

powder at 30,000 psi to form a coherent specimen for x-ray analysis.

As we have pointed out previously (Welday *et al.*, 1964, p. 902) adequate rock standards for x-ray analysis are rare and the best (W-1 and G-1) are now in very short supply. In an effort to cooperate with other laboratories, limited samples of Pomona College Standard #5 will be made available upon request to those seriously interested. Larger supplies of Pomona College Standard #4 are available. This rock, another biotite quartz monzonite with a chemical composition similar to #5, has been analyzed and calibrated by the methods used for standard #5.

We thank Professor Volborth for reading the manuscript of this note, but we take full responsibility for the chemical values reported and the conclusions reached. This work was supported by National Science Foundation grants GP-1336 and GE-1038.

## REFERENCES

- BAIRD, A. K., D. B. MCINTYRE AND E. E. WELDAY (1963) Direct quantitative determination of oxygen in silicates by X-ray spectrography. Preliminary results (*abs.*) *Geol. Soc. Am., Cordilleran Sect., Ann. Meet.*
- (1963) Sodium and magnesium fluorescence analysis, Part II: application to silicates. *Advances in X-ray Analysis*, 6, 377, Plenum Press, New York.
- ESKOLA, P. (1954) A proposal for the presentation of rock analyses in ionic-percentage. *Ann. Finn. Acad. Sci. Ser. A*, III, 1-15.
- HENKE, B. L. (1964) X-ray fluorescence analysis for sodium, fluorine, oxygen, nitrogen, carbon and boron. *Advances in X-ray Analysis*, 7, 460, Plenum Press, New York.
- SHARMA, T. AND R. N. CLAYTON (1964) Oxygen analyses of minerals and oxides. *Anal. Chem.* 36, 2001.
- VOLBORTH, A. (1964) Biotite mica effect in x-ray spectrographic analysis of pressed rock powders. *Am. Mineral.* 49, 634.
- AND H. E. BANTA (1963) Oxygen determination in rocks, minerals and water by neutron activation. *Anal. Chem.* 35, 2203.
- WELDAY, E. E., A. K. BAIRD, D. B. MCINTYRE AND K. W. MADLEM (1964) Silicate sample preparation for light element analyses by X-ray spectrography. *Am. Mineral.* 49, 889.

THE AMERICAN MINERALOGIST, VOL. 50, MAY-JUNE, 1965

## DISCUSSION OF "EMPRESSITE AND STUETZITE REDEFINED"

BY R. M. HONEA

L. J. P. CABRI,<sup>1</sup> *Department of Geological Sciences,  
McGill University, Montreal, Canada*

I would like to make three comments regarding the very interesting paper on empressite and stuetzite from Colorado by Dr. R. M. Honea (1964, p. 325-338). These comments refer to:

- 1) Apparently incorrect statements regarding the assemblages  $Ag_{5-x}Te_3$ -petzite, and  $Ag_{5-x}Te_3$ -petzite-hessite.

<sup>1</sup> Present address: Dept. Mines and Technical Surveys, Ottawa, Ont., Canada.

- 2) Misleading reference to Galbraith's telluride identifications (in Eckel, 1949).
- 3) An interesting correlation between certain extra reflections in silver-rich compositions of synthetic  $\text{Ag}_{5-x}\text{Te}_3$  and some of the stronger reflections in the published AgTe pattern.

(1) It is difficult to understand Honea's statement (p. 337) that the natural associations involving  $\text{Ag}_{5-x}\text{Te}_3$  observed by him are compatible with Markham's (1960) 300° C. isothermal section of the system Au-Ag-Te. He reports the following associations observed from the Golden Fleece mine:  $\text{Ag}_{5-x}\text{Te}_3$ —petzite, and  $\text{Ag}_{5-x}\text{Te}_3$ -hessite-petzite. If these are equilibrium assemblages, then a tie line must occur between  $\text{Ag}_{5-x}\text{Te}_3$  and petzite (inferred by Markham as occurring only above  $315 \pm 10^\circ \text{C}$ ., and *not* at 300° C.). However, an alternate explanation of the associations observed by Honea is contained in the following paragraphs.

