

# THE AMERICAN MINERALOGIST

JOURNAL OF THE MINERALOGICAL SOCIETY OF AMERICA

Vol. 50

NOVEMBER-DECEMBER, 1965

Nos. 11 and 12

## GETCHELLITE, $\text{AsSbS}_3$ , A NEW MINERAL FROM HUMBOLDT COUNTY, NEVADA

B. G. WEISSBERG, *Chemistry Division, Department of Scientific and  
Industrial Research, Lower Hutt, New Zealand.*

### ABSTRACT

Getchellite is a new arsenic antimony sulfide mineral,  $\text{AsSbS}_3$ , from the Getchell Mine, Humboldt County, Nevada. It occurs in an epithermal arsenical gold deposit intimately associated with orpiment, realgar, stibnite, cinnabar and quartz.

Getchellite forms transparent, dark red, sectile crystals with a perfect {001} cleavage (yielding flexible, inelastic cleavage lamellae).  $H=1\frac{1}{2}$  to 2;  $G=3.92$  (obs.), 4.01 (calc., synthetic); it melts in the range  $340^\circ$  to  $355^\circ$  C.; its luster is pearly to vitreous on cleavage surfaces, otherwise resinous; its streak is orange-red. Getchellite is monoclinic, biaxial (+);  $2V_Z < 46^\circ$ ;  $\alpha > 2.11$  (white light),  $\beta > 2.72$  (Li light);  $Z=b$ ;  $Y \wedge a = 15^\circ \pm 5^\circ$ ;  $Y \wedge c = 101^\circ \pm 5^\circ$ ; dispersion crossed  $r > v$  strong. In reflected white light getchellite is grayish-white with a blue tint; it is anisotropic and shows strong blood-red internal reflections. Its reflectivity is in the range 25–30 (by visual comparison). Chemical analyses give 25.09 wt. % As, 42.04 wt. % Sb, and 32.82 wt. % S; the corresponding formula is  $\text{As}_{0.98}\text{Sb}_{1.01}\text{S}_3$ . The strongest lines in the x-ray diffraction powder pattern are 2.89 (100), 4.44 (80), 3.63 (70), 2.54 (60) and 2.33 (60) Å. Weissenberg equi-inclination photographs of synthetic single crystals give  $a=11.85$ ,  $b=8.99$ ,  $c=10.16$  Å,  $\beta=116^\circ 27'$ , space group  $P2_1/a$ , cell volume  $969$  Å<sup>3</sup>, cell content 8 [ $\text{As SbS}_3$ ].

The physical properties and x-ray data of synthetic getchellite, crystallized hydrothermally from  $\text{As}_3\text{SbS}_6$  glass in sodium sulfide solutions at  $260^\circ$  C. and 1,000 bars pressure, match those of the natural material.

Getchellite is named in honor of the Getchell Mine, at which it was discovered.

### OCCURRENCE AND ORIGIN

Getchellite ( $\text{AsSbS}_3$ ), is a new mineral from the Getchell Mine, which is about 20 miles north of Golconda, Humboldt County, Nevada. Ore specimens containing getchellite were collected by the author during a visit to the mine in August 1962 as part of an investigation of the paragenetic relations between the sulfides of iron, arsenic, antimony and mercury, and associated gangue minerals, but its presence was not recognized until the specimens were examined nearly a year later. The mineral is named in honor of the Getchell Mine, at which it was discovered. The name has been approved in advance of publication by the Commission on New Minerals and Mineral Names, IMA.

The geology of the Getchell Mine, previously described by Hardy (1941) and Joralemon (1951), consists essentially of a narrow steeply dipping fault zone cutting interbedded Paleozoic (?) shales, argillites, and limestones near a granodiorite intrusive. Mineralization is mainly confined to the sheared rocks in the fault zone and consists principally of quartz, calcite, realgar, orpiment, stibnite, pyrite, marcasite, cinnabar, barite, fluorite, gypsum and very fine-grained gold. Structures in the sheared rocks are complex, open spaces are common, and open-space filling is the dominant mode of mineral emplacement.

