CONTRIBUTIONS TO CRYSTALLOGRAPHY: CLAUDETITE; MINASRAGRITE; SAMSONITE; NATIVE SELENIUM; INDIUM

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CLAUDETITE

Our knowledge of the crystal form of the oxide of arsenic, claudetite, rests chiefly on the study by Schmidt¹ of crystals formed as the result of a mine fire at Szomolnok, Hungary. The crystals measured by Schmidt were mostly paper thin and striated so that the values recorded are not altogether reliable.

Crystals of claudetite from two sources have recently come into the hands of the author and proved to give such constant and consonant measurements that new elements have been based upon them. I am indebted to Mr. Wilke of Palo Alto for this material.

(a) CLAUDETITE FROM IMPERIAL Co., CALIFORNIA:—The hand specimen is a whitish rock which appears to be an altered schist, largely sericitized. It contains lenses of massive realgar and thin veins, parallel to the schistose structure, of claudetite. Where they are open, the veins are lined with radiating masses of brilliant claudetite crystals, the largest a millimeter or more in diameter and up to 4 mm. long. With the claudetite are crystals of sulphur.

Two crystals were detached from the wall of a cavity without distorting them, the extraordinary softness and ease of cleavage of the mineral making this a matter of great difficulty.

Two excellent crystals were measured, and the angles obtained from these crystals are shown in the following table.

(b) CLAUDETITE FROM JEROME, ARIZONA:—A small cavity 3 cm. across lined with beautiful silky crystals of claudetite comes from the United Verde Mine and is undoubtedly a product of the mine fire.² The crystals are plates or laths, terminated only at the free ends and measuring at maximum 3 by 10 mm. with thickness less than .5 mm. Both prism and terminal faces give excellent measurement. Four crystals were measured, one of which was a twin on (100). Table I presents the angles obtained from the claudetite crystals of both localities.

¹ Zeit. Krist., vol. **14,** p. 575, 1888.

² See Lausen, C., Hydrous Sulphates formed under Fumarolic Conditions at the United Verde Mine: Am. Mineral., vol. 13, p. 203, 1928.

TABLE I

A-Crystals from Imperial Co., California

Form	No. of faces	φ	Range ϕ	ρ	Range ρ
110	5	67°38′	67°29′-67°53′	90°00′	
T01	2	90 00	89 49 -90 01	37 51	37°47′-37°55′
I11	1	-6601		40 33	-
111	4	69 24	69 24 69 25	44 49	44 44 -44 53
B—Cryst	als from	Jerome, Ariz	ona		
130	10	$38\ 45\frac{1}{2}$	37 12 -39 40	90 00	
011	2	12 00	-	20 04	1
101	2	89 43	89 35 -89 50	43 00	_
₹01	4	-9014	89 04 -90 30	37 53	37 38 -38 06
T 11	10	-6552	64 43 -67 02	40 29	40 17 -40 45
111	6	69 23	69 09 -69 32	44 48	44 40 -45 03
T21	2	-4824	48 00 -48 49	46 15	46 13 -46 18

The projection elements were calculated separately for crystals of each locality with the following results:

	Imperial Co.	Jerome	Mean
p_0'	. 8547	.8572	.85595
$q_0{'}$. 3487	. 3499	. 3493
e'	.0753	.0761	.0758
μ	85°41′	85°38′	85°40′

From these mean values the following elements are calculated, with which in adjoining column are the elements of Schmidt.

ELEMENTS OF CLAUDETITE

F	Palache	Schm	idt
a = 0.4092	$p_0 = 0.8535$	a = 0.4040	$p_0 = 0.8527$
c = 0.3493	$q_0 = 0.3483$	c = 0.3445	$q_0 = 0.3437$
$\beta = 94^{\circ}20'$	$\mu = 85^{\circ}40'$	$\beta = 93^{\circ}57'$	$\mu = 86^{\circ}03'$

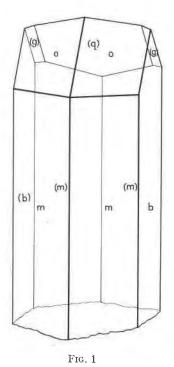
Table II gives the calculated position and interfacial angles of all known forms of claudetite together with some of the observed angles of Palache and Schmidt. The table is based on Palache's elements.