I have investigated the phase relations in the synthetic system Au-Ag-Te and have confirmed the existence of a phase intermediate in composition between  $\text{Ag}_{5-x}\text{Te}_3$  and  $\text{Ag}_2\text{Te}$ , first reported by Kiukkola and Wagner (1957). The presence of this phase, termed the gamma-phase, was confirmed using a high-temperature x-ray powder diffraction camera as well as with charges heated in sealed evacuated silica glass capsules and quenched. The x-ray powder data for this phase at 18° C. are given in Table 1. Two patterns are shown, because the presence of gold in solid solution gives better resolution with more measurable reflections. The silver content of the gamma-phase varies from 61.3 to 61.7 wt. per cent at 300° C. (Kiukkola and Wagner, 1957), and it is stable only between the temperature limits 120° to 460° C. above which it melts incongruently to  $\text{Ag}_2\text{Te}$  and liquid. The gamma-phase can be quenched for x-ray or polished section study at room temperature, but will noticeably decompose after thirteen hours to  $\text{Ag}_{5-x}\text{Te}_3$  plus hessite. It is similar to petzite in optical properties and etch reactions except for  $\text{FeCl}_3$  with which it etches grey-black with iridescent edges. This is quite distinct from the iridescent tarnish formed by petzite,  $\text{Ag}_{5-x}\text{Te}_3$ , or hessite.

Phase relations determined in isothermal sections of the area bounded by calaverite, sylvanite,  $\text{Ag}_{5-x}\text{Te}_3$ , hessite, and petzite at 356°, 335° and 290° C. show that the gamma-phase can take more than ten weight per cent Au into solid solution. Bulk compositions within this solid solution field will break down, on slow cooling, to form petzite +  $\text{Ag}_{5-x}\text{Te}_3$  + hessite, the assemblage reported by Honea. Markham (1960, p. 1161) states that the hessite-sylvanite join at 300° C. changes to a petzite- $\text{Ag}_{5-x}\text{Te}_3$  join above  $315 \pm 10^\circ \text{C}$ . This conclusion was based on the results of two runs at 330° C. whose compositions lay within the petzite-hessite-sylvanite field. These results are open to question since the compositions of these two runs, as given by Markham (p. 1155), fall on opposite sides of

the supposed petzite- $\text{Ag}_{5-x}\text{Te}_3$  join and yet both are reported to contain petzite + sylvanite +  $\text{Ag}_{5-x}\text{Te}_3$ .

2) This writer considers that caution should be maintained when referring to Galbraith's telluride identifications (Eckel, 1949, and Galbraith, 1940). Galbraith used only etch and microchemical tests for his identifications and, as reported by Honea (1964, p. 337), "hessite" from the May Day mine, La Plata district, Colorado, has been shown to be  $\text{Ag}_{5-x}\text{Te}_3$ . Furthermore, Galbraith (Eckel, 1949, p. 58) records a sub-graphic intergrowth of tiny worm-like inclusions of calaverite in hessite, a most unlikely assemblage in view of the synthetic system. Galbraith (1940) is the only writer to have described calaverite as being strongly anisotropic and to report petzite as galena-white in color.

3) At 194° and 170° C. several runs of a composition equivalent to  $\text{AgTe}$  all produced  $\text{Ag}_{5-x}\text{Te}_3 + \text{Te}$  after heating for three months, although ground and pelletized several times. Binary compositions equivalent to  $\text{Ag}_{5-x}\text{Te}_3$  (58.23 wt. % Ag) and more silver-rich compositions all gave the  $\text{Ag}_{5-x}\text{Te}_3$  pattern with at least five extra reflections ( $d = 10.15, 3.17, 2.80, 2.54, 2.22$ ); whereas binary compositions equivalent to 57.73 wt. % Ag and more tellurium-rich did not give these extra reflections but a pattern in perfect agreement with Berry and Thompson (1962). The three largest  $d$ -values of these extra reflections cannot be indexed according to the published patterns of  $\text{Ag}_{5-x}\text{Te}_3$  (Table 2). These extra reflections, which do not accompany the normal  $\text{Ag}_{5-x}\text{Te}_3$  x-ray pattern, cannot be attributed to the gamma-phase or hessite. It is noteworthy that these reflections correspond fairly closely to some of the stronger reflections in the pattern for natural  $\text{AgTe}$  as reported by Honea.

By a further coincidence, most, if not all of the remaining reflections in Honea's  $\text{AgTe}$  pattern also correspond to other reflections in the  $\text{Ag}_{5-x}\text{Te}_3$  pattern (Table 2). The reason for these relations is not understood at present. It might be noted that the pattern for synthetic  $\text{Ag}_{5-x}\text{Te}_3$ , which contains many more reflections than were observed by either Berry and Thompson (1962) or by Honea (1964), is in much closer agreement to the cell dimensions and indexing of Berry and Thompson (Table 2).