Getchellite occurs near the southern end of the ore body on the west side of the "South Pit Extension" where it is intimately associated with abundant orpiment and realgar and with lesser amounts of quartz, stibnite and cinnabar, all of which occur in the fault zone in veins up to eight inches thick cutting sheared and brecciated country rocks. In these veins getchellite was apparently one of the first hydrothermal minerals deposited after quartz. It completely fills some vugs lined with euhedral crystals of comb quartz, but more commonly it only partly fills such spaces, the remaining space being occupied by orpiment and realgar. Both orpiment and realgar commonly are molded around subhedral crystals of getchellite, but in some places anhedral grains of getchellite and a few rare euhedral crystals of getchellite were observed as inclusions in orpiment, suggesting that some getchellite and orpiment was deposited simultaneously or nearly so. Realgar is commonly molded around euhedral crystals of orpiment and also contains some euhedral crystals of orpiment. Minute doubly terminated crystals of quartz occur as inclusions in getchellite, orpiment and realgar, although most of the quartz in vugs and along vein walls underlies these minerals. Minute acicular crystals of stibnite are included in getchellite, orpiment and realgar, and thus stibnite was presumably deposited more or less continuously, but in small amounts, throughout the period of ore deposition. Cinnabar occurs in very minor amounts as very small, dark-red, pseudocubic rhombohedral crystals along the grain boundaries between realgar and orpiment and presumably was among the last of the minerals deposited. Small veinlets of realgar transect fragments of country rock, quartz, getchellite and orpiment, and indicate deposition or redistribution of realgar after some movement along the fault subsequent to the main sequence of ore deposition. Crystals of getchellite and orpiment are commonly bent or otherwise deformed by movements subsequent to ore deposition. From these textural relations it is concluded that quartz, getchellite, orpiment, realgar, stibnite and cinnabar are very closely associated both in time and in space and were probably transported by, and

deposited from the same or very similar solutions. The close association of the arsenic and antimony sulfides in many places throughout the world raises the question of why getchellite should be so rare a mineral. With its unusual physical properties, it is unlikely to have been overlooked.

#### PHYSICAL AND OPTICAL PROPERTIES

Getchellite, apart from its dark blood-red color and orange-red streak, closely resembles orpiment. In places, crystal faces and cleavage surfaces of getchellite display a purple to green iridescent tarnish. Its luster is pearly to vitreous on fresh cleavage surfaces, but elsewhere it is resinous. Getchellite has a perfect micaceous cleavage parallel to  $\{001\}$  yielding flexible but inelastic cleavage lamellae. Its fracture is splintery which suggests a second less perfect cleavage. Getchellite is sectile, and has a Mohs scale hardness of  $1\frac{1}{2}$  to 2. The specific gravity of getchellite, determined by means of a Berman torsion balance, is  $3.92 \pm 0.03^1$  and the calculated specific gravity is 4.01 (synthetic).

Getchellite turns darker red when heated. Above  $320^\circ$  C. it gradually turns black, sublimes, and minute black acicular crystals are formed on the cooler cover plate of the heating stage. In the range  $340$  to  $355^\circ$  C. getchellite apparently melts to an extremely viscous black mass which retains the gross shape of the original crystal fragments. Near  $470^\circ$  C. the melt loses viscosity very rapidly and begins to boil, and acicular metallic gray crystals, similar in appearance to stibnite, begin to form at  $490^\circ$  C.

In transmitted light getchellite is blood-red in color; it is biaxial positive with  $\alpha > 2.11$  (white light) and  $\beta > 2.72$  (lithium light); its birefringence is very strong to extreme with  $2H_z = 80^\circ \pm 3^\circ_z$  and  $2V^2 < 46^\circ$ . The orientation of getchellite is  $Z = b$ ,  $Y \wedge a = 15^\circ \pm 5^\circ$ , and  $Y \wedge c = 101^\circ \pm 5^\circ$ , and the dispersion is crossed,  $r > v$  strong. Pleochroism and differential absorption were not detected.

In vertically reflected white light the color of getchellite is grayish-white with a slight blue tint. Its reflectivity in white light is greater than that of the associated orpiment and realgar, but less than that of stibnite, and was visually estimated to be in the range 25–30. The weak anisotro-

<sup>1</sup> The actual measured value of the specific gravity, determined from 27 observations on three crystals, is  $3.88 \pm 0.02$  (probable error). This was corrected to  $3.92 \pm 0.03$  to account for 2.0 wt. % quartz as inclusions within the crystals indicated by the chemical analyses, and also includes an additional  $\pm 0.01$  error to allow for  $\pm 0.5$  wt. % variation in quartz content.

