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The Determination of  
Safflorite-Loellingite and Danaite  
at the Little Gem Mine.

Geology 9

The University of British Columbia

April

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### ACKNOWLEDGEMENTS

The author would like to thank Dr. H. V. Warren for his useful hints and suggestions and untiring assistance in connection with this work.

I would also like to thank Mr. R. Thompson for helping in the laboratory, and Mr. J. Donnan for his assistance in the polishing of the mineral sections. My thanks are also due to Mr. R. Taylor for supplying the material for the sections.

John L. De Leen.

April 1943.

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and Danaite at the Little Gem Mine.

Introduction

(1)

Location

The Little Gem mining property consists of 11 claims located on Roxy Creek, a tributary to Gun Creek, in the Lillooet Mining District of British Columbia.

In addition to trenching, two tunnels have been driven on the property. One tunnel at an elevation of 6400 feet follows high-grade ore for most of its total length of 200 feet. The other tunnel at 6350 feet elevation is 500 feet long and follows the ore for 60 feet.

Geology (1)

The country rock is a biotite-quartz-diorite. It is in the form of a tongue 2000 feet wide and  $2\frac{1}{2}$  miles long extending from a larger body of biotite-quartz-diorite that covers Gun Creek valley. A massive dark green serpentine borders the biotite-quartz-diorite to the north west and shows a banded structure along the contacts where it is badly sheared and broken. Numerous aplitic dykes traverse the biotite-quartz-diorite and follow closely a brown weathered dyke of ferruginous carbonate. There are numerous small diabase dykes.

(1)

Ore Occurances

The ore bodies appear to be lenticular and vary in

width from a few inches to several feet. Two veins which are thought to be part of the same ore body, are being developed at present. The foot wall vein which is vertical strikes east-west and is exposed for 50 feet on the surface. It varies in width from a few inches at the lower end to about six feet at the upper end, and is covered by an altered diorite cap-rock. The hanging wall vein is parallel to the foot-wall vein 17 feet to the south. This vein appears wider and longer than the foot-wall vein and averages six feet in width over 90 feet of length.

Throughout the ore bodies are numerous masses of quartz and feldspar. Very little alteration appears at the borders of the vein.

A shear, 6 to 12 feet wide, striking east-west was traced for 200 feet on the surface. The shear cross-section assays from 0.22% to 0.40% Cobalt. Where small dykes cut the shear, arsenopyrite and cobalt bloom are found.

#### MINERALOGRAPHY

Specimens of low grade ore were selected for the polished sections. Six sections were cut in triplicate. The author's sections were mounted in demar gum in brass molds. A

#### Reagents

The etch reactions of Ellis Thompson<sup>(2)</sup> were tried on samples of Rammelsbergite, Cobaltite, Safflorite-loellingite, arsenopyrite and Gordon Bell's section E.

Table of Etch Reactions from Ellis Thompson Report

	Conc. HNO <sub>3</sub>	1:1 HNO <sub>3</sub>	3:10 HNO <sub>3</sub>	Sat. FeCl <sub>3</sub>	Aqua R. CoNiAsS	A.R. & FeAsS
Arsenopyrite	dk. grey	Irrid. dk. bn. rem.	—	—	—	Ft. bn. rubs clean
Cobaltite	—	—	—	—	—	—
Rammelsbergite	dk. grey rem.	dk. grey bk. rem.	lt. bn. rem.	dk. bn. rem.	—	lt. dk. bn. rem.
Safflorite	grey diff. rem.	grey diff. rem.	ft. bn. diff. rem. or neg.	—	—	—
Loellingite	dk. grey rem.	dk. grey rem.	Ft. bn. rem.	—	lt. bn. diff. rem.	—

Results of Etch Reactions

Reagent	Arseno- Pyrite	Cobal- tite	Safflorite- Loellingite?	Rammels- bergite	Bell Section E (Saff-loel) Inclusion	(Danaite) ground mass
Conc. HNO <sub>3</sub>	dk. grey	—	dk. grey rem.	dk. grey rem.	dk. grey to brown	Irred. strong blue
1:1 HNO <sub>3</sub>	Irred.	—	dk. grey rem. to ft. brown	ft. grey	lt. grey to ft. brown	col. dk. brown
3:10 HNO <sub>3</sub>	ft. Irred.	—	ft. brown rem. to neg.	lt. brown	—	ft. bn.
Sat. FeCl <sub>3</sub>	—	—	—	dk. bn.	—	—
A.R. & FeAsS	—	—	—	dk. bn.	—	—
A.R. & CoNiAsS	—	—	—	—	—	—
HgCl <sub>2</sub>	—	—	—	—	—	—

These etch results checked closely with those of Mr. Ellis Thompson, and Mr. G. Bell.

