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The Staining Effect of the Hydrochloric Acid-Chromate Trioxide Solution on the Minerals of the Chalcocite-Stibnite-Galena Ternary System.

John W. Johns Jr.

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Johns, J. W. jr

THE STAINING EFFECT OF THE HYDROCHLORIC ACID-
CHROMATE TRIOXIDE SOLUTION ON THE MINERALS OF THE
CHALCOCITE-STIBNITE-GALENA TERNARY SYSTEM

By

John W. Johns Jr.

A Thesis
Submitted to the Department of Metallurgy
in partial fulfillment of the
Requirements for the Degree of
Bachelor of Science in Metallurgical Engineering

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JOHNS
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CHALCOCITE-STIBNITE-GALENA TERNARY SYSTEM

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INTRODUCTION

The object of this work has been to devise a method by which the different phases in the chalcocite-stibnite-galena ternary system may be identified. As the mineralogists have no precise methods for the identification of these phases, a hydrochloric acid-chromate trioxide staining solution, similar to that employed in previous investigations conducted in the Ore Dressing Department of the Montana School of Mines, was employed. The mechanics of this staining solution is not thoroughly understood. That the action is one of oxidation is apparent. The fact that metallic sulphides oxidize slowly, insuring the formation of thin films in the staining bath, and that the oxides of the metals are light colored and reasonably transparent seems to indicate that the action is one of chemical oxidation. The fact that the color assumed by the minerals varies with the time of immersion in a certain definite order corresponding with the increasing wave-length of light led Professor A. M. Gaudin to develop an interference-phenomenon theory as to the mechanics of this action.

"When light falls on a coated surface having a transparent coat, light is reflected both from the surface of the coat and from the surface of the substrate. If monochromic light is used, and the two reflecting beams are in phase, an intense color will be noted, that of the light used, of course; if the beams are in opposition, a faint color only will be seen, unless the two reflecting beams have the same intensity, in which case the surface will be devoid of color or black. With white light consisting of all the wave-lengths of light in the visible spectrum, some of the wave-lengths will be entirely missing or reduced in intensity in the compound reflected beam, thus causing the surface to appear colored.

Thus to get a film appearing of a color corresponding to wave length γ , that is, one in which the reflected beams of wave-length γ are in phase, the film should have a thickness of θ such that $2\theta = \gamma$. In the case of blue light ($\gamma = 0.5$), θ must be equal to 0.25.

Clearly the film will involve in the order: violet, blue, green, yellow, orange, red after which a second order of interference colors may appear in which the two reflected beams that give the color to the surface differ by two phases."

The staining method employed was to immerse the specimens by means of iron forceps in the staining solution for the required time. Then washing them under a stream of water to remove the staining solution and finally with acetone or alcohol to facilitate drying."

The materials used to prepare the specimens were cleaned galena, cleaned chalcocite, Merck's antimony sulphide (Sb_2S_3), and flowers of sulphur. The galena, chalcocite, and stibnite were weighed out in the proper proportions and thoroughly mixed with about one to two per cent excess sulphur. This material was then placed in small porcelain crucibles, covered with porcelain covers, and placed in a small muffle-furnace

which had been previously heated to a cherry red. The furnace was heated until all the excess sulphur burned off. The gas was then turned off and the specimens were allowed to cool slowly to room temperature in the furnace. The crucibles were then removed from the muffle, and the specimens dislodged by a slight shock, produced by gently knocking the crucible against a table. The material adhering to the side of the crucibles was carefully scraped off, combined with the specimen and weighed. If a loss greater than one or two per cent was encountered the specimen was discarded.

The specimens as they came out of the crucible resembled a small truncated cone. The small base was ground smooth on an emery wheel, and then polished with rouge on a wheel covered with old linen. A satisfactory polish was obtained by this method. The specimen was mounted on plasticene, examined as polished and again when immersed for various periods of time in the staining solution.

THE CHALCOCITE-STIBNITE BINARY SERIES

The following minerals are attributed by the mineralogists to the cuprous sulphide-antimony sulphide system of sulpho-salts:

Chalcocite	Cu_2S
Tetrahedrite	$3\text{Cu}_2\text{S} \cdot \text{Sb}_2\text{S}_3$
Chalcostibite	$\text{Cu}_2\text{S} \cdot \text{Sb}_2\text{S}_3$
Stibnite	Sb_2S_3

Seven specimens were prepared from ninety to thirty per cent stibnite, at ten per cent intervals. Following is a discussion of each specimen.