<sup>2</sup> The optic-axial angle in oil (1-bromonaphthalene,  $n = 1.661$ ),  $2H_z$ , was measured directly using a needle stage and a synthetic crystal, the optic plane of which was mounted normal to the needle axis.

pism tends to be obscured by strong blood-red internal reflections. Nearly all the crystals of getchellite examined with the reflecting microscope were bent, so that internal reflections arising from slip surfaces along cleavage lamellae were readily apparent. Getchellite polishes better than realgar and about the same as or slightly better than orpiment. Its polishing hardness appears to be slightly greater than that of orpiment, and it scratches and exfoliates readily as does orpiment.

#### CHEMICAL ANALYSIS

Samples of getchellite were prepared for analysis by crushing ore specimens in a jaw crusher, sieving, and then hand picking clean crystals and cleavage fragments of getchellite from the 7 to 18 mesh fraction, after washing the sieved fraction with distilled water and drying in air. The handpicked material was re-washed in distilled water, dried in air at 105° C. and stored in a desiccator until used.

Preliminary analysis of finely-ground getchellite by *x*-ray fluorescence indicated the presence of only arsenic, antimony, sulfur and a trace of selenium.

The arsenic and antimony in getchellite were determined in three samples taken into solution by a mixture of sulfuric and nitric acids. After removal of the nitric acid by evaporation and reduction of the solution with hydrazine sulfate, arsenic was separated from antimony as the sulfide by the method given by Hillebrand *et al.* (1953). After re-solution, the arsenic and antimony were determined separately by titration with potassium bromate using the method of Smith and May (1941).

Sulfur in getchellite was analysed separately in three samples. The samples were dissolved with bromine in carbon-tetrachloride (the method of Hillebrand *et al.*, 1953), and sulfur was determined gravimetrically as barium sulfate. The results of the analyses are given in Table 1.

The formula of getchellite calculated from the analyses is  $As_{0.98}Sb_{1.01}S_3$ . The excess of antimony in relation to arsenic indicated by the formula can be at least partly accounted for by an incomplete separation of arsenic from antimony in the analytical procedure and subsequent inclusion of some arsenic with antimony in the determination of antimony. Previous checks on the analytical procedure using known standard mixtures of  $As_2S_3$  and  $Sb_2S_3$  indicated that arsenic consistently ran from 0.4 to as much as 1.0 per cent low and that antimony ran from 0.1 to 0.5 per cent high. Inclusions of stibnite within the getchellite analysed probably also contribute to the high antimony content indicated by the analyses.

The trace elements in getchellite, determined by spectrographic analysis, are listed in Table 2.

TABLE 1. CHEMICAL ANALYSES OF GETCHELLITE

Analysis Number	Sample Weight	Insoluble Residue		Wt. Arsenic	Wt. Antimony	Wt. Sulfur	Wt. % As	Wt. % Sb	Wt. % S	
		Weight	Weight %							
1	0.2240	0.0051	2.3	0.0548	0.0917	—	25.03	41.89	—	
2	0.2151	0.0070	3.2	0.0527	0.0869	—	25.32	41.76	—	
3	0.1933	0.0035	1.8	0.0473	0.0806	—	24.92	42.46	—	
4	0.1576	0.0023	1.5	—	—	0.0508 <sub>4</sub>	—	—	32.74	
5	0.1448	0.0031	2.1	—	—	0.0465 <sub>8</sub>	—	—	32.87	
6	0.1678	0.0030	1.8	—	—	0.0541 <sub>4</sub>	—	—	32.85	
Average							25.09	42.04	32.82	Total 99.95
Theoretical composition of As <sub>3</sub> SbS <sub>6</sub>							25.59	41.57	32.84	100.00

NOTE: The insoluble residue was composed of minute doubly-terminated crystals of quartz, the weight of which was subtracted from the initial sample weight in calculating the weight percentage of As, Sb and S in getchellite. Analyst, B. G. Weissberg.

