

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INTRODUCTION

After two field and collecting excursions to 102 Black Hills pegmatites in the summers of 1971 and 1972, it became clear that the Victory mine pegmatite was chemically unique, so peculiar in fact that a major mineralogical investigation is now in progress. Not only is the pegmatite of interest to the mineralogist for an unusual abundance of Na-rich primary phosphates rarely encountered elsewhere, but the textures of the cocrystallizing phases are in themselves a source of interest to petrologists as well. Our account concerns a new species which evidently occurred in considerable abundance during operation of the pegmatite.

The Victory mine pegmatite is located two miles northeast of the center of Custer, Custer County, South Dakota on the Custer Quadrangle, Section 18, SE 1/4, SW 1/4, T.3S, R.5E. It is one of the hundreds of pegmatites distributed around Harney Peak and mined during the Second World War. Compared with the dimensions of many other worked pegmatites, the Victory mine was small. According to Pray, et al. (1953) it cropped out as a lenticular form about 23' x 93' in dimension. Hanging wall and foot wall dip from 40° to 50°, defining a tabular sausage-like body with a length to width ratio of about 5:1 concordantly intruded into the enveloping schists. From 60' to 120' in depth, a second sausage-like pegmatite lens was encountered. Two shafts were driven, a 40' inclined shaft and a 70' vertical shaft, the latter eventually sunk to 120' with a crosscut to the pegmatite. Drifts facilitated removal of the rock.

The pegmatite consisted of two zones, a wall zone ranging from 1' to 7' in thickness and consisting of albite-quartz-muscovite which grades into the coarse quartz-perthite-albite core. High grade mica books were confined to the wall zone. Despite the small dimension of the pegmatite, it was the largest producer of high grade mica during 1944 and rewarded its owners with \$500,000 from 360 tons of mined and cobbled mica. The mica crystals ranged up to 18" across and were of prime ruby quality.

The interesting accessory minerals appeared to be confined to the region between the wall zone and the core. Pray et al. (1953) mention accessory tourmaline, blue phosphates (probably scorzalite) and triphylite-lithiophilite nodules (probably wyllieite) in the wall zone. No triphylites were encountered in our study. Pray et al. (1953)

mention that optical studies indicated a 70% triphylite composition. We have good evidence that this phase was indeed the wyllieite. A 70% triphylite would have a mean index of refraction around 1.695, the value found by Pecora and Fahey (1949) for their "triphylite" from the Victory mine, the sample of which we found to be in fact not triphylite but wyllieite. This index of refraction is practically identical to our observations on wyllieite. Finally, we remark that our wyllieite specimens show abundant coarse quartz and perthite as well as the plagioclase, suggesting that the mineral crystallized toward the end of the wall zone formation and during early core consolidation. Pray et al. (1953) mention that the "triphylite" nodules were rare but we find the dumps reveal a fairly abundant wyllieite. But the core of the Victory mine was probably investigated only casually since only the wall zone was of principal economic interest.

All specimens were collected on the dumps which had suffered severe diminution through back-filling and removal for road gravel. The entrances to the pegmatite's interior are largely caved in and the interior chamber filled with dump debris and water. We secured about 100 specimens for further study which shall form the basis of our chemical investigations on the Victory mine paragenesis and it is urged that what does remain of the dump be protected from total obliteration through cartage since to our knowledge aspects of its crystal chemistry are unique and the crystal textures spectacular.

