ART. XLVI.--On Spangolite, a new Copper Mineral;
S L PENFIELD
American Journal of Science (1880-1910); May 1890; 39, 233; American Periodicals Series Online
pg. 370

ART. XLVI.—*On Spangolite, a new Copper Mineral:* by S. L. PENFIELD.

DURING the summer of 1889, while visiting Mr. Norman Spang of Etna, Allegheny County, Pa., my attention was called by him to a very beautifully crystallized specimen of an unknown mineral which he had obtained from a man living near Tombstone, Arizona. The original owner had a small

---

* Loc. cit.

† Notes on the Geology of Mynydd Mawr and the Nantle Valley, Geol. Mag., v, 1888, p. 221 and p. 455.

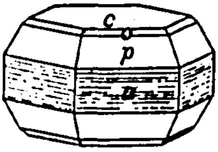
collection of minerals which he had gathered together within a radius of about two hundred miles, but he had no idea of just where he had found the specimen, though he thought it was from the Globe District. Mr. Spang had forgotten the name of the man from whom he had secured it, so that until other specimens are found uncertainty must exist about the exact locality and mode of occurrence. On expressing a desire to investigate the mineral, Mr. Spang very generously lent me the specimen and has since presented me with it, and it is now deposited in the collection of Professor Geo. J. Brush, at New Haven.

A preliminary blow-pipe examination showed that the mineral was undoubtedly a new species and essentially a hydrated sulphate and chloride of copper, and I take pleasure in not only expressing at this time my thanks to Mr. Spang for his kindness but *also in naming* the *mineral*, which as will be shown, is of unusual interest, *Spangolite* after him.

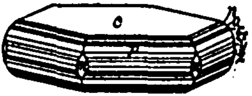
The original specimen, which was about the size of a small hen's egg, consisted of a rounded mass of impure cuprite which was mostly covered with hexagonal crystals of spangolite, associated with a few crystals of azurite and some slender prismatic crystals of a copper mineral containing chlorine, probably atacamite.

The crystallization of spangolite is hexagonal, rhombohedral. The habit of the crystals does not vary much as they all show a prominent hexagonal basal plane and a series of apparently holohedral hexagonal pyramids, which as will be shown must be taken as pyramids of the second order. Some of the crystals have the habit of fig. 1 showing a prism, which is always so dull and striated that it gives no reflection of light, associated with pyramids and a basal plane. Others are flatter, figure 2, and show a large series of pyramids which oscillate with one another, giving rise to prominent striations which run horizontally about the crystal and make the identification of the pyramids a difficult matter. On the crystals we occasionally find a prism of the first order, *m*, which is small but gives good reflections. The material which could be used for the investigation was limited, but great care was taken to select only the best and purest crystals for making the crys-

1.



2.



tallographic and chemical investigation. A number of small crystals were selected which were measured in the prominent pyramidal zones between the basal planes. The basal planes usually gave very good reflections of the signal but on turning the crystal on the goniometer, after the pyramids came into a position to reflect light, there usually followed an unbroken band of signals reflected from these faces owing to their striations and oscillatory combinations. In this band of reflections prominent parts could usually be located which indicated the position of distinct pyramidal faces. On measuring fifteen independent pyramidal zones on five different crystals, only one crystal was found, which we will designate as No. 1, that gave sharp reflections from the pyramidal faces; from this an angle was obtained of $c \wedge p$, $0001 \wedge 2\bar{1}\bar{1}2 = 63° 32'$. Still better results were obtained on measuring from pyramid to pyramid when the reflections of the signal are not so much disturbed by the striations and the value given below, which will be accepted as the fundamental measurement, was obtained

$$p \wedge p', \; 2\bar{1}\bar{1}2 \wedge 11\bar{2}2 = 53° \; 11' \; 30''$$

from which we calculate the length of the vertical axis $c = 2{\cdot}0108$.