While I do not wish to question the occurrence of  $\text{AgTe}$  as a mineral, perhaps sufficient irregularities have been cited to indicate that the mineralogy of the silver tellurides is still far from completely understood. Is it not then premature to redefine the term "empressite" for which a composition near  $\text{Ag}_5\text{Te}_3$  has long been accepted, *e.g.* Thompson *et al.* (1951), Uytenbogaardt (1951), Kracek and Rowland (1955), Donnay *et al.* (1956), Markham (1957, 1960), Berry and Thompson (1962), and Cloke (1963)? Phase relations of the synthetic gold and silver tellurides will be clarified in more detailed account now in press.

TABLE 1. X-RAY POWDER DATA FROM GUINIER\* PHOTOGRAPHS.  $\text{CuK}\alpha_1$  RADIATION FOR  $\gamma$ -PHASE, d VALUES IN Å. RUNS QUENCHED IN ICE WATER FROM 335° C. AND X-RAYED IMMEDIATELY AT 18° C.  
( $\gamma_1=61.08$  Ag, 38.92 Te wt. %,  $\gamma_2=4.69$  Au, 57.76 Ag, 37.55 Te wt. %)

$\gamma_1^1$		$\gamma_2^2$	
I obs.	d meas.	I obs.	d meas.
		$\frac{1}{2}$	11.14
$\frac{1}{2}$	8.51	2	8.47
		$\frac{1}{2}$	8.10
		2	7.65
1	6.93	3	6.86
1	6.68		
		$\frac{1}{2}$	5.39
1	5.31	1	5.26
		$\frac{1}{2}$	5.13
		$\frac{1}{2}$ b**	4.43
2b	3.86	2	3.85
1	3.76	3	3.77
3	3.72	3	3.70
2	3.66	3	3.64
2	3.62	2	3.62
3	3.53	3	3.52
2	3.41	3	3.41
2	3.38	1	3.39
2b	3.31	3b	3.30
1b	3.20	1	3.20
3	3.09	4	3.09
		2	3.06
3	3.01	5b	3.02
		1	2.96
		$\frac{1}{2}$	2.94
3b	2.91	2	2.91
		3	2.81
		1b	2.83
8	2.69	6	2.69
1	2.67	2	2.66
3	2.63	4	2.63
1	2.61	$\frac{1}{2}$	2.61
		1b	2.58
		1b	2.54
		$\frac{1}{2}$	2.52

Reflections with smaller d-values are too broad or too weak or both—not measured.

\* Guinier forward reflection focussing camera.

\*\* Signifies broad reflection.

<sup>1</sup> A few reflections of hessite (low) ignored as well as one reflection of  $\text{Ag}_{5-x}\text{Te}_3$ .

<sup>2</sup> Only gamma-phase reflections observed on film.

TABLE 1—(continued)

$\gamma_1^1$		$\gamma_2^2$	
I obs.	d meas.	I obs.	d meas.
2	2.52	4	2.51
$\frac{1}{2}$	2.49	2	2.48
$\frac{1}{2}$	2.47		
$\frac{1}{2}$	2.46	$\frac{1}{2}$	2.46
1	2.43		
$\frac{1}{2}$	2.41		
3b	2.38	3	2.37
		$\frac{1}{2}$	2.29
		$\frac{1}{2}$	2.28
		$\frac{1}{2}$	2.26
		7	2.22
10	2.21	10	2.20
1	2.18	2	2.18
6	2.161	2	2.169
		6	2.164
6	2.149	7	2.150
4	2.120	5b	2.126
		$\frac{1}{2}$	2.115
		$\frac{1}{2}$	2.108
		$\frac{1}{2}$	2.099
4	2.095	4	2.091
1	2.070	1	2.071
2	2.059	3	2.056
		$\frac{1}{2}$	2.035
		1b	2.022
		$\frac{1}{2}$	1.994
		$\frac{1}{2}$	1.975
		$\frac{1}{2}$	1.968
		$\frac{1}{2}$	1.956
		$\frac{1}{2}$	1.936
$\frac{1}{2}$ b	1.930	2	1.923
		1	1.917
$\frac{1}{2}$ b	1.910	2b	1.906
1	1.878	3	1.883
1b	1.856	3	1.851
		1	1.840
		2	1.822