From the results of the above tests, it was found that the inclusions (safflorite-loellingite) etched light grey and the ground mass (danaite) dark brown. Concentrated and 1:1 HNO<sub>3</sub> were found to be the best reagents to distinguish between safflorite-loellingite and danaite.

#### Microchemical Tests

Microchemical tests were run on pure samples of safflorite-loellingite and Danaite.

The following table gives the results of these tests.

Mineral	Iron	Cobalt	Arsenic
Safflorite-Loellingite	Present (Strong)	Positive (weak)	Present
Danaite	Present (Strong)	Positive (weak)	Present

No distinction between safflorite-loellingite and danaite was thus tried on the basis of microchemistry.

#### DETERMINATION OF DANAITE

The six sections were etched with 1:1 HNO<sub>3</sub>. All sections gave reactions of safflorite-loellingite or danaite. Section 1 was found to be composed of crystals of danaite in a black carbonaceous ground mass.

The opposite half of section 1 was polished and broken. The crystals were found to have the characteristic striations of arsenopyrite group given in Dana<sup>(3)</sup>. The crystal striations were as shown in fig. 1.

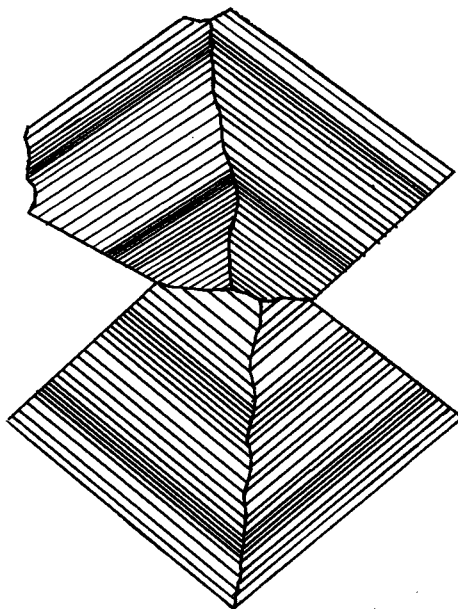


Fig. 1

Specific gravities of these crystals were:

6.05  
5.93  
5.64  
5.82  
5.80  
5.90

These specific gravities were found to check with those given in Dana, as follows;

Dana Sp. G. (Danaite)	5.9	6.3
De Leen Sp. G.	5.64	6.05

Some of the crystals were oxidized to a brown on the surface. This accounts for the large variation of specific gravity.

To check that the above samples were not arsenopyrite one of the crystals was spectrographically analysed. Cobalt and iron were found to be present in amounts exceeding one percent.



Sections giving the same etch test as used above were analysed by Mr. G. Bell. Bell's results checked closely with those of Dana:

	sample	As	S	Fe	Co	Total
Bell-percentage	B	47.50	17.11	29.00	6.39	100.00
Composition	F <sub>1</sub>	48.40	18.05	27.10	6.45	100.00
Dana-percentage		47.45	17.78	30.91	4.75	98.89
Composition for Danaite		41.11	17.84	32.94	6.56	98.78

X-ray powder photographs, of Bell's material, were found, by Dr. Holmes of Columbia University, to compare closely with the x-ray spectrogram of danaite. Samples of the above crystals were also sent to Dr. Holmes for further x-ray powder analysis. Dr. Holmes found that the x-ray powder photographs of these crystals to be that of pure danaite.

Thus with the crystal striations, the specific gravities, the percentage compositions, and the x-ray powder photographs, the mineral was proved to be danaite.

#### DETERMINATION of SAFFLORITE-LOELLINGITE

Sections E and F<sub>2</sub> of Bell were repolished and etched. The surfaces etched a faint brown to grey with 1:1 HNO<sub>3</sub> so a corner of section F was removed for examination.

The specific gravity of these samples agreed with the values of safflorite-loellingite given in Dana, thus

Dana	7.2
De Leen	7.1, 7.2

Furthermore Bell's percentage compositions agree with those of Dana, as follows:

	As	S	Fe	Co	Total
Bell-percentage Composition	70.00	1.67	22.0	6.32	99.99
$F_2$ $E^2$	65.30	3.68	25.30	5.84	100.12
Dana-percentage Composition for safflorite-loellingite	70.59 63.66	1.65 3.66	28.67 21.22	6.10 6.44	107.01 95.08

Thus by the etch reaction, the specific gravity and the percentage composition the mineral was proved to be safflorite-loellingite.