The largest of all the crystals, which we will designate as No. 2, was fortunately so situated that it could be measured without detaching it from the specimen. It was very symmetrical and had the habit shown in fig. 1; it measured about 8^{mm} in diameter and was $5\frac{1}{2}^{mm}$ high. Five out of the six pyramidal faces p at one end of the crystal were quite perfect, being only slightly distorted by the horizontal striations. Some of the faces were so situated that reflections could not be obtained from them without detaching the crystal, but a number of very satisfactory measurements were made which are given below. The accompanying table contains a summary of the measurements which were made on the two best crystals from the faces c, 0001, O; p, $2\bar{1}\bar{1}2$, 1–2; o, $2\bar{1}\bar{1}4$, ½–2 and m, $10\bar{1}0$, I; all of which gave sharp reflections of the signal. The measurements were made on a Fuess goniometer, using the signal and the ocular β of Websky. The one marked with an asterisk was taken as fundamental.

TABLE I.

	Crystal No. 1.	Crystal No. 2.	Calculated.
$p \wedge p'$, $2\bar{1}\bar{1}2 \wedge 11\bar{2}2$	53° 11′ 30″*	53° 13′ 45″	53° 11′ 30″
$p' \wedge p''$, $11\bar{2}2 \wedge \bar{1}2\bar{1}2$		53° 11′ 45″	"
$p'' \wedge p'''$, $\bar{1}2\bar{1}2 \wedge \bar{2}112$		53° 14′	"
$p \wedge p^{iv}$, $2\bar{1}\bar{1}2 \wedge \bar{1}\bar{1}22$		101° 43′	101° 41′
$p \wedge o^{v}$, $2\bar{1}\bar{1}2 \wedge 1\bar{2}14$	50° 48′	50° 51′	50° 51′
$p \wedge c$, $2\bar{1}\bar{1}2 \wedge 0001$	63° 32′		63° 33′ 30″
$p \wedge m$, $2\bar{1}\bar{1}2 \wedge 10\bar{1}0$	39° 4′		39° 9′

In the above table we find a very satisfactory agreement between the measured and calculated angles from which we may conclude that the measurement which we have accepted as fundamental is very nearly correct.

When all of the approximate measurements, which were obtained from fifteen different zones measuring from the basal plane above over the pyramids on the base below, are tabulated it is found that with few exceptions they fall into groups indicating the existence of a series of pyramids, among which we may safely assume the following, in addition to those which have already been mentioned: *k*, $2\bar{1}\bar{1}8$, $\frac{1}{4}$–2; *n*, $2\bar{1}\bar{1}6$, $\frac{1}{3}$–2; *r*, $6\bar{3}\bar{3}8$, $\frac{3}{4}$–2; *l*, $6\bar{3}\bar{3}7$, $\frac{6}{7}$–2; *x*, $6\bar{3}\bar{3}4$, $\frac{3}{2}$–2; *y*, $2\bar{1}\bar{1}1$, 2–2; *z*, $6\bar{3}\bar{3}2$, 3–2.

The following table contains a summary of the measurements which can be referred to the definite pyramids just mentioned.

TABLE II.

	No. of times observed.	Limiting measurements.	Average.	Calculated.
$c \wedge k$, $0001 \wedge 2\bar{1}\bar{1}8$	3	25° 48′–26° 7′	25° 55′	26° 41′
$c \wedge n$, $0001 \wedge 2\bar{1}\bar{1}6$	6	33 54 –34 46	34 26	33 50
$c \wedge o$, $0001 \wedge 2\bar{1}\bar{1}4$	19	44 19 –46	45 3	45 9
$c \wedge r$, $0001 \wedge 6\bar{3}\bar{3}8$	3	55 46 –56 53	56 18	56 27
$c \wedge l$, $0001 \wedge 6\bar{3}\bar{3}7$	5	59 10 –60 33	59 50	59 53
$c \wedge p$, $0001 \wedge 2\bar{1}\bar{1}2$	22	63 12 –63 46	63 30	63 33½
$c \wedge x$, $0001 \wedge 6\bar{3}\bar{3}4$	6	71 17 –72 30	71 51	71 39
$c \wedge y$, $0001 \wedge 2\bar{1}\bar{1}1$	4	76 48 –75 36	76 13	76 2
$c \wedge z$, $0001 \wedge 6\bar{3}\bar{3}2$	7	79 17 –80 39	80 4	80 35

An examination of the above table will give an idea of the frequency of occurrence of the different pyramids and the accuracy of the determination.

