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MINERALOGICAL INVESTIGATION OF REFRACTORY ORE FROM DENISON MINES LIMITED

by

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EXTRACTION METALLURGY DIVISION

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SUMMARY

Microscopic examination shows that brannerite is the most abundant uranium-bearing mineral in the head samples as well as in the leach tailings. From the presence, in the tailings, of radioactive grains with exposed brannerite surfaces it is concluded that inadequate leaching conditions, rather than unusually refractory uranium minerals, probably account for the low extraction results.

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INTRODUCTION

In a letter dated January 5, 1962, Mr. A.B. Black, Metallurgist, Denison Mines Limited, requested that a microscopic study be made of two refractory ore samples which had yielded poor extraction results in laboratory leach tests carried out at the mine. Two head samples and two leach tailing samples, designated 753H, 756H, 753T and 756T respectively, were received on January 9 and were assigned our Reference No. 1/62-2.

The leach tailing samples were sized and superpanned and a detailed microscopic examination was made of various size fractions and gravity concentrates in an attempt to determine the cause of the reported low extractions.

The present report gives the results of the mineralogical investigation, together with the results of some related tests and analyses (see Appendixes A and B).

RESULTS OF INVESTIGATION

1. Sample Analysis

Chemical analysis of the submitted head samples and leach tailing samples gave the results shown in Table 1.

TABLE 1

Analysis of Submitted Samples

	Sample No.			
	753H	753T	756H	756T
U_3O_8	0.11 %	0.045 %	0.17 %	0.075 %
Probable secondary U_3O_8*	0.04	0.01	0.025	0.04
Fe	3.55		3.50	
S	3.16		3.25	
CO_2 (comb)	0.3-0.9**		1.6-4.3***	
CO_2 (evol)	0.02		0.15	
As	< 0.01		0.015	
P_2O_5	0.14		0.12	
Ti	0.28		0.20	
ThO_2	0.021		0.025	
Al	2.74		2.39	

* % U_3O_8 soluble in hot carbonate solution
** Range covered by 4 analyses
*** Range covered by 3 analyses

For comparison, composite mill feed and leach tailing samples for December 1961 were analysed and gave the following results:

	Composite Head	Composite Leach Tailing
U_3O_8	0.14 %	0.008 %
Probable secondary U_3O_8	0.037 %	0.0005 %

It is obvious from the analyses that while the present head samples contain approximately the same proportions of uranium and of secondary U_3O_8 as the December composite, the leach tailings contain considerably more uranium than that resulting from normal ore. Also, the December tailing sample contains a much smaller proportion of readily soluble uranium, as indicated by the carbonate dissolution test. The analyses also show that the secondary uranium content of sample 756T is higher than that of the head sample. This apparent anomaly indicates either poor washing of the leach residue or the presence of re-precipitated uranium.

2. Contaminants

Examination of the submitted head samples under a stereoscopic microscope showed that they contain small irregular fragments of a black, elastic material. This material has a specific gravity < 1.6 and when heated it emits an odour similar to that of burning rubber. It is estimated that this contaminant comprises approximately 1% by weight of sample 756H and a smaller proportion of sample 753H. In the tailing samples a similar material was observed. The fragments of this material in the tailings differ from those in the heads, however, in that they are slightly harder and less elastic. The range in the results obtained in the chemical determination of CO_2 by the combustion method is believed to be due to sampling difficulties caused by the presence of this rubber-like contaminant.

A small proportion of iron or steel contamination is also present in the head samples. Approximately 0.5% by weight of iron plus pyrrhotite is present in 753H. The iron is probably introduced during grinding of the ore.

3. Size Analysis

The tailing samples were screened and infrasized. Sample 753T was dry-screened whereas sample 756T was wet- and dry-screened so as to eliminate slime-sized particles from the coarser fractions.