90% Sb_2S_3
 10% Cu_2S

This specimen contains 15% chalcostibite and 85% stibnite, which gives the following apparent composition:

4.8% Cu_2S
 95.2% Sb_2S_3

The only explanation for the discrepancy in the estimated composition is that the chalcocite is soluble in the minerals present, thus giving an apparent composition which does not check with the actual composition.

Table I gives the staining time for the two minerals.

TABLE I

COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF THE MINERALS IN THE 10% Cu_2S SPECIMEN.

<u>TIME</u>	<u>STIBNITE</u>	<u>CHALCOSTIBITE</u>
5 seconds	white	white
30 seconds	blue	yellow
45 seconds	secondary colors	blue

80% Sb_2S_3
20% Cu_2S

This specimen contains 34% chalcostibite, 64% stibnite, and 1% tetrahedrite, which gives an apparent composition of:

11.5% Cu_2S
88.5% Sb_2S_3

The tetrahedrite has crystallized out in small, three-pointed crystals, resembling a shamrock.

Again, in this specimen the apparent composition is high in stibnite and low in chalcocite, which indicates that Cu_2S is soluble in the other minerals.

The tetrahedrite does not stain, but remains white.

Table II contains a summary of the staining time of the other two minerals.

TABLE II
COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE MINERALS OF
THE 20% Cu_2S SPECIMEN.

<u>TIME</u>	<u>STIBNITE</u>	<u>CHALCOSTIBITE</u>
5 seconds	white	white
30 seconds	Purple to dark blue	brown
45 seconds	secondary colors	Dark blue to brown
50 seconds	secondary colors	blue

70% Sb_2S_3
30% Cu_2S

This specimen contains 79% chalcostibite, 10% tetra-
drite and 11% stibnite, which gives the following apparent
composition:

30.5% Cu_2S
69.5% Sb_2S_3

The chalcostibite seems to be faster staining whenever
it comes in contact with the tetrahedrite. An explanation
for this phenomenon is that when the melt cooled the tetra-
hedrite became solid, partially reacted with the liquid form-
ing chalcostibite. As the temperature was falling too rapid-
ly for this reaction to go to completion, some of the tetra-
hedrite was left, along with the chalcostibite already formed,
and a liquid lower in Cu_2S than chalcostibite. This substance
would then have a tendency to stain faster than chalcostibite.

TABLE III
COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF
THE MINERALS IN THE 30% Cu_2S SPECIMEN

<u>TIME</u>	<u>STIBNITE</u>	<u>CHALCOSTIBITE</u>
5 seconds	white	white
30 seconds	blue	yellow
45 seconds	light blue	purple to dark blue

60% Sb_2S_3
40% Cu_2S

75% chalcostibite and 25% tetrahedrite were present in this specimen, giving an apparent composition of:

38.5% Cu_2S
61.5% Sb_2S_3

The apparent composition checks with the composition as prepared, indicating that there is little solubility of Cu_2S in any of these minerals. The tetrahedrite does not stain but remains white through out the entire staining time.

The chalcostibite shows gradations around its edges, which are slower staining.

TABLE IV

COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF THE CHALCOSTIBITE IN THE 40% Cu_2S SPECIMEN.

<u>TIME</u>	<u>COLOR</u>
5 seconds	white
30 seconds	yellow
45 seconds	purple brown
50 seconds	dark blue

50% Sb_2S_3
50% Cu_2S

This specimen contains 15% chalcostibite and 85% tetra-
hedrite, which gives the following apparent composition:

Cu_2S 54.3%
 Sb_2S_3 45.7%

The chalcostibite was the last to crystallize out, and
fills all the voids between the tetrahedrite crystals. When
stained the chalcostibite has a tendency to peel, producing
a light blue color. From Table V, it can be seen that the
chalcostibite is considerably slower staining than usual.

TABLE V

COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF
THE CHALCOSTIBITE IN THE 50% Cu_2S SPECIMEN.

<u>TIME</u>	<u>COLOR</u>
20 seconds	white
45 seconds	light buff
60 seconds	yellow
90 seconds	blue

40% Sb_2S_3
60% Cu_2S

This specimen contains 19% chalcocite, 79% tetrahedrite,
and 2% chalcostibite, which give an apparent composition of:

65.4% Cu_2S
34.6% Sb_2S_3

This specimen is just on the Cu_2S side of the tetrahedrite composition. In this field tetrahedrite and chalcocite should be the only two minerals present, but as it is so close to the tetrahedrite-chalcostibite field, it would be almost impossible to eliminate all the chalcostibite. There is an eutectic formed between the tetrahedrite and the chalcocite.