Samples of this safflorite-loellingite were also sent to Dr. Holmes of Columbia University for x-ray powder photograph. Dr. Holmes found the x-ray powder photographs of these samples to be that of safflorite-loellingite.

#### DESCRIPTION of SECTIONS

##### Section 1.

Section 1 (fig. 2) is composed of crystals of danaite in a black carbonaceous ground mass. It was the opposite half of this section that was used in the "Determination of Danaite" on page 4.



x 20

Fig. 2

### Section 2.

This section is a mixture of danaite and safflorite-loellingite, separated by small veinlets of calcite. These sulphides are surrounded by a green calcareous and a black carbonaceous gangue. The black gangue is replaced by radiating dendritic inclusions of molybdenum 3-4 millimeters in diameter.

### Section 3.

Section 3 is a mixture of the crystals of danaite and safflorite-loellingite in a greyish-green calcareous groundmass. The crystals of danaite etched differentially brown, from the surrounding safflorite-loellingite.

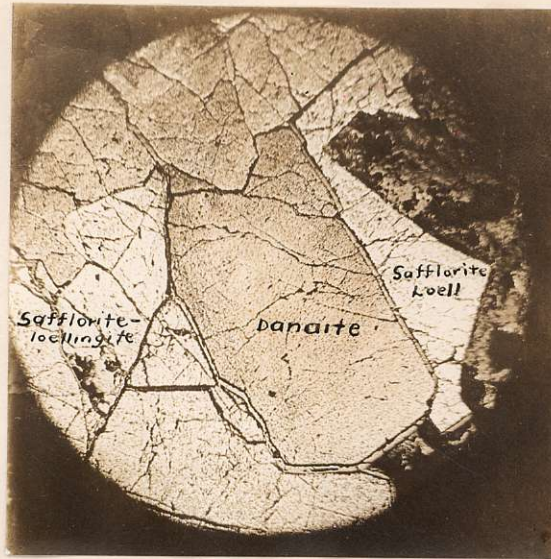
### Section 4.

Section 4 is a mixture of safflorite-loellingite and danaite. Figure 3 etched with 1:1 HNO<sub>3</sub>, shows a crystal of danaite separated from the safflorite-loellingite by a small fracture of quartz gangue. This fracture suggests the danaite and safflorite-loellingite were deposited contemporaneously, and the danaite on cooling contracted leaving the fracture. The fracture was later filled with quartz.

A small fracture and change in colour in the quartz suggests the injection of a second generation of quartz.

The ground mass is a mixture of greyish-green calcareous and black carbonaceous gangue. In the black carbonaceous gangue is a small dendritic inclusion, 1-2 mm. in diameter, of molybdenite. The molybdenite was also found in the black gangue of section 2. The molybdenite, a high

temperature mineral, may have been injected into the calcareous gangue, thus driving off the carbon-dioxide and leaving a black carbonaceous ground mass.

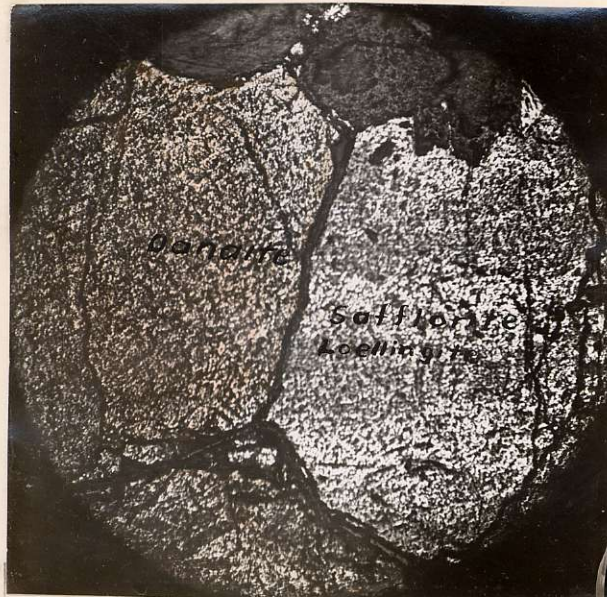


x 20

Fig. 3

#### Section 5

This section is similar to section 4. The crystals of danaite are separated from the safflorite-loellingite by small calcite veins (fig. 4).



x 45

Fig. 4

Section 6.

Section 6 is a solid mass of safflorite-loellingite cut by small veinlets of calcite.