TABLE II. CLAUDETITE ANGLE TABLE

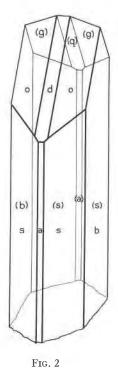
			Average	age lated	Observed, Palache	Palache		Calculated		Opse	Observed, Schmidt	idt
٥	0	001	,00°06	ρ 4°20′	φ	d	0	A 85°40'	B 90°00′	0	A 85°34′	$B 90^{\circ}00'$
9	8 0	010	00 0	00 06	,00.0	,00.06	,00.06	00 06	1	1	00 06	
a	0 8	100	00 06	3	90 14	3	85 40]	00 06	85°34′	1	90 05
**	1/1000	1.10.0	13 46	33	l	1	88 57	76 14	13 46	t	1	14 33
Α	8	150	26 06½	39	1	1	90 88	$63 53\frac{1}{2}$	$26\ 06\frac{1}{2}$	1	1	1
S	8	130	$39 14\frac{1}{2}$	3	$38 \ 45\frac{1}{2}$	00 06	$87 \ 15\frac{1}{2}$	50 451	$39 14\frac{1}{2}$	1	1	39 03
η	64hr	250	44 25½	3			86 58	45 341	44 253	1	I	Ì
	8	120		33	I		$86\ 38\frac{1}{2}$	39 13	50 47	1	[50 31
ш	8	110	67 48	3	67 38	00 06	$85 59\frac{1}{2}$	22 12	67 48	1		90 29
5	01	011	12 15	$19 ext{ } 40\frac{1}{2}$	1	l	$19 12\frac{1}{2}$	85 542	$70.39\frac{1}{2}$		85 48	71 02
82	02	021	$611\frac{1}{2}$	$35\ 05\frac{1}{2}$	1	1	$34\ 51\frac{1}{2}$	86 27	55 08½		1	55 03
α	04	041	$3.06\frac{1}{2}$	54 27	1	1	54 20	$87 28\frac{1}{2}$	35 40		I	
q	10	101	00 06	$4258\frac{1}{2}$	89 43	43 00	$38\ 38\frac{1}{2}$	$47\ 01\frac{1}{2}$	00 06	37 22		90 03
b	10	101	-90 00	$3757\frac{1}{2}$	-9014	37 52	$42\ 17\frac{1}{2}$	$52\ 02\frac{1}{2}$	33	42 24		90 22
0	1	111	69 27		$69\ 23\frac{1}{2}$	$44 48\frac{1}{2}$	40 49	48 40	75 40		48 24	75 48
6.0	Н	T11	-65 53	$40\ 31\frac{1}{2}$	-65 53	$50\ 29\frac{1}{2}$	$44\ 30\frac{1}{2}$	$-5337\frac{1}{2}$	74 36	1	-52 13	74 43
h	12	121	$-48 09\frac{1}{2}$	$46\ 19\frac{1}{2}$	-4824	46 15		-5724	61 09	1	[-
и	17	171	$20.51\frac{1}{2}$	69 05]	67 36	70 34½	$29 12\frac{1}{2}$	1	1	I

It will be noted in this table that the agreement of calculated position and interfacial angles with observed angles is for almost all forms excellent. Reference to Schmidt's article will show further that for interfacial angles his measurements agree better in all cases with the calculated values from the new elements than with his own calculated values. There seems no doubt that the new elements are established.

The habits of the claudetite crystals from the two new localities are shown in figures 1 and 2.



Claudetite, Imperial Valley, Calif.



Claudetite, Jerome, Ariz.

MINASRAGRITE

Minasragrite is a highly hydrous sulphate of vanadium first described and named by Schaller.³ As the name implies, the mineral

³ Schaller, W. T., Minasragrite, a hydrous sulphate of vanadium: Wash. Acad. Sci. Jour., vol. 7, p. 501, 1917.

was found at the vanadium mine at Minasragra, Peru, where it occurs as a blue incrustation on patronite. Schaller described the physical properties of the new mineral and assigned to it the formula V₂O₄.3SO₃.16H₂O. He found no measureable crystals but determined from its optical properties that it was monoclinic or triclinic.