## SYNTHESIS

Early in the investigation the composition of getchellite was incorrectly estimated as As<sub>3</sub>SbS<sub>6</sub> from the peak height ratios of arsenic to antimony determined by *x*-ray fluorescence, and glass of this composition was prepared by air quenching a melt of As<sub>2</sub>S<sub>3</sub> and Sb<sub>2</sub>S<sub>3</sub> mixed in a 3:1 mole ratio. Portions of this glass were sealed in gold capsules containing 2 weight per cent sodium sulfide solution and held at 260° C. and 1,000 bars pressure for 5 days, 14 days and 50 days. The product in each of the runs was composed of both orpiment and getchellite in roughly equal

TABLE 2. EMISSION SPECTROGRAPHIC ANALYSIS OF GETCHELLITE

Constituent	Percentage
As, Sb, and S	Major
Si	0.2
Mg	<0.003
Al	<0.01
Ca	0.02
Cu	0.01
Ag	0.002
Pb	0.01
Tl	0.0005
Hg	~0.0001

No other elements were detected.

Analysts: H. J. Todd and W. C. Tennant, Chemistry Division, D.S.I.R., Lower Hutt, New Zealand.

quantities. In the run of only 5 days duration about 50% of the initial glass remained unreacted, but in the runs of longer duration no trace of the initial glass remained. Crystals of the synthetic getchellite thus produced were used in the crystallographic investigations that follow.

#### CRYSTALLOGRAPHY

Getchellite was found variously as small equant subhedral crystals less than 0.5 millimeters in diameter up to anhedral grains as large as 4 millimeters in maximum dimension. All the getchellite found was badly deformed, and the author located only one crystal fragment which yielded results with the two-circle reflecting goniometer and the single crystal  $x$ -ray camera. This crystal fragment was sub-equant, approximately 0.15 millimeters in diameter, and although the crystal was bent through about ten degrees of arc in the  $ac$  plane about  $b$  as an axis, it was possible to determine the Miller indices of the better developed crystal faces and to match the single crystal  $x$ -ray photographs with those of synthetic getchellite. The crystal was bounded by  $\{001\}$  cleavage surfaces and the faces  $(0\bar{1}0)$ ,  $(201)$ ,  $(1\bar{1}\bar{1})$ ,  $(2\bar{1}\bar{1})$ , all equally well developed, and by  $(2\bar{1}\bar{2})$ ,  $(2\bar{1}\bar{2})$ ,  $(2\bar{1}\bar{1})$  and  $(4\bar{1}\bar{2})$  smaller and less well developed. It was not possible to index several other smaller faces on the crystal because of the large uncertainty in their positions. A rotation photograph about the crystallographic  $a$ -axis and a normal beam Weissenberg zero layer-line photograph about the  $a$ -axis of this crystal matched perfectly with similar photographs of synthetic getchellite in spite of the streaky nature of the  $x$ -ray reflections of the natural crystal.

Single crystals of synthetic getchellite were up to 1.0 millimeter long and were prismatic parallel  $[100]$  and slightly tabular with the form  $\{001\}$  predominating over  $\{011\}$  and with  $\{010\}$  very small and commonly absent. Terminating forms were  $\{2\bar{1}\bar{1}\}$ ,  $\{1\bar{1}\bar{1}\}$ , and more rarely  $\{2\bar{0}\bar{1}\}$ ,  $\{10\bar{1}\}$ . Simple and polysynthetic twins were commonly observed in the synthetic getchellite with the twin plane and composition plane parallel to  $\{001\}$ .

The  $x$ -ray powder diffraction patterns of natural and synthetic getchellite are given in Table 3. The  $x$ -ray powder diffraction pattern of the synthetic getchellite produced in the synthesis of  $4\frac{1}{2}$  days duration was somewhat more diffuse than that of natural getchellite, but no significant visible differences, other than degree of exposure, were observed between the powder patterns of natural getchellite and synthetic getchellite produced in the syntheses of 14- and 50-days duration. The powder pattern was indexed by graphically comparing the measured spacings of natural getchellite with the interplanar spacings obtained graphically from the cell dimensions of synthetic getchellite. The interplanar spacings listed