TABLE 2

Size Analysis of Tailings

Size Fraction	753T	756T
- 48 + 65 mesh	4.2 % wt	3.5 % wt
- 65 + 100 "	7.9	4.6
-100 + 150 "	5.3	10.5
-150 + 200 "	12.0	12.4
-200 mesh + 56 microns	6.2	4.1
- 56 + 40 "	17.3	16.6
- 40 + 28 "	18.0	18.9
- 28 + 20 "	2.7	2.0
- 20 + 14 "	4.9	5.1
- 14 + 10 "	4.9	5.2
- 10 "	12.3	11.9
Loss	4.3	5.2
	100.00	100.00

The coarser size fractions of 753T, especially those coarser than 200 mesh, contain a considerable proportion of agglomerated fines. The size analysis of this sample is therefore not truly representative.

4. Uranium Distribution

The sized fractions of sample 756T were analysed for uranium. The uranium distribution is shown in Table 3.

TABLE 3

Uranium Distribution in Sample 756T

Size Fraction	Weight* (%)	U_3O_8 (%)	U_3O_8 Distn (%)
- 48 + 65 mesh	3.7	0.033	1.6
- 65 + 100 "	4.8	0.048	3.0
-100 + 150 "	11.1	0.057	8.2
-150 + 200 "	13.1	0.064	8.3
-200 mesh + 56 microns	4.3	0.068	3.8
- 56 + 40 "	17.5	0.052	11.8
- 40 + 28 "	20.0	0.045	11.7
- 28 + 20 "	2.2	0.14	4.0
- 20 + 14 "	5.4	0.095	6.6
- 14 + 10 "	5.4	0.10	7.0
- 10 "	12.5	0.21	34.0
Total	100.0		100.0

*Corrected for dusting loss in sizing

It is seen from the above results that there is a slight increase in uranium content in the finer-sized fractions. The minus 200 mesh fractions comprise 67.3% by weight of the tailings and contain 78.9% of the uranium. Particularly noteworthy is the concentration of uranium in the minus 10 micron fraction.

5. Microscopic Study

Polished sections of various size fractions were prepared for microscopic examination to determine the nature of occurrence of the uranium minerals in the head samples and in leach tailings. Alpha autoradiographs were used to locate radioactive grains in the polished sections (see Figure 2), and the identities of the grains were confirmed by means of X-ray powder diffraction patterns. The following is a summary of the results of the microscopic study.

Sized fractions of sample 753H consist mainly of grains of non-metallic, siliceous, gangue minerals. A minor portion of sulphide minerals is present and a smaller proportion of grey oxide minerals.

The gangue minerals are mainly quartz and mica (sericite and muscovite). Pyrite is the predominant sulphide mineral in the ore but minor amounts of chalcopyrite and pyrrhotite are also present. The grey oxide minerals include rutile, anatase, and the radioactive minerals, as well as trace amounts of ilmenite.

The major radioactive mineral in the head sample is brannerite. It is usually very fine-grained and occurs intimately intergrown with siliceous gangue minerals and with the titanium oxide minerals, rutile and anatase (Figure 1). A small proportion of the radioactive grains consists of fairly pure brannerite. In addition to brannerite, trace amounts of other radioactive minerals were noted. These include uraninite, monazite, and a mineral believed to be uranothorite.

Figure 1. Radioactive grain in sample 753H consisting of an intergrowth of brannerite (grey), rutile (white) and siliceous gangue mineral (black). X120

Microscopic examination of various Superpanner concentrates of leach tailing sample 753T shows that the most abundant mineral constituents are pyrite, rutile and anatase. Subordinate amounts of brannerite are present and minor amounts of siliceous gangue minerals (Figure 2A). All of the radioactive grains appear to contain brannerite which is usually intergrown with varying proportions of rutile, anatase and siliceous gangue minerals, and at times, also, some fine-grained galena (Figure 3). Only rarely do the radioactive grains consist of pure brannerite, however brannerite is often exposed at the grain edges (Figure 4).

In a Superpanner tip of tailing sample 756T (plus 100 mesh fraction) brannerite occurs mainly in intergrowths with titanium oxide minerals and gangue. A few grains of almost pure brannerite were

Figure 2. Polished section, A, and alpha autoradiograph, B, of tailing sample 753T, showing brannerite-bearing grains(br). Grains of titanium oxide minerals (TiO), pyrite (py), siliceous gangue mineral (sil) and pyrrhotite (pyrr) are also present in the section. X110.