Conclusion

The crystals of danaite are found in a black carbonaceous ground mass. A mixture of safflorite-loellingite and danaite (danaite crystals usually surrounded by safflorite-loellingite) is associated with a greyish green calcareous gangue. The solid sulphides tend to be pure safflorite-loellingite.

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Etch Tests.

Thompson's in black.  
Thomson's in red.

Mineral	Conc. HNO <sub>3</sub>	1-1 HNO <sub>3</sub>	3:10 HNO <sub>3</sub> .
Arsenopyrite FeAs <sub>2</sub>	Dark gray etch Rough surface remains. Dk. gray with rough surface remains.	Iridescent dk. brown etch remains. Irid. dk. br. remains.	Negative. Negative.
Loellingite FeAs <sub>2</sub>	Dark gray etch. rem. Some octahedra. Gray diff. rem.	Dark gray etch rem. Very few octahedra. Gray diff. rem.	Negative Negative.
Safflorite- Loellingite. FeAs <sub>2</sub>	Dark gray etch Abundant octahedra of As <sub>2</sub> O <sub>3</sub> ?	Gray etch Brings out relief. Stains black and brings out grain structure.	Negative.
Danaite.	Dark gray etch Rough surface remains. No octahedra.	Brown etch remains.	Negative

#  
Short

December 7, 1943

Confidential

Dr. J. F. Walker,  
Deputy Minister of Mines,  
Department of Mines,  
Victoria, B. C.

Dear Dr. Walker:

During the last week the editorial board of the Graduate Chronicle have been rounded up by our President, and I was asked to write an article on the Research Council apropos of minute 2 from our Annual Meeting of October 29th, 1943.

I discussed the matter with Dean Finlayson, and I pointed out that I spoke to the Annual Meeting just after the matter had been discussed by Faculty Committee and Board, but before we had been asked to keep the subject quiet until the Premier was ready to make his announcement. You may remember that the Board gave out a statement which, if I recall correctly, signified general approval with details to be worked out at a later date. I spoke briefly and gave no details except those which were available through the press, and Art Lord followed with a few words.

The Dean is very fearful of any statement appearing prematurely and when I told him what was already in our minutes and which would therefore be published next month, he seemed to feel that I had been ill-advised to discuss the matter at all with the alumni. Incidentally, Mr. Robinson, President of the alumni, can see no reason why information which has appeared in the papers should not appear in our minutes. Therefore, I telephoned Art Lord last night and he suggested that I drop you a line.

However, I hope you will feel that the enclosed minute is hardly deserving of so much worry and that it can be inserted without comment. You can readily see that the idea of removing minutes from a meeting presents difficulties.

I am very sorry to trouble you, but in view of Dean Finlayson's remarks and my ensuing discussions with Mr. Robinson and Art Lord, I thought that you had better be informed.

With kindest personal regards,

Sincerely yours,

c.c. Dean Finlayson  
Mr. Art Lord  
Mr. B. Robinson.

Harry V. Warren.



April 2, 1943.

Dr. Ralph J. Holmes,  
Department of Geology,  
Columbia University,  
New York City.

Dear Holmes,

Your letter of March 16 reached us and I think has settled the controversy about the minerals concerned once and for all. As I think I told you in New York last year we thought we had the answer over two years ago but in view of the controversial nature of our conclusions, nothing could be considered final until we had x-ray patterns.

I have seen Dr. Kidd and he is going to write to you about the Great Bear Lake matter. We have specimens here but they have not been critically determined. We are making up some samples of danaites and safflorite-löllingite. We can let you have several pounds more of the danaites, if you wish it. The Department and I myself particularly appreciate your kindness and cooperation and when we finally publish

this material we shall of course acknowledge  
all that you have done.

Kindest personal regards to Dr. Behre,  
Dr. Shand and yourself.

Sincerely,

Harry V. Warren

December 13th, 1941.

G.L. Mill, Esq.  
c/o Kelowna Exploration Co.  
Bedley, B.C.

Dear George,

As you know, I have your letter of November 14th concerning the possible composition of the Little Gem ore mixture on which you are running flotation tests.

We have spent quite a lot of time on the problem but, so far, have not found anything that would give any clear-cut answer to your question. I have discussed the matter with Dr. Warren and he suggests the possibility that leucopyrite ( $\text{Fe}_3\text{As}_4$ ) may be present. This mineral is practically indistinguishable from arsenopyrite, except by chemical analysis. He has made a preliminary survey of the polished sections of material similar to that sent to you for flotation testing and finds that there is a remarkable variation in the characteristics of the mineral specimens obtained from different parts of the property. The mineral described in the paper by Warren, Leckie-Ewing, and Adams came from one particular section of the mine.