One of us (PBM) has found that all geological reports which list accessory phosphate phases in the Black Hills pegmatites are notoriously unreliable in the correctness of mineral identification of these phases. Commonly grouped as "lithiophilite" or "heterosite" or simply "Fe, Mn phosphates", many are in fact in the triphylite-lithiophilite series, the arrojadite-dickinsonite series, the grafftonite-beusite series, the alluaudites, the heterosite-purpurite series, the ferrisicklerite-sicklerite series, the triplicate-zwieselite series, the triplodite-wolfeite series, griphite, etc. Studies on the compositional variation of the Fe:Fe+Mn ratio within a pegmatite and among pegmatites, important problems which bear on theories of pegmatite genesis, are virtually absent in the literature. In fact, the labyrinthine mineralogical problem of secondary phosphate paragenesis and chemistry is closely rivalled by the

TABLE 1 CHEMICAL ANALYSES OF WYLLIEITE AND ARROJADITE FROM THE VICTORY MINE

	1	2	3	4	5	6	7
K ₂ O	0.05	0.0	-	-	1.09	1.80	2
Na ₂ O	8.0	9.75	8.9	5.26	7.55	19.44	18
Li ₂ O	0.01	n.d.	-	-	n.d.	-	
H ₂ O ⁺	0.70	n.d.	0.7	1.44	n.d.	-	
CaO	2.5	2.21	2.3	0.77	5.39	7.68	8
MgO	1.97	1.80	1.9	0.86	1.38	2.72	9
MnO	4.3	3.62	4.0	1.03	3.62	4.12	
FeO	29.2	29.9	29.6	7.58	37.01	41.20	10
ZnO	0.04	n.d.	-	-	0.0	-	
Al ₂ O ₃	7.9	8.46	8.2	2.96	2.85	4.48	4
Fe ₂ O ₃	0.33	n.d.	-	-	n.d.	-	
SiO ₂	0.8	0.0	-	-	0.0	-	
P ₂ O ₅	43.8	45.27	44.5	11.57	42.26	48.00	15
	99.56	100.99	100.1		101.15		

¹ Wet chemical analysis on wyllieite in weight percent. Jun Ito, analyst. W. K. Sabine provides 0.47% H₂O⁺ determined calorimetrically. Spectrographic traces of Sr, Ba, Pb, and Be were detected.

² A.R.L. electron probe microanalysis. G. R. Zechman, analyst.

³ Average of 1 and 2.

⁴ Atoms in the unit cell.

⁵ ARL electron probe microanalysis on arrojadite from the Victory Mine. G. R. Zechman, analyst.

⁶ Atoms based on P = 48 for arrojadite.

⁷ Suggested ideal limits, atoms in cell.

chemical problems of the primary giant crystal phases. Much of this derives from the principal emphasis of geological reports during and after the war years (when pegmatites supplied virtually all the mica, beryllium and lithium minerals) on the economics of the deposits rather than their systematic crystal chemistry. Consequently, pegmatite structures were emphasized. This proves a boon to the mineral chemist since many excellent maps appeared in the literature and are especially important when a section of a pegmatite was obliterated by further operations and whose phases can be found only on the dumps. Broad surveys like Page et al. (1953) are rapidly becoming classics in the field of pegmatite description.

As noted before, the new species was recorded by Pecora and Fahey (1949) during their investigation on the Victory mine scorzalite but it was erroneously identified as triphylite. Identity with our new species was assured through an X-ray investigation on one of the samples used in their study and kindly donated by the late W. T. Pecora.

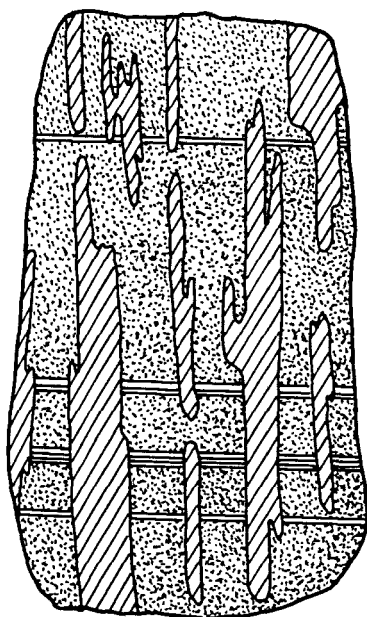
DESCRIPTION OF THE SAMPLES (PBM)