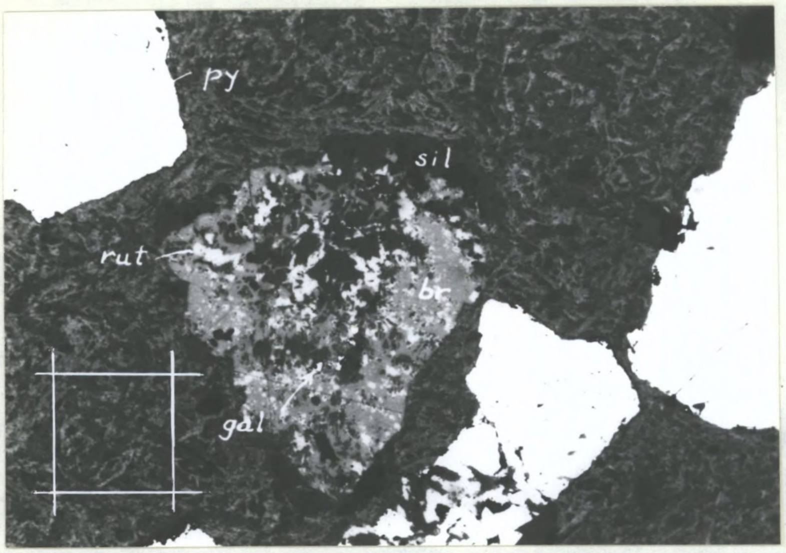

Figure 3. Radioactive grain in tailing sample 753T containing brannerite (br), rutile (rut), siliceous gangue mineral (sil) and fine inclusions of galena (gal). Grains of pyrite (py) are also present in the section. X208.

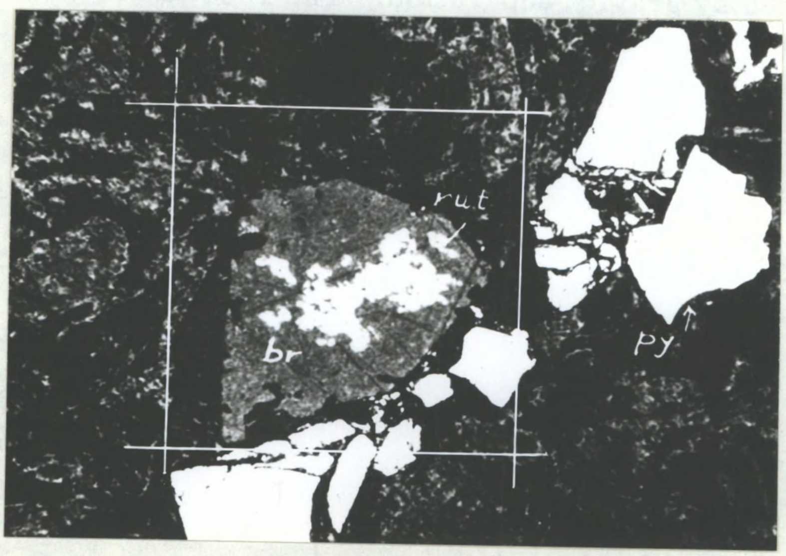

Figure 4. Radioactive grain in tailing sample 753T containing irregular mass of rutile (rut) enclosed in brannerite (br). Grains of pyrite (py) are also present in the section. X615.

observed, but some grains consist largely of rutile with only small particles or thin seams of brannerite. One weakly radioactive grain of zircon was noted and several grains of uraninite. The uraninite appears to be completely enclosed by gangue mineral.

Superpanner fractions of tailing sample 756T (plus 40 micron size) contain brannerite as the major uranium-bearing mineral. A very small proportion of a strongly radioactive mineral, believed to be uraninite, is also present.

6. Gravity Separation Test

Since the uranium in the leach tailing samples occurs mainly in intergrowths of heavy minerals (brannerite, rutile, anatase), a Superpanner test was carried out to confirm that the uranium could be concentrated by gravity separation. The results are shown in Table 4.