TABLE 2. X-RAY POWDER DIFFRACTION DATA FOR SYNTHETIC AND NATURAL  $\text{Ag}_{5-x}\text{Te}_3$  COMPARED TO NATURAL  $\text{AgTe}$ 

Natural $\text{AgTe}^1$ Honea (1964)		Synthetic $\text{Ag}_{5-x}\text{Te}_3^2$ Present Study			Natural $\text{Ag}_{5-x}\text{Te}_3^3$ (Empressite I) Berry & Thompson (1962)			Natural $\text{Ag}_{5-x}\text{Te}_3^4$ (Stuetzite) Honea (1964)			
I	d meas.	I	d meas.	hkl	I	d meas.	d calc.	I	d meas.	d calc.	hkl
		3b*	11.72	100			11.68	5	11.59	11.59	100
4	10.04	1	10.15								
		1	6.86	101			6.86			6.82	101
		$\frac{1}{2}$	5.27	111			5.27			5.24	111
		1	4.81	201			4.81	1	4.77	4.78	021
1	4.37	3	4.41	120	$\frac{1}{2}$	4.40	4.41	4	4.37	4.38	210
4	4.02	1	3.97	012			3.98			3.97	102
6	3.81	3	3.91	121	$\frac{1}{2}$	3.97	3.92	3	3.88	3.89	211
		4	3.58	112			3.59	6	3.56	3.57	112
		5	3.53	031	1	3.56	3.54	6	3.52	3.52	031
				022			3.43			3.41	022
6	3.33	4	3.37	220	$\frac{1}{2}$	3.40	3.37	5	3.35	3.35	220
		2	3.23	130	$\frac{1}{2}$	3.23	3.24	2	3.21	3.21	310
5	3.18	1	3.17								
		4	3.12	221			3.13	5	3.11	3.11	221
1	3.04	6	3.05	122			3.06	7	3.03	3.04	212
$\frac{1}{2}$	2.97	4	3.02	131	2	3.04	3.03			3.00	131
4	2.89	$\frac{1}{2}$	2.91	040			2.92			2.90	040
		$\frac{1}{2}$	2.86	302			2.87			2.85	302
3	2.85	2	2.82	003	$\frac{1}{2}$	2.83	2.83	3	2.82	2.82	003
10	2.70	2	2.80								
		$\frac{1}{2}$	2.75	041			2.76			2.74	041
		2	2.67	320			2.68			2.66	320
		6	2.63	222	1	2.64	2.64	7	2.62	2.62	222
2	2.60	1	2.60	113			2.61			2.60	113
		7	2.57	132			2.57	8b	2.55	2.56	132
		6	2.55	231, 140	5	2.55	2.55			2.54	321, 140
2	2.51	1	2.54	023			2.54			2.53	023
2b	2.43	1	2.43	141			2.44			2.42	141
		3	2.40	042			2.40	$\frac{1}{2}$	2.39	2.39	042
		2	2.37	213			2.38	$\frac{1}{2}$	2.37	2.37	213
4	2.32	3b	2.33	050			2.33	3	2.32	2.32	500
1	2.29	3	2.28	033			2.29	2	2.27	2.28	033
		3	2.26	232			2.26	4	2.24	2.25	322
		4	2.24	051			2.25			2.24	051
8	2.23	$\frac{1}{2}$	2.22	330	1	2.24	2.25			2.23	330
		2	2.20	240			2.21			2.19	240
3	2.18	10	2.18	142			2.18	10b	2.16	2.17	412
		6	2.17	331	10	2.17	2.17			2.16	331
5	2.14	4	2.163	223			2.16			2.15	223
4	2.12	2	2.127	241			2.14	6	2.11	2.12	421
		4	2.118	133	2	2.12	2.13			2.12	133
		1	2.095	004			2.12			2.11	004

\* Signifies broad.

<sup>1</sup> Orthorhombic,  $Pmm$  or  $Pmn$ ,  $a=8.90$ ,  $b=20.07$ ,  $c=4.62$  Å from Empress Josephine mine, Kerber Creek district, Colorado.<sup>2</sup> 58.23 wt. % Ag (synthesized from the pure elements in sealed evacuated silica glass capsules, initially melted, ground and pelletized; heated @ 335° C. for total 13 days, ground after four and six days—quenched in ice water). Pattern obtained with a Guinier forward reflection focussing camera using  $\text{CuK}\alpha_1$  radiation, and  $\text{LiF}$  as internal standard ( $a=4.0270$  Å).<sup>3</sup> Hexagonal,  $P6/mmm$ ,  $a=13.49$ ,  $c=8.48$  Å from Empress Josephine mine, Kerber Creek district, Colorado.<sup>4</sup> Hexagonal,  $C6/mmm$ ,  $a=13.38$ ,  $c=8.45$  Å from May Day mine, La Plata district, Colorado.