All of the measurements which have not already been given in Tables I and II are collected together in the following table where they are again arranged into groups, which indicate the occurrence of vicinal faces agreeing nearly with simple forms.

TABLE III.

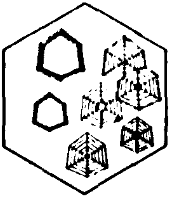
Mean angle on the base.	Limiting measurement.	No. of these observed.	Simple form to which they approximate.
23°		1	$2\bar{1}\bar{1}10$, $\frac{1}{5}$–2
35 44′	35° 30′–36°	6	*n*, $2\bar{1}\bar{1}6$, $\frac{1}{3}$–2
43 40	43 15 –44	5	*o*, $2\bar{1}\bar{1}4$, $\frac{1}{2}$–2
57 40	57 26 –57 53′	2	*r*, $6\bar{3}\bar{3}8$, $\frac{3}{4}$–2
61 37	60 47 –62 41	4	*p*, $2\bar{1}\bar{1}2$, 1–2
73 2	73 –73 5	2	*x*, $6\bar{3}\bar{3}4$, $\frac{3}{2}$–2
77 53	77 5 –78 53	4	*y*, $2\bar{1}\bar{1}1$, 2–2
88 15		1	*a*, $2\bar{1}\bar{1}0$, *i*–2

In all of the zones which were measured there was no indication of any hemihedral development of the pyramids.

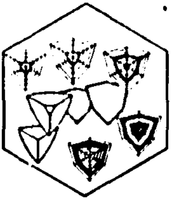
Cleavage.—The cleavage of spangolite is very perfect parallel to the base; this was a great help in studying the crystals, as many of the measurements recorded in Tables II and III, were obtained from cleavage-planes. Inclined to the base the crystals usually broke with a conchoidal fracture; in only one case a distinct cleavage was observed parallel to the pyramid *p*, the angle of the cleavage measured on to the base being 63° 28′, calculated 63° 33½′. Thin plates of the mineral are non-elastic and brittle.

Etching.—Experiments made by etching the mineral with acids gave results which add very much to a proper understanding of the crystals. It is readily soluble in dilute mineral acids and the perfect basal cleavage makes it easy to obtain orientated sections suitable for etching. The figures differed both with the character and strength of the acid, but always showed a decided hemihedral and rhombohedral symmetry. Fig. 3 represents the character of some etchings produced by dilute sulphuric acid. The figures which are very perfect are about ·066^{mm} in diameter and have the shape of a section across a scalenohedron. Some of the depressions are bounded below by a basal plane, others taper to a point while the scalenohedrons oscillate and give rise to delicate striations which are beautifully brought out under the microscope by a slight change of focus. The obtuse angle of the scalenohedron section measured under the microscope was about 133°, from which we calculate that its relation to the lateral axis is $a:\frac{8}{5}a:\frac{8}{3}a$, which requires an angle of 133° 10′. Fig. 4 represents some etchings produced by very dilute sulphuric acid: 1^{cc} of concentrated H_2SO_4 diluted with 80^{cc} of water. These also have a scalenohedral cross section and are about ·06^{mm} in diameter.

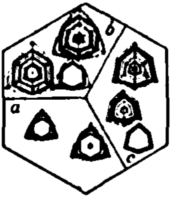
3.



4.



5.