Specimens of patronite in the Harvard mineral collection, which in the course of many years have become coated with various al-

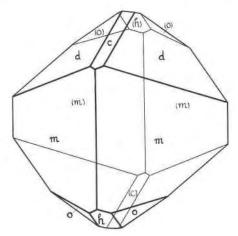


Fig. 3. Minasragrite

teration products, showed in places a vivid blue incrustation which proved to be minasragrite. The crust is composed of minute but very perfect crystals forming an open net work which falls to pieces at a touch. Some of the tiny crystals are quite complete and proved to be measureable. The two best which were completely measured, have an average diameter of .2 mm. and a maximum dimension of .25 mm. Notwithstanding their minute size the crystals gave excellent reflections and proved to be monoclinic. The habit of the crystals is shown in figure 3. The measured and calculated angles of the forms are contained in Table I. Table II contains calculated interfacial angles.

No close relation in form to any previously described sulphate is apparent from these angles.

Efforts to prove the presence of any distinct cleavage were not successful. The crystals were so minute that the handling of them

TABLE I. ANGLE TABLE FOR MINASRAGRITE

$p_0 = 0.9251$	$q_0 = 0.6216$	$\mu = 69^{\circ}03'$	
$p_0' = 0.9905$	$q_0' = 0,6656$	e' = 0.3827	
a = 0.7196	c = 0.0656	$\beta = 110^{\circ}57'$	

		0	Calculated		M	Measured		
		٠	Q.	*	Range	d	Range	No. of faces
0	001	,00.06	20°57′	90°22′	90°00′ –90°44′	21°01′	20°56′-21°06′	2
8	110	26 06	00 06	56 09	56 04 -56 15	00 06	ı	7
01	011	29 54	37 31	$29 50\frac{1}{2}$	$29 \ 16\frac{1}{2} - 30 \ 07$	37 31	37 21 -37 35	4
10 I	101	00 06-	$31\ 17\frac{1}{2}$	00 06	-90 05 -89 55	31 17	31 10 –31 24	2
j—	Ĭ11	-4224	42 02	42 16	42 05 -42 31	42 02	42 00 -42 02	3

Table II. Interpacial Angles of Minasragrite

Angle to 001		Angle to 100	Angle	Angle to 010
m 72°4	.4' m	33°54′	ш	56°06
31 52	i2 d	72 19\\\	p	58 08
52 1	4 <u>1</u> h	$58 \ 42\frac{1}{2} \ (\text{to I}00)$	N	00 06
57 50	0.2	63 10 (to T00)	0	60 22

was difficult. There seems to be a tendency to break in the general direction of the side sinacoid but with no true cleavage parallel to that plane as Larsen suspected on optical ground.

SAMSONITE

The crystal form of samsonite has been described by three authors whose measurements yield closely agreeing results. W. Bruhns⁴ calculated elements from three angles between two forms. His position differs from that adopted by later observers and here used. Calculating his measurements to the new position, we obtain the elements:—

$$a = 1.2787$$
, $c = 0.8204$, $\beta = 92^{\circ}42'$

F. Slavik⁵ gave a fuller description based on the measurement of many crystals. His elements, derived from three angles between three forms, are:—

$$a = 1.2776$$
, $c = 0.8180$, $\beta = 92^{\circ}46'$

The third description is by F. Kolbeck and V. Goldschmidt, who measured six crystals on the two-circle goniometer. The elements calculated from their two best crystals are:—

$$a = 1.2777$$
, $c = 0.8192$, $\beta = 92^{\circ}42'$

The author measured five crystals, also by the two-circle method and his calculation yielded the elements:—

$$a = 1.2786$$
, $c = 0.8217$, $\beta = 92^{\circ}34'$

Since these four independent values for the elements are in such close agreement, it was felt that the mean of all of them was better than any one. This mean value is as follows:—

$$a = 1.2782,$$
 $c = 0.8198,$ $\beta = 92^{\circ}41'$
 $p_0 = 0.6414,$ $q_0 = 0.8189,$ $\mu = 87^{\circ}19'$

An angle table was calculated from these elements and has been published elsewhere.⁷ Table I contains the position-angles for all

- ⁴ Kristallform des Samsonit von St. Andreasberg, IV Jahresb. d. Niedersächs. geol. Vereines, Hannover, 1911.
- ⁵ Morphologie des Samsonits, Bull. internat. de l'acad. des sciences de Bohême, 1911, p. 16.
 - ⁸ Ueber Samsonit von Andreasberg, Zeit. für Krist., vol. 50, p. 455, 1912.
- ⁷ Crystallographic Angle-Tables, 1933. Dept. of Mineralogy, Harvard University, privately printed.