The chalcostibite in this specimen is very slow staining, taking five minutes to turn blue.

30% Sb_2S_3
70% Cu_2S

This specimen contains 20% chalcocite and 80% tetrahedrite, giving the following apparent composition:

66.4% Cu_2S
33.6% Sb_2S_3

A very fine eutectic constitutes about 20% of the melt.

The chalcocite can be easily distinguished from the tetrahedrite, by the blue coating formed on the chalcocite as contrasted with the white tetrahedrite. This blue coating is undoubtedly formed by an oxidation reaction but does not show interference colors. This is provably due to the formation of cupric sulphide and not an oxide. The color of this coating somewhat resembles the color of covellite (CuS).

SUMMARY

Figure 2 is an equilibrium diagram prepared from the data obtained in this investigation. This diagram is offered only as a possibility, as the data is not conclusive. The

following changes in the diagram seem to be tolerable.

1. There is undoubtedly a solubility of Cu_2S in the stibnite as shown from the apparent composition in the high Sb_2S_3 specimens.
2. Tetrahedrite was first observed in the 20% Cu_2S specimen and continued to form in all the following melts. Thus it seems possible that the tetrahedrite field should be extended nearer the Sb_2S_3 side of the diagram.

A method has been devised by which the Cu_2S - Sb_2S_3 minerals can be indentified. Table VI is a summary of the colors obtained at various staining times on the surface of the minerals.

TABLE VI

SUMMARY OF THE COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF THE MINERALS IN THE Cu_2S - Sb_2S_3 SPECIMENS

MINERAL	TIME		
	5 sec.	30 sec.	45 sec.
Stibnite	cream	blue	secondary colors
Chalcostibite	white	yellow	blue
Tetrahedrite	white	white	white
Chalcocite	slate blue	slate blue	slate blue

Note. In the high copper sulphide specimens the chalcostibite took five minutes to turn blue.