TABLE 3. X-RAY POWDER DIFFRACTION PATTERNS OF GETCHELLITE<sup>1</sup>  
AND SYNTHETIC GETCHELLITE<sup>2</sup>

Getchellite		Synthetic Getchellite		hkl	d (calc.) Å
d (meas.) Å	I/I <sub>0</sub>	d (meas.) Å	I/I <sub>0</sub>		
9.19	40	9.17	40	001	9.11
6.46	50	6.46	40	11 $\bar{1}$	6.44
				011	6.40
5.91	5	5.94	5	20 $\bar{1}$	5.86
5.40	5	5.30	5	200	5.30
4.97	50	4.92	50	21 $\bar{1}$	4.91
				111	4.85
4.60	50 broad	4.58	50	20 $\bar{2}$	4.62
				210	4.57
				002	4.55
4.44	80	4.44	80	020	4.50
				11 $\bar{2}$	4.43
4.15	5			21 $\bar{2}$	4.11
		4.10			
		(not resolved)	40 broad	120	4.10
4.06	40			012	4.06
				12 $\bar{1}$	4.04
				021	4.01
3.63	70 broad	3.61	70 broad	31 $\bar{1}$	3.61
3.38	5	3.38	5	220	3.43
				31 $\bar{2}$	3.42
				112	3.37
				12 $\bar{2}$	3.37
				20 $\bar{3}$	3.36
3.33	20	3.30	10	310	3.31
3.24	10	3.23	10	22 $\bar{2}$	3.22
				022	3.20
3.14	40	3.15	40	21 $\bar{3}$	3.14
				11 $\bar{3}$	3.12
3.08	40	3.10	40	003	3.04
2.99	10	2.98	10	32 $\bar{1}$	2.96
2.89	100 broad	2.88	100 broad	40 $\bar{2}$	2.93
				40 $\bar{1}$	2.92
				31 $\bar{3}$	2.91
				202	2.88
				130	2.88
				013	2.88
				32 $\bar{2}$	2.86
2.80	50	2.79	40	122	2.83
				320	2.78
				41 $\bar{2}$	2.78
2.69	40	2.68	30	22 $\bar{3}$	2.69
				12 $\bar{3}$	2.68
				23 $\bar{1}$	2.67
				40 $\bar{3}$	2.67
2.60	10	2.59	20	230	2.61
				13 $\bar{2}$	2.58
2.54	60	2.53	70	41 $\bar{3}$	2.56
				20 $\bar{4}$	2.54

<sup>1</sup> Getchellite from the Getchell Mine, Humboldt County, Nevada.

<sup>2</sup> Synthetic getchellite hydrothermally crystallized from As<sub>2</sub>SbS<sub>3</sub> glass in an aqueous sodium sulfide solution held at 260° C. and 1,000 bars pressure for 14 days.

Data from photographs taken with a 114.6 mm diameter Debye-Scherrer camera, using Cu radiation with Ni filter, powder suspended in a rod of "Duco" cement, and no correction for film shrinkage.

TABLE 3—(continued)

Getchellite		Synthetic Getchellite		hkl	d (calc.) Å
d (meas.) Å	I/I <sub>0</sub>	d (meas.) Å	I/I <sub>0</sub>		
				410	2.54
				32 $\bar{3}$	2.54
				113	2.52
				023	2.52
				23 $\bar{2}$	2.51
2.46	5	2.46	5	214	2.45
				42 $\bar{2}$	2.45
2.41	10	2.40	10	321	2.43
				222	2.42
				31 $\bar{4}$	2.39
				114	2.38
2.33	60 broad	2.32	50 broad	33 $\bar{2}$	2.33
				400	2.31
				132	2.31
				42 $\bar{3}$	2.30
2.25	50	2.24	60	312	2.26
				040	2.25
				203	2.24
				41 $\bar{4}$	2.24
				23 $\bar{3}$	2.24
				13 $\bar{3}$	2.23
2.20	5	2.18	5	22 $\bar{4}$	2.22
				411	2.22
				51 $\bar{3}$	2.21
				014	2.21
				140	2.20
				123	2.18
				32 $\bar{4}$	2.17
2.10	40	2.09	20	52 $\bar{2}$	2.10
				24 $\bar{1}$	2.10
				43 $\bar{2}$	2.09
				43 $\bar{1}$	2.09
2.03	30 broad	2.04	20	510	2.06
				42 $\bar{4}$	2.05
				52 $\bar{1}$	2.05
				421	2.04
				024	2.03
				52 $\bar{3}$	2.03
				514	2.03
				24 $\bar{2}$	2.02
				205	2.02
1.977	5	1.979	5	420	1.986
				21 $\bar{5}$	1.978
				133	1.975
				21 $\bar{5}$	1.972
				60 $\bar{2}$	1.971
1.919	40 broad	1.910	30 broad	61 $\bar{2}$	1.925
				34 $\bar{2}$	1.922
				520	1.919
				41 $\bar{5}$	1.919
				142	1.913
				33 $\bar{4}$	1.911
				61 $\bar{3}$	1.908
				13 $\bar{4}$	1.902
				412	1.902
				60 $\bar{1}$	1.901
				11 $\bar{5}$	1.901