Phases identified in this study include major quartz, muscovite (some of high grade, occurring as thick ruby-red books), plagioclase showing superb lamellar twinning, perthite, albite var. cleavelandite, and schorl. Subordinate minerals, some occurring locally in considerable abundance, include wyllieite, graftonite, sarcopside, and arrojadite. More rarely appear scorzalite, arsenopyrite, hydration products of the primary phosphates such as ludlamite and vivianite, garnet and numerous inconspicuous uniden-

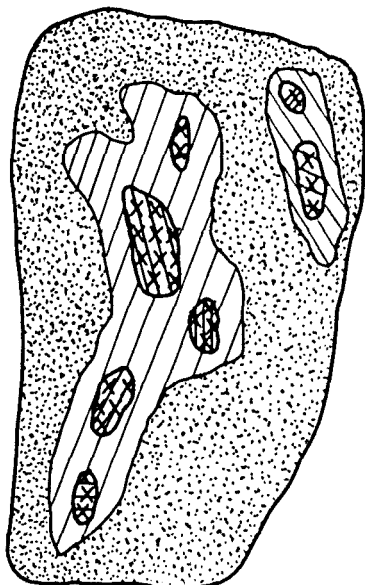
tified phases. Noteworthy is the persistent absence of lithium minerals. Instead sodium-rich phases appear.

The largest mass of wyllieite recovered from the dump weighed about 20 kilograms of which the mineral comprises 70% of the total mass. It occurs as coarse, rude euhedral interlocking crystals with one perfect cleavage of a deep greenish-black color, closely resembling hornblende. Earliest formed was muscovite and minor black tourmaline followed by the wyllieite and plagioclase and perthite feldspars, the former showing lamellar twinning and finally quartz. Several hundred kilograms of similar specimens were noted and doubtless the species was abundant *in situ*.

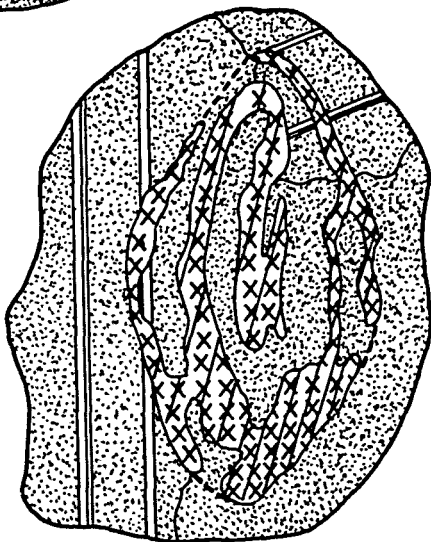
Most remarkable are the textures of arrojadite in association with the wyllieite. Arrojadite, its chemistry discussed later, was apparently completely overlooked by earlier investigators. It occurs as perfect parting surfaces of a pale greenish-yellow color, sometimes superficially altered wine yellow or orange. The arrojadite occurs as droplets and "fingers" whose extremities are crystallographically concordant to each other and immersed in or interfingering with crystallographically concordant plagioclase. Grain size of individuals, some of them showing incompleting crystal faces, range up to ten centimeters in length and two centimeters in thickness. More remarkable are vermiform intergrowths of rhythmic arrojadite and plagioclase, with a rude outline of the arrojadite morphology. Everywhere, thin films of wyllieite rim the arrojadite. Specimens exposing up to 150 square centi-



a. Arrojadite "fingers" (NE-SW lines) in plagioclase (stippled); twin lamellae of the feldspar crystal are shown as horizontal lines.



b. Graftonite-sarcopsid lamellar islands in arrojadite blebs. The arrojadite is embedded in plagioclase (stippled).



c. Crude outline of a graftonite crystal in plagioclase. The crystal is actually a connected boxwork of "fingers" showing concordant sarcopsid exsolution lamellae. Two plagioclase crystals are noted and their twin lamellae run continuously in each. Dashed lines show interrupted outline of the graftonite crystal.