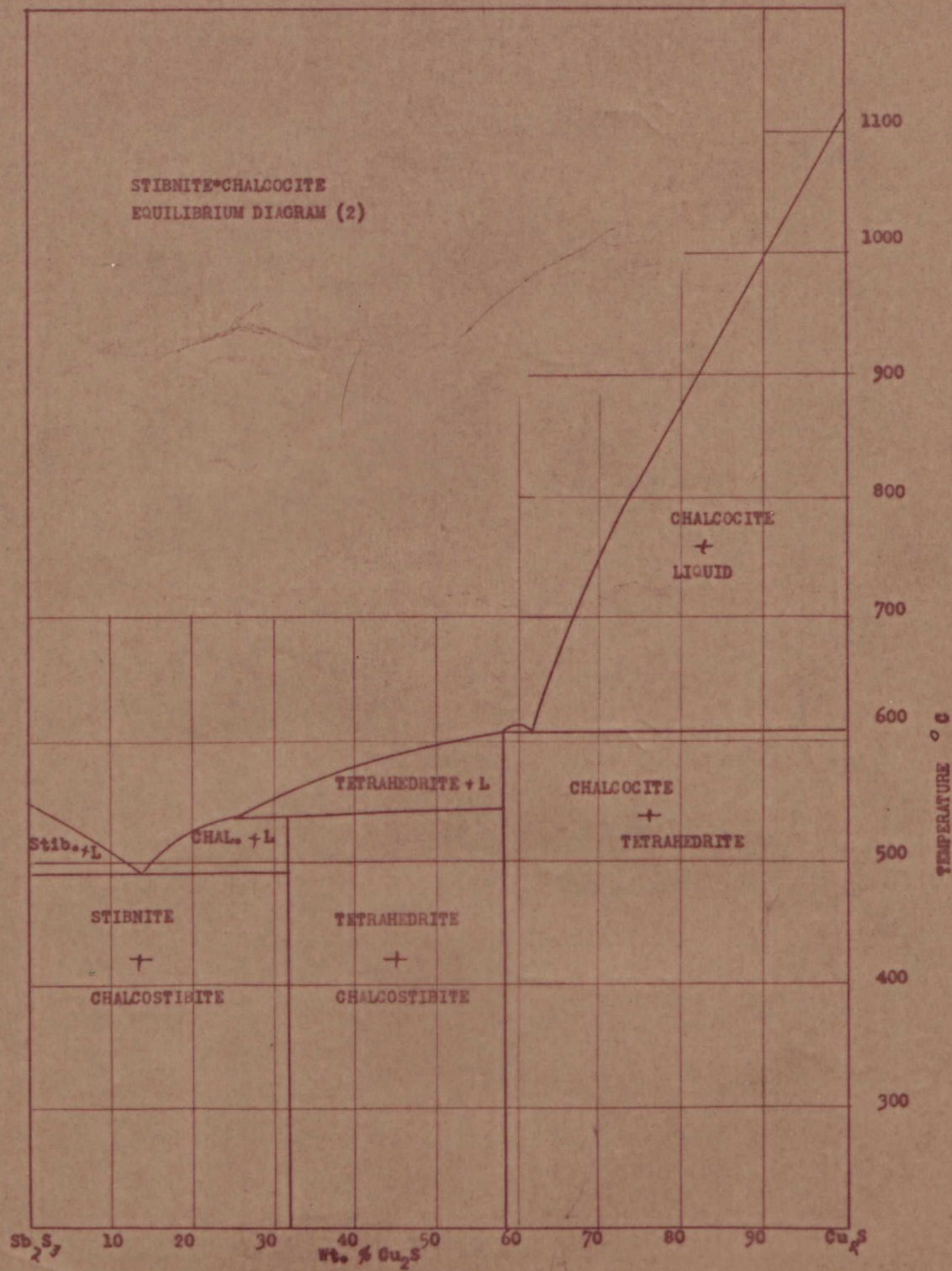


Figure 1

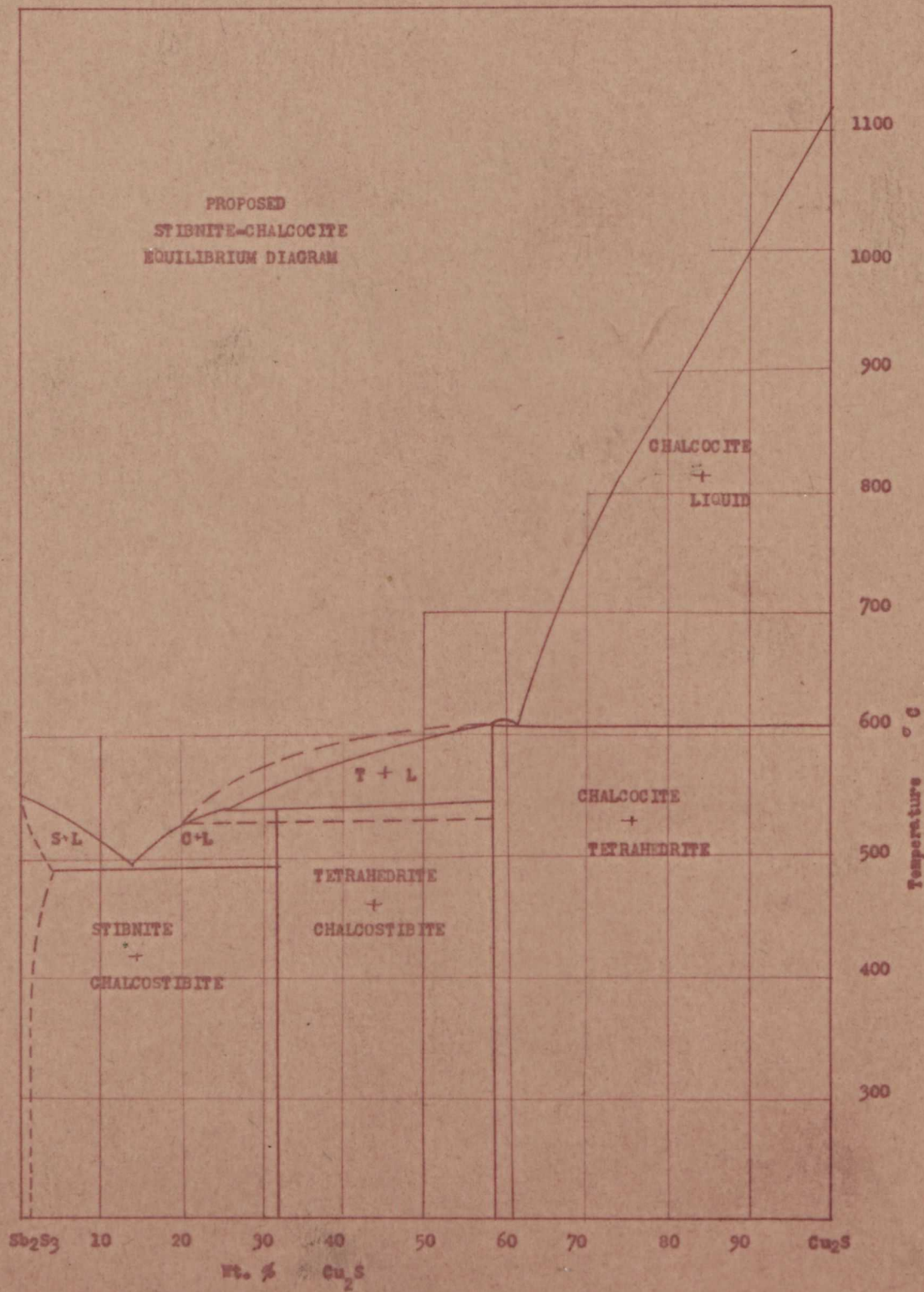


FIGURE 2

THE GALENA-STIBNITE BINARY SERIES

Following is a list of minerals attributed by mineralogists to the lead sulphide-antimony sulphide system of sulfosalts:

Galena	PbS
Quirozite	23PbS.Sb ₂ S ₃
Kilbrickenite	6PbS.Sb ₂ S ₃
Geocronite	5PbS.Sb ₂ S ₃
Meneghinite	4PbS.Sb ₂ S ₃
Embrithite	10PbS. 3Sb ₂ S ₃
Boulangerite	3PbS.Sb ₂ S ₃
Semseyite	9PbS.4Sb ₂ S ₃
Jamesonite	2PbS.Sb ₂ S ₃
Heteromorphite	7PbS.Sb ₂ S ₃
Warrenite	3PbS.2Sb ₂ S ₃
Plagionite	5PbS.4Sb ₂ S ₃
Zinkenite	PbS.Sb ₂ S ₃
Fuloppite	2Pbs.3Sb ₂ S ₃
Stibnite	Sb ₂ S ₃

Figure III represents an equilibrium diagram (3) of this system. According to this diagram the only minerals that are formed are; stibnite, zinkenite, warrenite, jamesonite, boulangerite, and galena.

It seems possible that the other minerals do not exist, but that what is supposed to be these minerals is in reality a mechanical mixture. If the minerals are actually formed

in nature, they must be formed under conditions different from those obtained in this investigation.

Seven specimens were prepared in this series, from ninety to thirty per cent stibnite, at ten per cent intervals. Following is a discussion of each specimen.

90% Sb_2S_3
10% PbS

This specimen contains 80% stibnite, 13% zinkenite, and 7% of an unknown mineral. This unknown mineral is high in lead sulphide as it is fast staining. It is either jamesonite, boulangerite, or warrenite. The above estimation, considering the unknown mineral to jamesonite, gives the following apparent composition:

Sb_2S_3 9.5%
 PbS 90.5%

The presence of this unknown mineral can be explained by the fact that when the material was heated the galena was the last to melt. Sufficient time was not allowed for this galena to diffuse through the material, and as a result, a small volume, around where the galena crystal had been, contains a high percentage of lead sulphide. As the temperature was lowered this material formed jamesonite, which did not have time to react with the liquid to form zinkenite. Thus some of the jamesonite was left in the specimen as it solidified.