TABLE 3—(continued)

Getchellite		Synthetic Getchellite		hkl	d (calc.) Å
d (meas.) Å	I/I <sub>0</sub>	d (meas.) Å	I/I <sub>0</sub>		
1.865	10	1.864	5		
1.824	10	1.822	10		
1.793	5	1.789	5		
1.758	10	1.741	10		
		not resolved			
1.733	10				
1.699	20	1.697	10		
		1.674	5		
1.658	30 broad	1.653	20		
1.595	5				
1.567	30	1.573	10		
1.536	10	1.526	5		
1.471	10	1.451	5		
1.426	10	1.432	10		
1.382	5				
1.288	10				

were calculated from the lattice dimensions of synthetic getchellite for those planes the spacings of which, determined graphically, agreed with the measured values of natural getchellite.

Inasmuch as all the getchellite crystals found were badly deformed, synthetic crystals of getchellite were used in the single crystal *x*-ray investigations. Weissenberg (equi-inclination) photographs of crystals of synthetic getchellite rotated about the *a*-crystallographic axis and *b*-crystallographic axis were taken of the zero, first, and second layers. Cell dimensions of synthetic getchellite determined from the photographs are:  $a = 11.85 \pm 0.02$ ,  $b = 8.99 \pm 0.02$ ,  $c = 10.16 \pm 0.02$  Å,  $\beta = 116^\circ 27' \pm 12'$ . The unit cell has a volume of 969 Å<sup>3</sup> and contains eight AsSbS<sub>3</sub>. The calculated specific gravity is 4.01 in comparison with 3.92 for the natural material. The space group is  $P2_1/a$ , reflections (h0l) and (0k0) being absent when h and k are odd.

#### ACKNOWLEDGMENTS

The author wishes to acknowledge the financial support of the National Science Foundation, Grant N. P862, to Prof. F. W. Dickson for ore genesis research, in sponsoring the initial field investigation in which getchellite was found and also for providing, through the grant, the pressure vessels and accessory equipment necessary for the hydrothermal synthesis of the getchellite crystals used in this study. The General Manager and staff at the Getchell Mine were most courteous and helpful during the author's visits to the mine. Special thanks are extended to the staff of the Department of Geology at the University of California, Riverside, for providing space, facilities and support during most of the

investigation while the author was there as a research fellow. Polished sections were expertly made by Mr. John deGrossé of the Department of Geology, University of California, Los Angeles. Appreciation is expressed to the New Zealand Geological Survey for making available to the author reflecting and heating stage microscopes. Thanks are given to Dr. G. A. Challis and Prof. George Tunell for critically reviewing and improving the manuscript.

## REFERENCES

- HARDY, R. A. (1941) Geology of the Getchell Mine. *Am. Inst. Min. Met. Eng., Trans.*, **144**, 147-150.
- HILLEBRAND, W. F., C. E. F. LUNDELL, M. S. BRIGHT AND J. E. HOFFMANN (1953) *Applied Inorganic Analysis*. 2nd Ed., John Wiley & Sons, Inc., New York.
- JORALEMON, PETER (1951) The occurrence of gold at the Getchell Mine, Nevada. *Econ. Geol.* **46**, 267-310.
- SMITH, G. F. AND R. L. MAY (1941) Use of bromate in volumetric analysis—Determination of arsenic and antimony using internal indicators at ordinary temperatures. *Industrial Engineering Chemical Analysis, Analytical Ed.*, **13**, 460-461.

*Manuscript received, March 11, 1965; accepted for publication, March 29, 1965.*