1 2 3
scale in inches

Fig. 1. Sketches of hand specimens of phosphate-silicate intergrowths, Victory mine.

meters of crystallographically concordant patches of plagioclase and complementary arrojadite were collected.

Graftonite-sarcopsid intergrowths were also collected, showing the same textures in association with plagioclase as the arrojadite. In some instances, tourmaline "sausage rolls" occur growing parallel to the sarcopsid-graftonite laths. The scorzalite is later than the other phosphates and crystallized in the interstices left by the wylleite, or as isolated grains in mica aggregates.

Fig. 1a shows a sketch of an arrojadite-plagioclase intergrowth. Here, the parting planes of the arrojadite fingers are mutually parallel. The host mass of plagioclase shows broad twin lamellae. The twin lamellae of the plagioclase do not penetrate the arrojadite but are continuous through-

out the mass. Fig. 1b shows blebs of arrojadite in plagioclase, which poikilitically include graftonite-sarcopsid lamellae. Although the arrojadite mass is crystallographically uniform, the graftonite-sarcopsid blebs are not in themselves crystallographically concordant. Fig. 1c shows a graftonite-sarcopsid bleb in plagioclase. This bleb and others like it show a rude euhedral outline, the interior of which is constituted of connected crystallographically homogeneous material, with a texture like marble cake. Everywhere, the plagioclase is crystallographically concordant, as in Fig. 1a.

These textures, so reminiscent of graphic granite, cannot be explained solely on simple models of replacement or primary crystallization of one phase in the absence of the

TABLE 2. WYLLIEITE. CRYSTAL CELL CRITERIA

	1	2
$a(\text{\AA})$	11.868(15)	12.004(2)
$b(\text{\AA})$	12.382(12)	12.533(4)
$c(\text{\AA})$	6.354(9)	6.404(1)
β	114.52(8) $^\circ$	114.4(1) $^\circ$
$V(\text{\AA}^3)$	849.5 \AA^3	877.4 \AA^3
space group	$P2_1/n$	$C2/c$
cell formula	$\text{Na}_{5.26}\text{Ca}_{0.77}\text{Mg}_{0.86}\text{Mn}_{1.03}\text{Fe}_{7.58}^{2+}$ $\text{Al}_{2.96}(\text{P}_{11.57}(\text{H}_4)_{0.36})\text{O}_{46.95}$	$\text{Na}_{2.5}\text{Li}_{0.1}\text{Ca}_{0.5}\text{Mn}_{4.5}\text{Mg}_{0.2}\text{Fe}_{7.5}^{2+}[\text{PO}_4]_{12.0}$
specific gravity	3.601(3)	3.45
density(gm/cm 3)	3.60	3.62
sum of cations	30.5	27.7

¹ This study. The specific gravity was determined by a Berman torsion balance.

² Fisher (1955) for alluaudite from Buranga, Ruanda, Central Africa.

other. Rather, the patches of phosphate-silicate assemblages, their crystallographic concordance, the preservation of continuous exsolution profiles, and the morphological outlines suggest a phosphate-silicate eutectoid fluid which, prior to co-crystallization, possessed the property of a smectic liquid or gel permitting crystallographically oriented separation of individual phases like marble cake. These textures were repeatedly observed for arrojadite and graptone-sarcopside. Wyllieite, on the other hand, crystallizes idiomorphically against quartz, plagioclase and perthite when it is not rimming the arrojadite fingers. We sum up by proposing the paragenesis (from early to late) tourmaline-mica-arrojadite and plagioclase (graptone and sarcopside)-wyllyeite-scorzalite-perthite-quartz. Wyllieite evidently grew as homogeneous crystals as a primary phase and, like later scorzalite, is enriched in aluminum relative to the arrojadite.