Table VII presents a summary of the staining times of these constituents.

TABLE VII

COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF
MINERALS IN THE 10% PbS SPECIMENS.

<u>TIME</u>	<u>STIBNITE</u>	<u>ZINKENITE</u>	<u>UNKNOWN</u>
5 seconds	white	buff	brown
10 seconds	tan	yellow	light blue
15 seconds	brown	purple	secondary yellow
20 seconds	purple brown	blue	secondary colors
30 seconds	blue	secondary colors	

80% Sb₂S₃
20% PbS

This specimen contains 50% stibnite, 42% zinkenite, and 8% of an unknown, fast staining lead mineral. These estimations give the following apparent composition, considering the unknown to jamesonite:

22.4% PbS
77.6% Sb₂S₃

The unknown mineral shows gradation on staining, which indicates that the jamesonite was transforming into zinkenite, but the reaction was stopped before it went to completion. This mineral is the first to crystallize, followed by zinkenite. The stibnite forms the groundmass.

TABLE VIII

COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF
THE MINERALS IN THE 20% PbS SPECIMENS.

<u>TIME</u>	<u>STIBNITE</u>	<u>ZINKENITE</u>	<u>UNKNOWN</u>
5 seconds	cream	yellow	brown to purple
10 seconds	yellow	purple-brown	blue to light blue
15 seconds	dark yellow	purple	light blue
20 seconds	purple-brown	light blue	secondary yellow
30 seconds	blue	secondary colors	secondary colors

70% Sb_2S_3
30% PbS

This specimen contains 40% stibnite, 56% zinkenite, and 4% of an unknown mineral, similar to the unknown in the previous specimens. The above estimations give the following apparent composition, considering the unknown to be jamesonite:

27.8% PbS
72.2% Sb_2S_3

The unknown mineral was the first to solidify, followed by zinkenite. The stibnite forms the groundmass.