 $\label{eq:Table_Table} T_{\rm ABLE}\ I$ Samsonite, calculated and measured angles

	Calculated	ated	Mea	Measured	Lin	Limits	No.		;
•		d	· o	ď	ф	ď	faces	xIs.	Quality
0.0	0.000	,00.06	1	1	{		1	1	
00 06	00	3	90°17′	90.00	90°04′-89°43′	1.	2	6	V. DOOF
11	11 04½	a.	12 46	а	1		ι π	-	poor
14 38	38	30	14 25	ä	14 11 -14 34	1	9	17	v. good
21	23	3	21 39	'n	1 - 1]	, _	-	V DOOR
38 04	94	ä	37 37	3	37 33 –38 08		6	ייט	boog
57 27	27	7,7	58 01	ä	56 55 -58 38		γ:	~;	noor
9	312	22 25	4 33	22 37	1	1	· -	-	pood
3	$3.16\frac{1}{2}$	$39\ 23\frac{1}{2}$			1	i.	' [1	0
00 06	00	$14.37\frac{1}{2}$	90 04	14 32	90 01 -90 11	14°18′-14°42′	ın	ιĊ	good
77		34 34	90 04½	34 32	77 77	34 28 -34 38	4	4	v. good
00 06-	00	30 46	-90 05	30 53	90 00 90 04	30 49 -30 57	4	4	boog
33		61 59	-90 01	62 12	[1	-		good
"		72 27 3	-90 05	72 29 3	90 02 -90 04	72 25 -72 34	2	2	pood
$40.02\frac{1}{2}$	02 1/2	$46\ 57\frac{1}{2}$			1	1			
-35	29	45 22 }	-36 06	45 301	35 47 -36 17	45 25 -45 47	6	ıν	v. good
59 15	15	38 43	1		I		,	, [0
-19	57	$60\ 10^{\frac{1}{2}}$	$-20\ 15\frac{1}{2}$	$61\ 01\frac{1}{2}$	20 08 -20 23	60 38 -61 25	2	2	poor
-22 56	26	64 171	-22 59	64 28	22 53 –23 04	64 24 -64 35	ec.	. 2	DOOL

the forms hitherto observed and, for the forms observed by the author, the measured angles.

In Table II there are shown the forms found by the various observers.

Table II

Forms of Samsonite by various observers

		Bruhn's symbol	Slavik	Kol. & Cold.	Palache
b	010	010	_	x	-
a	100		X	x	X
q	140	-	х	-	3
S	130	_	?	?	x
n	120	140?	x	_	-
m	110	120?	X	х	X
l	210	_	x	x	5
k	012	-	_	-	X
i	011	-	-	х	-
f	103	203	x	_	X
d	101	T01	x	_	X
e	101	001	X	-	х
g	301	101	x	_	x
h	501	201	_	_	X
Þ	111	T11	-	х	-
π	T 11	011	x	x	x
r	212	212	-	_	_
x	T21	_	_	-	X
δ	473	_	x	_	x

It is somewhat remarkable that the forms observed by the four observers are so different, for the mineral samsonite seems to have been found in only two vugs in the Samson shaft at the St. Andreasberg mine and one would expect more uniformity in the habit of the crystals. The total absence of the orthodome forms on the crystals of Kolbeck and Goldschmidt is in strong contrast to their strong development in all others measured. The author's study

establishes (130) as a well-developed member of the form series and adds the two forms k(012) and $x(\overline{1}21)$, both weakly developed.