PHYSICAL PROPERTIES

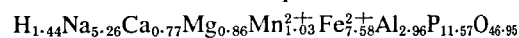
Wyllieite is deep bluish-green to deep oily green, greyish-green (not unlike triphylite) to greenish-black. Grains about 0.5 mm are transparent and pale green in color. The luster is vitreous to submetallic and the mineral lacks the shiny black surface of the associated tourmaline. The streak and powder are dirty olive-green, hardness 4+; cleavage {010} perfect with distinct {101}. The mineral is very brittle and removal of the quartz matrix around crystal surfaces usually results in shattering. In appearance, cleavage surfaces of wyllieite are strikingly similar to hornblende. The specific gravity is 3.601 ± 0.003 determined in triplicate on a Berman microbalance.

Crude crystals up to 10 cm across were encountered. Favorable fracture of the quartz occasionally reveals superior single crystals of the mineral. One specimen, 6 x 8 x 12 cm, shows a superior euhedral crystal 2 x 2 x 3 cm embedded in quartz and yellow mica. Morphologically, wyllieite crystals are similar to those of "hühnerkobelite" (= alluaudite group) illustrated by Moore (1965).

CHEMICAL COMPOSITION

Table 1 lists one partial and one complete analysis of wyllieite, and one partial analysis of arrojadite from the Victory mine. The wyllieite analyses, performed by two different techniques, show fairly good agreement when it is realized that composition from grain to grain may vary somewhat and that the larger samples contain poikilically included mica and quartz.

Average of the two analyses and computation based on the unit cell contents provide



Interpretation of the formula appears in the subsequent section on the X-ray analysis.

OPTICAL PROPERTIES

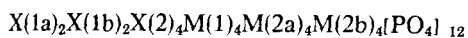
Wyllieite is biaxial (+). Owing to its weak birefringence, thick grains were studied in order to observe pleochroism and to obtain reliable indices. The results are $\alpha 1.688$, $\beta 1.691$, $\gamma 1.696$, all ± 0.002 , $2V(\text{obs}) \sim 50^\circ$, $r < v$ strong. Thick plates are pleochroic with $Z(\text{green}) > Y(\text{smoky bluish-green}) \sim X(\text{smoky bluish-grey})$. The mean calculated index of refraction from the specific refractive energies of Larsen and Berman (1934), the specific gravity and the average chemical composition is $\langle n \rangle_{\text{calc}} = 1.680$, in fair agreement with $(\alpha + \beta + \gamma/3) = 1.692$ and adds confidence to the cumulative data in this study. We note that the "triphylite" (= wyllieite) in Pecora and Fahey (1948) revealed $\beta = 1.690$, a large $2V$ and $Y = \text{blue-green}$, essentially in accord with our results.

X-RAY CRYSTALLOGRAPHY

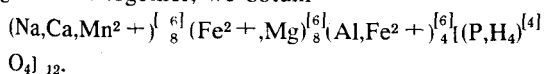
Fragments of wyllieite single crystals were prepared for Weissenberg photography. In addition, a thorough three-dimensional crystal structure analysis has been completed and allows us to interpret the wyllieite crystal chemistry with some reliability. Discussion of the structure, because of its inherent specialty, is deferred to a separate paper to be published elsewhere. The crystal cell parameters for wyllieite are presented in Table 2. The indexed

powder pattern, utilized for the refinement of the cell parameters, appears in Table 3. Correct assignment of indices was facilitated by the intensity measurements of the single crystal study.