TABLE IX

COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF
THE MINERALS OF THE 30% PbS SPECIMEN.

<u>TIME</u>	<u>STIBNITE</u>	<u>ZINKENITE</u>	<u>UNKNOWN</u>
5 seconds	white	yellow	brown
10 seconds	white	yellow	blue
15 seconds	light yellow	brown	light blue
20 seconds	yellow	blue	secondary colors
30 seconds	dark blue	light blue	secondary colors

60% Sb₂S₃
40% PbS

This specimen contains 24% zinkenite, 71% jamesonite, and 5% unknown. This unknown is faster staining than the unknown mineral in the previous specimens. The above estimation, considering the unknown to be warrenite, gives the following apparent composition:

54.05% PbS
45.95% Sb₂S₃

This specimen contains no stibnite, which indicates that it falls into the zinkenite-warrenite field, instead of in the zinkenite-stibnite field. A small amount of Sb₂S₃ volatilized would cause this. Assuming that the above is true, no jamesonite should be present. Thus the specimen was cooled too rapidly to permit the jamesonite to decompose into zinkenite and warrenite. The appearance of the specimen verifies

the above statements. That is the Jamesonite was the first to crystallize. The warrenite is formed around the crystals faces of the jamesonite. Zinkenite forms the groundmass.

TABLE X

COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF THE MINERALS IN THE 40% PbS SPECIMEN.

<u>TIME</u>	<u>ZINKENITE</u>	<u>JAMESONITE</u>	<u>WARRENITE</u>
5 seconds	white	yellow	purple to dark blue
10 seconds	yellow	dark blue	secondary colors
20 seconds	dark blue	secondary yellow	" "
30 seconds	secondary colors	secondary colors	" "

The three remaining specimens, from 50% to 30% Sb_2S_3 , have little if any value. In these compositions, very complicated reactions take place, making it very difficult to obtain equilibrium. In all these specimens there are four or five different constituents, some undoubtedly are products obtained by incomplete reactions. Thus no conclusive data were obtained on the staining time of the minerals with the exception of galena, in these specimens.

Galena was the only mineral that could be positively identified. It stains in less than one second to a blue color, which on longer staining forms small crystals on the surface, thus producing a very rough appearance under the microscope. These crystals are probably a lead chloride compound which obtain a red coloration from the staining solution retained in the crystals.

STIBNITE-GALENA
EQUILIBRIUM DIAGRAM (3)