The obtuse angle of the cross sections measures about 152°, from which its relation on the lateral axes $a:\frac{4}{3}a:4a$ can be calculated, which requires an angle of 152° 12′. There are also some steep rhombohedral depressions, with somewhat curved contours developed on this section. Some of the etchings produced by hydrochloric acid are shown in fig. 5, where the hexagon is divided into three parts. The figures shown in part *a* represent deep hexagonal depressions whose cross section is that of a pyramid of the second order, these have a diameter of about ·035^{mm} and are surrounded above with shallow and very delicate rhombohedral depressions with curved contours. In part *b* we have again scalenohedral, surrounded by shallow and more delicate rhombohedral, depressions. The obtuse angle of the scalenohedral sections measured 129°, from which its relation on the lateral axes $a : \frac{7}{4}a : \frac{7}{3}a$ can be calculated, which requires an angle of 129° 26′. In part *c* we have again represented simple scalenohedron depressions which are about $\cdot 025^{mm}$ in diameter and were produced by a very dilute acid, 1^{cc} concentrated HCl diluted with 160^{cc} of water. The obtuse angle of the scalenohedron measured about 142°, indicating a relation on the lateral axes of $a : \frac{8}{7}a : 8a$, which requires an angle of 141° 48′. With nitric acid the figures are very similar to those produced by hydrochloric; in all cases it was observed that with very dilute acids there was a tendency to form scalenohedral and with stronger rhombohedral depressions. When we compare the position of these rhombohedral and scalenohedral etchings to the outer hexagon, which in figs. 3, 4 and 5 indicates the outline of the crystal section, we see at once that the pyramids on the mineral must be of the second order. It should be stated here that the etchings were of very great beauty and perfection, the outline of the scalenohedral cross sections being in almost all cases very distinct and free from distortions of any kind, so that the angles could be measured with comparative accuracy.

Optical properties.—The color of the mineral by reflected light is dark green, cleavage plates by transmitted light are light green. Prof. H. L. Wells examined a basal section of the mineral $\cdot 4^{mm}$ thick with the spectroscope. When the slit was very narrow the light transmitted by the mineral gave a narrow spectrum with a maximum of light in the green at about $\lambda 525$. There was a total absorption of the red and yellow, running well into the yellowish-green. At the other end of the spectrum there was a decided absorption of the blue and a total absorption of the violet. Pleochroism is not very marked. The ordinary ray is green while the extraordinary is a decided bluish green. Cleavage plates show perfectly normal optical properties In convergent polarized light they yield a black cross surrounded by rings which are bordered by green and blue. The double refraction is quite strong and negative. Considerable difficulty was obtained in making a prism from a small crystal of the mineral with its edge at right angles to the perfect basal cleavage, but a small one was obtained, with an angle of 37° 48′, from which the indices of refraction were determined. The prism was opaque to the red and yellow lights of lithia and soda flames, even to the yellowish-green light of a thallium flame. With an ordinary kerosene flame the prism yielded two narrow green spectra with a minimum deviation of 26° 25′ for the extraordinary and 28° 46′ for the ordinary, measuring from the brightest part of the spectra, which from the spectroscopic examination we have located at about λ 525, and we obtain for the two indices of refraction ω 1·694, ϵ 1·641.

Other physical properties.—The hardness of the mineral on the basal plane is about 2, on the pyramidal faces nearly 3. The specific gravity was taken very carefully with a chemical balance on the purest material, which was selected for chemical examination. After boiling the crystals in water, to expel any air, three separate portions weighing respectively 0·2143, 0·1787 and 0·1538 grams gave 3·147, 3·133 and 3·142, an average of 3·141 as the specific gravity.

Chemical composition.—More than three grams of exceptionally pure material were readily obtained by sacrificing about one-half of the crystals on the specimen. As the material was somewhat limited a method of analysis was adopted by which nothing could well escape detection and a qualitative and quantitative analysis was carried on with a single sample, the results of which are given below. The fourth analysis was made on an entirely different sample from that which yielded the figures in the first three columns.

	I.	II.	III.	IV.	Average.	Ratio.		Calculated for Cu_6Al $ClSO_{10}9H_2O$
SO_3	....	10·08	10·11	10·14	10·11	·126	1·01	10·03
Cl	4·13	....	4·10	4·11	4·11	·116	0·93	4·45
Al_2O_3....	6·59	....	6·51	6·70	6·60	·064	0·51	6·45
CuO	59·57	....	59·47	59·50	59·51	·7495	6·00	59·75
H_2O	....	20·32	....	20·49	20·41	1·134	9·07	20·32
					100·74			101·00
		O, equivalent of Cl....			·92			1·00
					99·82			100·00