The crystal structure analysis reveals that wyllieite possesses the general crystal-chemical formula



in the unit cell, where X are large cations whose oxygen coordination numbers are greater than 6, M are octahedrally coordinated cations, and P is in tetrahedral coordination. Least-squares refinement of the structure converged to $X(1a) = Na_{1.82} + \square_{0.18}$, where \square is a hole; $X(1b) = Ca_{1.0}^{2+} + Mn_{1.0}^{2+}$; $X(2) = Na_{2.78} + \square_{1.22}$; $M(1) = Fe_{3.0}^{2+} + Mg_{1.0}$; $M(2a) = Fe_{4.0}^{2+}$; and $M(2b) = Al_{3.0} + Fe_{1.0}^{2+}$ in the unit cell. Summation of the individual cationic species reveals a formula similar to the average obtained in the chemical analyses, a result which is at best approximate owing to the complex nature of the crystal structure. Ideally representing the compound as an end-member composition by grouping the large cations together, we obtain



With 4 such molecular units in the cell, the formula is condensed to

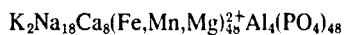


which we propose as the wyllieite limiting end-member composition with all cationic positions occupied in the structure.

We have investigated the thermal behavior of wyllieite in greater detail. A dehydration curve provided by W.K. Sabine for wyllieite powder in pure nitrogen atmosphere shows three distinct peaks at 525°C, 775°C and 955°C. The first peak accounts for 81.6% of the total H_2O^+ . It is possible that the loss of water at this stage is accompanied by the oxidation of Fe^{2+} to Fe^{3+} through cleavage of the protons by an auto-oxidation-reduction reaction. The peak at 775°C accounts for 14.8% additional water. Wyllieite powder was heated *in vacuo* at 775°C for 40 hours; X-ray examination of the product revealed increasing diffuseness and merging of the extra 'ordering' powder lines, demonstrating that solid solution of Al^{3+} and Fe^{2+} takes place at higher temperature. The peak at 955°C ended at 1040°C and accounted for 3.6% of the H_2O^+ . This terminal temperature is probably the melting temperature since Fisher (1965) notes that the "melting point of alluaudite is very close to 1000°C;" alluaudite is compositionally similar to wyllieite.

We also provide an analysis of Victory mine arrojadite in Table 1. The arrojadite differs in containing significant K_2O , more CaO , more FeO and less Al_2O_3 than wyllieite, but their compositions are sufficiently close to suggest a possible dimorphic relationship. Fisher (1965) observed that arrojadites heated in air begin to change to the alluaudite structure at 405°C. In vacuum, the compound apparently inverts to a ferrofillowite beginning about

745°C. It is clear that oxidation leads to alluaudite as the stable structure type at high temperature but fillowite forms instead *in vacuo*. It is possible that the presence of K_2O and the absence of oxidation of Fe^{2+} inhibits the formation of an alluaudite and fillowite forms instead. On the other hand, the absence of K_2O in wyllieite inhibits the growth of arrojadite through heating. Victory mine arrojadite contains the highest amount of Al^{3+} yet reported in the arrojadite-dickinsonite series. This series usually contains only minimal amounts of trivalent species. Based on the results of the analysis in Table 1, arrojadite from the Victory mine approaches the ideal limit



in the unit cell. But structure analysis will be necessary to better comprehend the chemistry of this complex series.

DISCUSSION

Wyllieite is structurally and compositionally related to the alluaudite group of minerals, a very complicated but important mineral group which occurs naturally as Na-metasomatic products of primary phosphate phases in granitic pegmatites (Moore, 1971). It differs from the alluaudites in the following ways, which are sufficient to warrant its specific status. *First*, it is a primary phase while alluaudites are derived from later Na-autometasomatic reactions of earlier phases, particularly triphylite-lithiophilite. *Second*, wyllieite occurs as idioblastic crystals while alluaudites are fine-grained nodular phases. *Third*, wyllieite crystallizes in space group $P2_1/n$ as a result of ordering over the general equivalent octahedral M(2) positions in the $C2/c$ space group of alluaudites. *Fourth*, Al^{3+} is an essential constituent in wyllieite and is minimal or absent in alluaudites. *Fifth*, the large alkali sites are extensively occupied in wyllieite, while they are largely empty in alluaudites. *Sixth*, the transition metals are unoxidized in wyllieite and occur in the Fe^{2+} and Mn^{2+} states. In alluaudites, these cations are extensively oxidized.