TABLE 2—(continued)

Natural AgTe <sup>1</sup> Honea (1964)		Synthetic Ag <sub>8-x</sub> Te <sub>8</sub> <sup>2</sup> Present Study			Natural Ag <sub>8-x</sub> Te <sub>8</sub> <sup>3</sup> (Empressite 1) Berry & Thompson (1962)			Natural Ag <sub>8-x</sub> Te <sub>8</sub> <sup>4</sup> (Stuetzite) Honea (1964)			
I	d meas.	I	d meas.	hkl	I	d meas.	d calc.	I	d meas.	d calc.	hkl
		$\frac{1}{2}$	2.084	014			2.08			2.08	014
5	2.04	3	2.044	052			2.05	4	2.03	2.03	052
4	2.01	3	2.032	151	1	2.04	2.04			2.02	511
				043			2.03			2.02	043
				114			2.02			2.01	114
1	1.962	1	1.982	332			1.986			1.972	332
		$\frac{1}{2}$	1.962	242			1.958			1.944	242
				060			1.947	5	1.931	1.933	323
		4	1.943	233	1	1.933	1.945			1.931	600
3	1.920	1	1.917	340			1.920	3	1.900	1.905	340
		3	1.907	124	$\frac{1}{2}$	1.910	1.911			1.903	214
		2	1.888	152			1.881	3	1.865	1.867	512
		3	1.877	341			1.873			1.858	431
$\frac{1}{2}$	1.864	2	1.866	250	$\frac{1}{2}$	1.873	1.871			1.855	520
		$\frac{1}{2}$	1.856	034			1.862				
$\frac{1}{2}$	1.833	2	1.823	251			1.827	$\frac{1}{2}$	1.816	1.812	521
1	1.796	2	1.796	224			1.795	$\frac{1}{2}$	1.790	1.790	053
1	1.771	2	1.770	134			1.774			1.786	224
2b	1.757	2	1.757	333			1.760	$\frac{1}{2}$ b	1.757	1.765	314
		2	1.708	252			1.711			1.756	062
2	1.699							$\frac{1}{2}$	1.699	1.707	044
		4	1.681	513			1.684			1.699	522
								2	1.674	1.674	513

## REFERENCES

- BERRY, L. G. AND R. M. THOMPSON (1962) X-ray powder data for ore minerals: The Peacock Atlas. *Geol. Soc. Am. Mem.* **85**.
- CLOKE, P. L. (1963) Synthetic and natural phases in the system Au-Ag-Te. *Econ. Geol.* **58**, 1163-1166.
- DONNAY, G., F. C. KRACEK AND W. R. ROWLAND (1956) The chemical formula of empressite. *Am. Mineral.* **41**, 722-723.
- ECKEL, E. B. (1949) Geology and ore deposits of the La Plata district, Colorado. *U. S. Geol. Survey Prof. Paper* **219**.
- GALBRAITH, F. A. (1940) Identification of the commoner tellurides. *Am. Mineral.* **26**, 368-371.
- HONEA, R. M. (1964) Empressite and stuetzite redefined. *Am. Mineral.* **49**, 325-338.
- KIUKKOLA, K. AND C. WAGNER (1957) Measurements on galvanic cells involving solid electrolytes. *Jour. Electrochem. Soc.* **104**, 379-387.
- KRACEK, F. C. AND W. R. ROWLAND (1955) The system silver-tellurium. *Ann. Rept. Geophys. Lab., Carnegie Inst. Washington Yearbook* **54**, 135-136.
- MARKHAM, N. L. (1957) Phase relations in the system gold-silver-tellurium. Unpubl. Ph.D. dissertation, Harvard University.
- (1960) Synthetic and natural phases in the system Au-Ag-Te. *Econ. Geol.* **55**, 1148-1178, 1460-1477.
- THOMPSON, R. M., M. A. PEACOCK, J. F. ROWLAND AND L. G. BERRY (1951) Empressite and stuetzite. *Am. Mineral.* **36**, 458-470.
- UYTENBOGAARDT, W. (1951) *Tables for Microscopic Identification of Ore Minerals*. Princeton Univ. Press, Princeton, N. J.