NATIVE SELENIUM

The element selenium has never been certainly identified as a mineral; nor have crystals of the metallic gamma-selenium ever been made artificially which were accurately measureable. Muthmann⁸ has, it is true, figured a combination of prism and rhombohedron of this substance, but the figure is based on approximate measurements on the microscope stage of the edges of minute crystals, and these measurements by no means justify his assertion that selenium is completely isomorphous with tellurium.

Much interest, therefore, attaches to specimens of selenium found in the fire-zone of the United Verde Mine which, like the claudetite already described, were secured through Mr. Wilke.

There are several hand specimens of quartzite and of fire-fritted sandstone partly encrusted with needles of selenium. The needles may reach a length of 2 cm. with a diameter not over 1 mm. Sometimes they are clustered in flattened sheets parallel to a prism face so that patches up to 5 mm. across reflect together. On one specimen the selenium is in melted films or in droplets of dark red glass. Loose crystals up to 2 mm. long were also present and while clearly of hexagonal form were generally bent and twisted so that they could not be measured. Many of the crystals were hollow and tube-like with facets on the edges. A few tiny crystals about .5 mm. in length proved to be undistorted and gave excellent reflections on the goniometer. They always showed the prism of the first order, m, and a rhombohedron. On the best crystal faces of other forms were found. The table shows the observations made on this crystal.

		No. of	Meas	ured			Calcu	lated
		faces	ϕ	Range	ρ	Range	ϕ	ρ
m	10 <u>T</u> 0	5	30°02′	$\pm 11'$	90°00′	0	30°00′	90°00′
a	1120	2	00 04	± 1	"	0	0.00	46
h	$21\overline{3}0$	4	19 22	± 15	"	0	19 06	"
r	$10\overline{1}1$	3	-3007	± 11	52 40	$\pm 4'$	-30~00	52 38
e	$01\overline{1}2$	1	29 17		34 20		30 00	33 13
f	$11\overline{2}3$	1	0.06	_	37 33		0.00	$37\ 05\frac{1}{3}$

⁸ Zeit. f. Krist., vol. 17, p. 357, 1889.

The mean angle ρ of the unit rhombohedron from the seven best readings on four crystals is 52°38′ with a range of 52°36′-52°43′. Calculating from this angle, we obtain the axial ratio and angles shown below in comparison with those of tellurium.

 $r \wedge r$

86°59½′

93 03

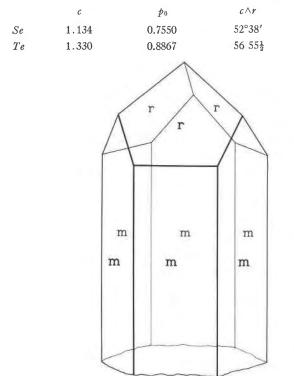


Fig. 4. Crystal of Native Selenium showing the forms $m(10\overline{1}0)$ and $r(10\overline{1}1)$.

Two crystals showed minute faces in the position of the negative rhombohedron. It was impossible to determine whether these were due to twinning on the vertical axis with complete penetration or to the normal development of this form. Figure 4 shows, idealized, the habit of the average crystal.

Cleavage of good quality was occasionally visible but, because of the softness and extreme flexibility of the crystals, it was very difficult to develop it without rendering the crystal unmeasureable. Approximate measurement showed, however, that it was parallel to the negative rhombohedron $e(01\overline{12})$. In tellurium the cleavage is prismatic and basal, another point of difference between the two elements.

A sample of the very homogeneous crystals was analyzed by F. A. Gonyer and proved to be without a trace of tellurium and to have but a trace of sulphur.

INDIUM

A sample of crystallized indium, prepared in the Harvard Chemical Laboratory by Chester M. Alter in 1932, was studied by Dr. M. A. Peacock. The sample consisted of arborescent groups of brilliantly silver-white crystals. Some of the groups were several centimeters long, but the individuals composing them were less than a millimeter in any dimension. The substance is so soft and wax-like that only occasionally could a crystal be detached without distortion. The symmetry proved to be tetragonal with close approach to isometric form. The angle $c \wedge p$, $(001) \wedge (111)$, is 56° 35′, the mean of nine measurements with the range 56°10′–57°00′. The value of c calculated from this angle is 1.072. The forms observed are c(001), a(100), m(110) and p(111). The arborescent groups were largely developed by elongation of the crystals parallel to an a-axis.