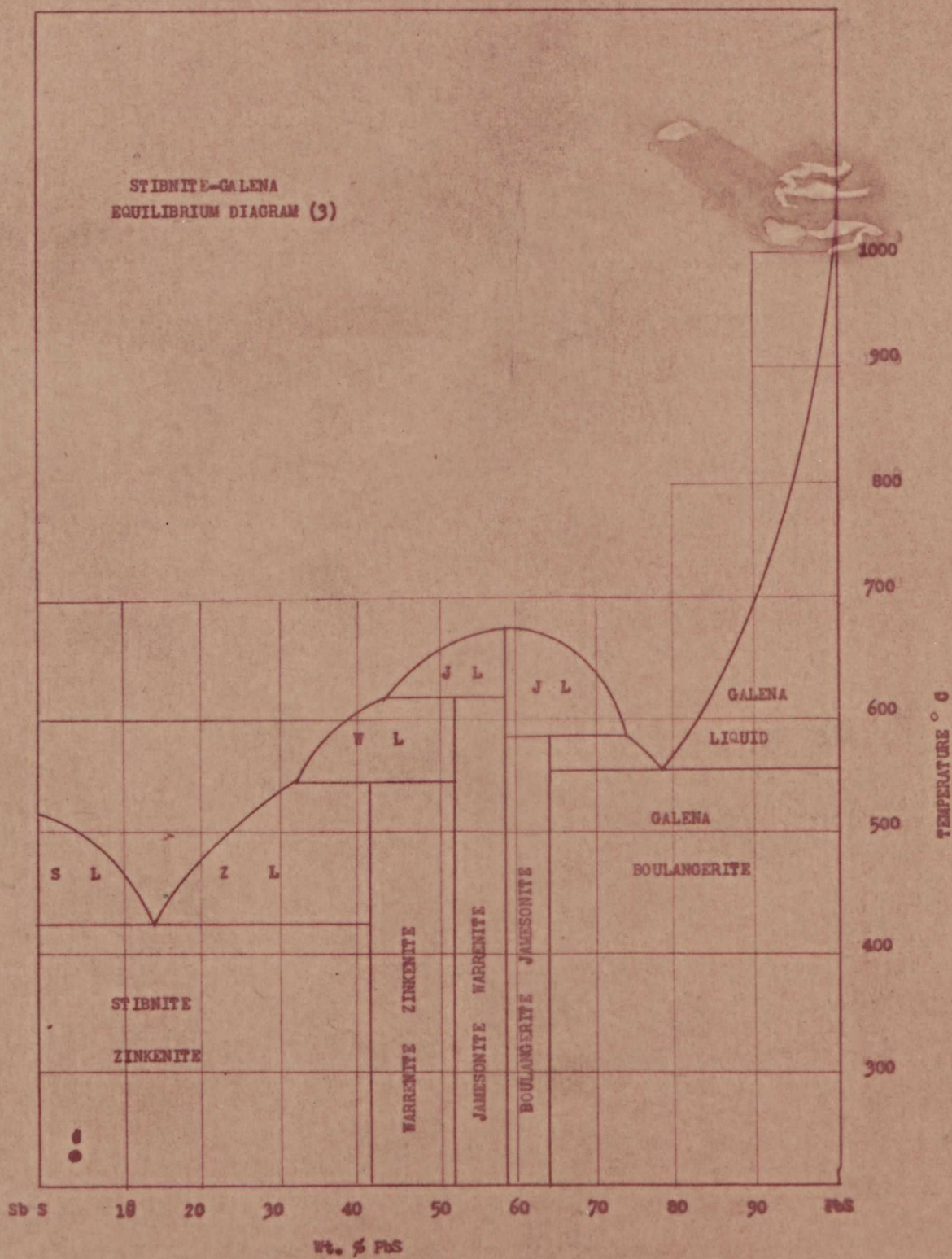


FIGURE 3

SUMMARY

A method has been devised, by which stibnite, zinkenite, jamesonite, and galena can be identified. However, no conclusive data was obtained as to the effect of the staining solution on boulangerite and warrenite.

Table XI contains a summary of the colors obtained at various staining times on the minerals indentified.

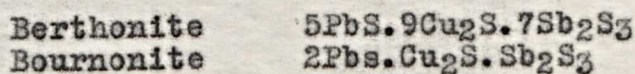
TABLE XI
COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF
THE MINERALS IDENTIFIED IN $PbS-Sb_2S_3$
BINARY SYSTEM

<u>TIME</u>	<u>Stibnite</u>	<u>Zinkenite</u>	<u>Jamesonite</u>	<u>Galena</u>
1 sec.	white	white	white	blue
5 sec.	white	cream	brown	sec. colors
10 sec.	buff	yellow	light blue	Rough surface
15 sec.	brown	purple	Sec. yellow	" "
20 sec.	purple brown	blue	sec. colors	" "
30 sec.	blue	sec. colors	" "	" "
45 sec.	sec. colors	" "	" "	" "

THE STIBNITE-GALENA-CHALCOCITE TERNARY SYSTEM

After the work progressed thus far on the binary systems, specimens were prepared in the stibnite-galena-chalcocite ternary system, in order to study the effect of the third constituent on the staining time of the minerals in the binary system.

In addition to the minerals already cited, the two following minerals are attributed to this ternary system by the mineralogists:



Neither of these two minerals were observed in this study.

As mentioned in the discussion of the galena-stibnite binary system, no distinction was obtained between boulangerite and warrenite, thus these two minerals and any intermediate reaction product formed are classified as boulangerite in the following tables. Table XII contains the twenty-four specimens prepared in this series, with the prepared composition, the estimated amount of each constituent present, and the apparent composition. Tables XIII to XVI, inclusive, contains the colors of each constituent at the various staining times.

SUMMARY OF THE TERNARY SYSTEM

The specimens were not placed close enough together to obtain any definite information on this ternary system. From the data obtained in the investigation of the ternary system the following are concluded:

1. No ternary minerals are formed.
2. The addition of Cu_2S has little, if any, effect on the staining rate of the minerals of the $\text{PbS-Sb}_2\text{S}_3$ binary system.
3. The addition of PbS has little, if any, effect on the staining rate of the minerals of the $\text{Cu}_2\text{S-Sb}_2\text{S}_3$ binary system.
4. The Sb_2S_3 seems to have a greater affinity for Cu_2S than it has for PbS . The $\text{Cu}_2\text{S-Sb}_2\text{S}_3$ minerals seems to form first and if any Sb_2S_3 is left, it unites with what PbS is present.