The analysis yields a ratio of $SO_3 : Cl : Al_2O_3 : CuO : H_2O$ = 1·01 : 0·93 : 0·51 : 6·0 : 9·07 or very nearly 1 : 1 : ·5 : 6 : 9, from which we obtain a rather complicated and remarkable formula, $Cu_6AlClSO_{10}$, $9H_2O$. No doubt can however exist concerning this formula; not only was the material beautifully crystallized and of unusual purity, but the analysis of two separate samples are identical within the error of analysis and the ratio is throughout very sharp; moreover the calculated composition agrees very well with the results of analysis. A slight deficiency in chlorine may result from a partial replacement of that element by hydroxyl, which, if true, would diminish somewhat the slight excess of water. The method of analysis was as follows: A weighed quantity of the mineral lost water slowly by standing in a desiccator over sulphuric acid, amounting to 0·30 per cent in thirty-six hours, but it regained almost all of this loss by standing uncovered in the air. Heated for an hour at 100° C. it lost about 0·49 per cent, but also regained this by standing in the air. The analyses were all made on air-dry powder. Water was determined by heating the mineral, weighed in a platinum boat, in a hard glass tube containing a loose plug of sodium carbonate at one end, through which the water vapors were conducted before absorption in a weighed chloride of calcium tube. A good deal of chloride of copper distilled off from the mineral. In some cases the contents of the boat was dissolved in nitric acid, the tube cleaned out carefully and a complete analysis made on one portion. A new portion being taken it was dissolved in nitric acid and the chlorine precipitated with silver nitrate. The weighed silver chloride when tested was found to contain no bromine or iodine and when ignited in hydrogen gas yielded a weight of metallic silver agreeing with the composition AgCl. After removing the excess of the silver from the solution with hydrochloric acid the SO_3 was precipitated with $BaCl_2$, care being taken to avoid a loss owing to the solubility of $BaSO_4$ in the aqua regia which was present in the analysis. After separating the excess of barium with sulphuric acid the solution was evaporated to expel the nitric acid after which the copper was precipitated with hydrogen sulphide and weighed after ignition in hydrogen gas as Cu_2S. A portion of the copper precipitate was carefully tested for other metals, but none were found. The filtrate from the copper sulphide, when evaporated to dryness and ignited left a residue which proved to be sulphate of alumina; this was dissolved in acid, precipitated in ammonia and weighed as Al_2O_3. A weighed quantity of the oxide was carefully tested for beryllium, but none was found, and after conversion into sulphate and evaporation with the right quantity of K_2SO_4 it yielded alum crystals. The filtrate from the alumina yielded no perceptible residue when evaporated to dryness proving that everything had been separated from the solution.

Pyrognostic and chemical tests.—Before the blowpipe the mineral fuses at about 3 to a black slaggy mass, coloring the flame green. On charcoal with soda in reducing flame it yields globules of metallic copper. Heated in the closed tube gives abundant water which has a strong acid reaction. Insoluble in water, but readily soluble in dilute acids.

There is at present no known mineral similar to spangolite in composition; the very rare connellite from Cornwall, England, whose chemical composition has never been determined, is the nearest approach to it, as it contains, according to Connel, copper in combination with sulphuric and hydrochloric acids, but the crystalline form and physical properties of the two minerals are entirely distinct. As far as can be found these two minerals and sulphohalite, $3Na_2SO_4$, $2NaCl$: Caracolite Na_2SO_4, $Pb[OH]Cl$ and Kainite $MgSO_4$, KCl, $3H_2O$ are the only mineral occurrences of sulphate and chloride in combination. The occurrence of small quantities of alumina in a copper mineral, although not unknown, is certainly very unusual. *It is also* interesting to note that the alumina in the formula, one atom, is just sufficient to satisfy the quantivalence of *the total acids* $[AlCl]SO_4$, leaving six molecules of cupric oxide basic.

Before closing the author desires to express the hope that some one living in the neighborhood of Tombstone, Arizona, will take an interest in examining both the collections and ores of that region so as to secure, if possible, an abundant supply of this mineral.

Mineralogical Laboratory of the Sheffield Scientific School,
New Haven, March, 1890.