TABLE 4

Superpanner Separation, Sample 756T, - 20 + 14 microns

Fraction	Weight (%)	U_3O_8 (%)	Content	U_3O_8 Distn (%)
Tip	6	0.7*	0.044	46.3
Tailing	94	0.054	0.051	53.7
	100		0.095	100.0

*by difference

This test shows that 46.3% of the uranium has been concentrated in 6% of the weight, by gravity separation.

DISCUSSION AND CONCLUSIONS

Most of the radioactivity in the leach tailing sample is due to brannerite. The occurrence of the brannerite in the tailings appears to be essentially similar to that observed in the head sample: it is usually intergrown with siliceous gangue and titanium oxide minerals. But the brannerite is often exposed at the surface of the grains and thus should be amenable to acid dissolution. Since this brannerite has not been completely dissolved it can be concluded that either the leaching solution was not of sufficient strength or that some brannerite is of a very refractory nature. Since an appreciable proportion of the residual uranium (especially in sample 756T) appears to be "secondary" or re-precipitated it might be assumed that the normal intensive acid leach conditions were not maintained and that this is the cause of the lower extraction, rather than the brannerite being more refractory than usual. Indeed, leaching tests on sample 756H (see Appendix A) show that excellent extraction is achieved when sufficient free acid is available.

If unusually high proportions of acid-consuming minerals are present in the ore, inadequate acid may be available, under standard leaching conditions, for efficient leaching of the uranium minerals. The potential acid-consuming minerals in Elliot Lake ores, in addition to the uranium minerals, are chlorite, pyrrhotite and carbonates. Chlorite was not identified in the present samples and the proportion of pyrrhotite as compared to that in normal mill-feed is not known. The carbonate content in both samples is very low. However, two contaminants present in the head samples, namely tramp iron and the rubber-like material, probably have an adverse effect on leaching efficiency. Tests carried out by V.F. Harrison of the Hydrometallurgy Subdivision (see Appendix A) indicate that the "rubber" contaminant consumes 19 lb sulphuric acid per ton of ore. Thus, the presence of this contaminant along with even slight increases of acid-consuming minerals may reduce the free acid content below the level desirable for optimum uranium extraction. Indeed, analysis of a leach liquor resulting from a test on refractory ore (see Appendix B) shows that the free acid content is approximately 1/4 that of normal ore and is thus substantially below the amount usually present in mill circuits.

ACKNOWLEDGMENTS

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APPENDIX A

In order to determine the effect of the rubber-like contaminant on the leaching solution and on extraction, comparative leaching tests were carried out by V. F. Harrison. One sample consisted of a plus 200 mesh fraction of sample 756H whereas the second sample consisted of the same size fraction from which the "rubber" had been floated off in in a heavy liquid of specific gravity 2. Both samples were leached for 48 hours at 60°C and pulp density of 6% solids. The results of the test were as follows:

Sample	Acid Consumption (lb/ ton of feed)	U_3O_8 Extraction (%)
Ore containing "rubber"	31	98.6
Ore without "rubber"	12	98.7

These tests indicate that the "rubber"-bearing sample consumed approximately 2 1/2 times as much sulphuric acid as the "rubber"-free sample but the "rubber" has no effect on uranium extraction under the leaching conditions employed.

APPENDIX B

Two samples of leach liquor from bench scale tests at the mill were submitted by Denison Mines Limited for analysis of the major constituents, and especially uranium, free acid, and ferric iron content. The results of the chemical analyses are as follows:

Sample Designation	pH	Assay (g/l)				
		U_3O_8	Free H_2SO_4	Fe^{+3}	Fe^{+2}	Al
NDC	0.35	2.19	61	2.34	12.8	7.2
S-633	0.90	1.71	16	3.04	14.8	8.9

NDC Normal disc filter cake
S-633 Refractory ore

Assuming similar leaching conditions in both tests, the above results show that the refractory ore has consumed considerably more acid than normal ore and the uranium extraction is correspondingly lower by approximately 20%.

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