TABLE XIII

COLOR OF CHALCOSTIBITE, IN THE TERNARY SYSTEM, AT VARIOUS STAINING TIMES

	5	22½	5	22½	15	20	10	40	30	20	10	50	40	30	20	10
PbS	5	22½	5	22½	15	20	10	40	30	20	10	50	40	30	20	10
Cu ₂ S	5	7½	15	7½	15	20	30	10	20	30	40	10	20	30	40	50
Sb ₂ S ₃	90	90	80	70	70	60	60	50	50	50	50	40	40	40	40	40
5 SEC.	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A
A White																
D Light yellow	10	B	A	A	A	A	A	B	B	B	B	B	B	B	B	B
C Yellow	15		B					C				C	D	D	C	
D Brown	20	C	C	O	B	B	C	B	C	C	C	C	D	D	D	C
E Purple brown	25															
F Purple	30															
G Dark blue	45															
H Medium blue	60															
I Light blue	90															
J Secondary colors	120															

TABLE XIV

COLOR OF JAMESONITE, IN THE TERNARY SYSTEM, AT

VARIOUS STAINING TIMES

	PbS	10	5	30	20	10	40	30	20	10	50	20
	Cu ₂ S	10	15	10	20	30	10	20	30	40	10	40
	Sb ₂ S ₃	80	80	60	60	60	50	50	50	50	40	40
KEY	5 sec.	E	F	C	C	D	C	C	C	C	C	C
	10 sec.	F	H	G	G	H	H	H		D	G	
	15 sec.	I	I								I	
A White	20 sec.	J	J	J	J	J	J		I	H	J	I
	25 sec.		J									
B Light yellow	30 sec.	J	J	J	J	J	J		J	J	J	J
	45 sec.			J	J							
C Yellow												
D Brown												
E Purple brown												
F Purple												
G Dark blue												
H Medium blue												
I Light blue												
J Secondary colors												

TABLE XV

COLOR OF STIBNITE, IN THE TERNARY SYSTEM, AT

VARIOUS STAINING TIMES

	PbS	7½	5	2½	10	5	22½	15
	Cu ₂ S	2½	5	7½	10	15	7½	15
	Sb ₂ S ₃	90	90	90	80	80	70	70
	5 sec.	A	A	B	A	A	A	B
	10 sec.	B	B	B	B	B	A	B
	15 sec.	D			D	D		C
	20 sec.		F		F	E	C	C
	25 sec.	F			G			
	30 sec.	H	H	H	H	H	H	H
	45 sec.		J	J		J	I	I

KEY

- A White
- B Light yellow
- C Yellow
- D Brown
- E Purple brown
- F Purple
- G Dark blue
- H Medium blue
- I Light blue
- J Secondary colors

TABLE XVI

COLOR OF BOULANGERITE, IN THE TERNARY SYSTEM, AT
VARIOUS STAINING TIMES

D Brown	PbS	40	30	50	40	30	60	50
	Cu ₂ S	10	20	10	20	30	10	20
E Purple brown	Sb ₂ S ₃	50	50	40	40	40	30	30
F Purple	5 sec.	F	H	G	G	E	H	H
	10 sec.	G	I	I	I	F	I	I
G Dark blue	20 sec.	J	J	J	J	H		I
	25 sec.				J	J		
H Medium blue	30 sec.				J	J		

CONCLUSION

A method has been devised by which the minerals of the chalcocite-stibnite-galena ternary system, with the exception of baoulangerite and warrenite, may be identified.

No ternary minerals were observed.

Table XVII is a summary of the colors obtained at various staining times on the surface of the minerals observed.

TABLE XVII
COLORS OBTAINED AT VARIOUS STAINING TIMES ON THE SURFACE OF
THE MINERALS IN THE CHALCOCITE-STIBNITE-
GALENA TERNARY SYSTEM

Minerals	Time					
	Less than 1 sec.	5 sec.	10 sec.	20 sec.	30 sec.	45 sec.
Galena	blue	sec. colors	sec. colors	black	black	black
Jamesonite	white	brown	blue	sec. colors	sec. colors	sec. colors
Zinkenite	white	yellow	purple brown	blue	sec. colors	sec. colors
Stibnite	white	buff	yellow	brown	blue	sec. colors
Chalcostibite	white	white	light yellow	yellow	brown	blue
Tetrahedrite	white	white	white	white	white	white
Chalcocite	white	slate blue	slate blue	slate blue	slate blue	slate blue

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