I have assembled a number of available assays as follows :

#1 - Kelowna Exploration - This is the assay of the material you are using for flotation.

#2 - U.B.C. No. 5 - This is the sample of the mixture we used for flotation tests. All of this ore was used to make concentrates and we have only a few grams of sample left.

#3 - Williams Composite - Williams' analysis of the sample submitted by Mr. Douglass. Similar to U.B.C. No. 5.

#4 - Warren - This is the average analysis of the ore, (about 20 lbs) from which the specimens noted by Warren, et al were selected.

#5 - Gem Crude - This is the average analysis of a 100-lb lot of high-grade ore obtained by R.R. Taylor in 1940 and used for experimental purposes at U.B.C. This is the ore we are using now to make calcine, sinter, etc.

Ore No.	Co	Fe	As	S	Insol.	CaO
#1- Kel.Exp.	2.0	17.7	36.4	6.02	29.4	-
#2-UBC#5	2.3	20.6	26.5	6.9	26.2	1.6
#3-Williams	2.49	20.46	27.17	6.87	-	-
#4-Warren	7.3	22.1	39.0	14.1	11.4	-
#5-GemCrude	5.46	22.0	36.1	11.1	13.7	1.2

Adding Co, Fe, As, and S to get % sulphides we find:

#1	- 62.1 %
#2	- 57.3
#3	- 55.0
#4	- 82.5
#5	- 74.7

Assuming that all the S, all the Co, and sufficient Fe and As are present as Danaite we get:

No.	S	As	CoFe	Residual	
				Fe	As
#1	6.02	14.1	10.7	9.0	22.3
#2	6.9	16.2	12.3	10.6	10.3
#3	6.87	16.1	12.2	10.75	11.1
#4	14.1	33.0	25.1	4.3	6.0
#5	11.1	26.0	19.8	7.7	10.1

If leucopyrite or lollingite are present there is no way of determining the relative amounts of each present by analysis. The most that can be said is that there are two fairly important differences between the ore represented by analysis #1 and that of #2-#3.

1. There is a larger percentage of sulphides in No.1
2. There is a much larger relative amount of some high-arsenic mineral in #1 than in #2 or #3. This is noted in the column headed 'residual Fe-As'. #4 and #5 are similar, and probably come from the same section.

### Flotation

On the following page are some of the flotation results. It was notable in flotation that the cobalt mineral was very slow to float - we used 'everything but the kitchen sink' - soda ash, pine oil, aerofloat, Barret #4, and either K ethyl xanthate or Na ethyl xanthate. Also added copper sulphate but, of course, had to add it judiciously or it completely killed the flotation.

Test No. 9.

Product	Wt	As	Insol	CaO
Conct.	510	49.3	4.5	nil
Tail	460	4.4	51.2	5.1

Test No. 10.

Conct.	485	53.1	5.6	nil
Tail	500	2.1	55.0	6.5

Test No. 11.

Product	Wt.	Au oz	Co %	Recovery Au %	Co %
Conct #1	955	2.30	4.28	93.0	90.3
" #2	55	1.84	4.98	4.3	6.0
Tail	980	.06	.17	2.7	3.7
Calc. head	1990	1.19	2.27	100.0	100.0

The above results are all that we have worked out - we did not run cobalts on other concentrates but assumed from the weights that they were similar.

The high As-content of the concentrates (49-53%) indicates that the minerals present must include other than danaite as pure danaite contains only 41-47% As

As a further point of interest we treated some of the sample U.B.C.No. 5 by stirring it in cold, 1:10 HCl for 20 minutes. Soluble Fe was 2.2 % probably from some oxidized iron in the sample.

That is all the information we have available at present and I am not sure that it points any way out of the difficulty. Dr Warren is going ahead with polished section determination of the minerals present in the various parcels of ore shipped to you for flotation tests - this may give the answer.

In the meantime, we are going ahead with the preparation of various products from Gem Crude (#5 sample). We have about 3 lbs of straight calcine, and about 10 lbs of sinter and will go on to produce speiss. We will also make more calcine and will cyanide a portion of the calcine.

There will thus be four products :

- (1) Calcine,
- (2) Cyanided calcine tails,
- (3) Sinter,
- (4) Speiss.

The hours are very much restricted at the University now as the buildings must be closed at 4 p.m. This and other activities connected with blackout operations have upset the routine. However, I feel that now, more than ever, cobalt is going to be needed so we are doing all we can to get the information for you.

Kindest regards,