We have good evidence that wyllieite or wyllieite-related phases occur at other pegmatites as well, in fact at those pegmatites where arrojadite is known to occur. At the Nickel Plate pegmatite near Keystone, South Dakota, a deep green phase occurs with abundant arrojadite. This phase, physically practically identical to the Victory mine material, reveals a powder pattern very similar to wyllieite and quite distinct from alluaudite. A similar phase occurs sparingly at the Smith mine, Newport, New Hampshire, rimming and occurring interstitially with large granular masses of yellow-green arrojadite. Owing to the intricacies of the wyllieite crystal chemistry, it will be necessary to thoroughly investigate these phases chemically in order to properly define them.

NAME

Wyllieite was approved as a species by the International Commission on New Minerals and New Mineral Names by a vote of 13-1, the dissenter feeling that ordered and disordered forms should not be given different names. We

TABLE 3. WYLLIEITE: POWDER DATA^a

<i>hkl</i>	<i>d</i> (obs)	<i>d</i> (calc)	<i>hkl</i>
4	8.12	8.14	110
6	6.15	6.19	020
3	5.37	5.40	200
5	3.449	3.456	310
4	3.047	3.040	112
2	2.998	3.008	131
2	2.907	2.890	002
4	2.848	2.844	312
3	2.807	2.807	222
2	2.726	2.729	041
10	2.693	2.699	400
10	2.674	2.685	240
2	2.621	2.619	022
2	2.585	2.577	402
5	2.498	2.497	132
		2.489	112
1	2.472	2.474	420
3	2.385	2.379	422
2	2.302	2.305	151
2	2.201	2.194	512
3	2.163	2.157	151
3	2.123	2.127	510
4	2.079	2.070	313
3	2.036	2.040	350
2	1.968	1.962	532
1	1.926	1.928	602
		1.928	260
1	1.914	1.913	530
1	1.888	1.889	352
1	1.881	1.879	312
2	1.845	1.841	622
2	1.793	1.796	351
2	1.774	1.775	511
2	1.749	1.745	243
4	1.726	1.727	332
		1.726	262
2	1.703	1.703	171
1	1.659	1.659	641
		1.656	623
3	1.639	1.630	133
1	1.611	1.611	462
2	1.603	1.602	422
3	1.577	1.579	153
3	1.555	1.556	640
1	1.528	1.531	710
2	1.514	1.513	372
1	1.498	1.495	081
		1.495	553
1	1.484	1.481	802
1	1.465	1.464	371
2	1.453	1.452	172

20 more lines, each <2

^aFeK_α, 114.6 mm camera diameter.
Film corrected for shrinkage.

feel our six criteria for differences between wyllieites and alluaudites are sufficient evidence for warranting a new species. The name was approved by 12-2, the dissenters feeling that the name was too difficult to pronounce and too close to wilkeite. Wyllieite should be pronounced WY-LE-ITE.

It is named for Peter J. Wyllie, Professor of Petrology and Geochemistry, the University of Chicago, who has added much to our knowledge of granite melting in dry and aqueous conditions and who is a leader in the fields of petrogenesis, chemistry of ultramafic rocks, granite melts, and pedagogy in the earth sciences. In 1965, he won the Mineralogical Society of America award. With his interest in rock-forming minerals, it was only fitting that a mineral which occurs in rock-forming quantities be named after him.

The type specimen and the specimen displaying the large single crystal are preserved in the collections of the National Museum of Natural History. In addition, several kilograms of wyllieite have been donated to the museum for exchange purposes.

ACKNOWLEDGEMENTS

Messrs. G. R. Zechman and W. K. Sabine provided additional analytical results to our study. Dr. P. Toulmin and the late Dr. W. T. Pecora arranged for loan of an original sample of the "triphylite" from the Victory mine. Mr. W. L. Roberts and Dr. D. H. Garske of Rapid City, South Dakota assisted in collection of the samples.

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