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X-RAY DIFFRACTION ANALYSIS OF MINE AIRBORNE DUST— AN INTERFERENCE PROBLEM AT HEMLO GOLD MINES

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by

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ABSTRACT

A set of samples from a Hemlo gold mine showed evidence of interference in the routine analysis, in that there was no agreement in the quantities of quartz estimated using four diffraction lines. Examination of the diffraction patterns for the common minerals in the ore showed that strong interference could be expected on all four quartz lines. Analysis of powder samples prepared from the four types of ore in the mine showed strong interference on one to three of the quartz lines for each type of ore and on all four lines for the four types together. Thus, the X-ray diffraction analysis for quartz in this type of ore is uncertain and further work is required to quantify the errors.

KEYWORDS: Mine airborne dust; Quartz analysis; Analytical interference.

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INTRODUCTION

The quartz component of the airborne mineral dust is, in many underground hard rock mines, the major concern in control of the health hazard to miners lungs. The Elliot Lake Laboratory has developed an X-ray diffraction system for assessment of the quartz component in mine airborne dust. This system assesses the quartz on up to four diffraction lines and only accepts a result when any two diffraction lines give agreement within the limits of experimental error. This procedure has been successful on most samples from most mines.

The exceptions have been:

1. Heavy samples: the effect of differing absorbtion on the analysis of the four diffraction lines due to differences in path length at the four angles is enough to introduce differences in measured quartz greater than the allowed error term.
2. A copper mine: as with the present problem mine, a proportion of the samples did not produce agreement between any pair of lines. Because the mine was small, had a low quartz value, and had a limited life, no detailed investigation was made.
3. An iron mine: some samples presented results in which one pair of quartz diffraction lines indicated one quartz content while the other pair indicated twice as much. This was attributed to interference by the high content of iron bearing minerals, and could have been resolved by chemical leaching. However, the proportion of samples affected was low, leading to only a small possible error in the overall mean at any one sampling station and this anomaly was not studied further.

DESCRIPTION OF HEMLO GOLD ORE

The ore is a gold ore containing some other metals in recoverable quantities. There is a total of over seventy identified mineral species present in the ore (3). Only the major minerals are, however, likely to interfere in the quartz analysis. These are listed in Table 1. This Table shows the reported mineral concentration and the occurrence of diffraction lines close to the quartz peaks. The near peaks are divided into two classes, first, the non-resolvable peaks which add to the X-ray intensity, and secondly, the resolvable peaks which have a negative effect leading to underestimation of the quartz. The lines are reported in terms of their per cent relative intensity. The unbracketed results refer to the most intense line of that mineral, set to 100, and the bracketed values refer to the relative intensity to the appropriate quartz line set to 100. An approximate allowance has been made for the absolute intensities of the lines, and for the relative amounts of mineral and quartz in the ore. It is clear that substantial interference is to be expected and that not even the results from one of the diffraction lines can be relied upon.

Table 1—Interference with quartz analysis as expected from published literature.

Table entry is intensity of line relative to minerals strongest line. Table entry in brackets is intensity relative to nearest quartz line allowing for absolute intensity and mean ore composition.

Quartz line..		1	2	3	4
Miller indices..		1,0,0	1,0,0	1,1,2	2,1,1
Relative intensity of quartz %		35	100	17	15
'd'spacing Å		4.260	3.343	1.817	1.541
Mineral	Content in Ore %	Additive interference range-d spacing Å[1]			
		4.55–4.18	3.40–3.26	1.83–1.804	1.5—1.53
Quartz	15				
Muscovite Vanadian muscovite	5-20	20 20 (52)	100 90 (11)	1 NL[2] (0.1)	10 NL (7)
Biotite Phlogopite	0-25	NL 2 (1)	100 71 (10)	NL 1 (10)	80 60 (50)
Microcline Barian Microcl.[3]	65	70 – (50)	100 – (70)	10 – (5)	10 – (7)
Barite	0-20	30 (30)	NL	NL	15 (30)
Titanite	?[4]	NL	NL	NL	NL
Pyrite	5-25	NL	NL	NL	NL
		Subtractive interference(adjacent line) range[5]			
		4.44-4.409	3.47-3.19	1.843-1.79	1.56-1.52
Muscovite Vanadian muscovite	5-20	50 50 (70)	60 60 (6)	1 NL (0.1)	10 10 (7)
Biotite Phlogopite	0-25	NL 2 (1)	NL 100 (5)	NL NL	NL 15 (5)
Microcline Barian microcline	65	NL –	100 – (70)	50 – (50)	5 – (5)
Barite	0-20	30 (30)	NL	NL	15 (30)
Titanite	?[4]	NL	100 (1)[4]	10 (0.1)	20 (0.2)
Pyrite	5-25	NL	NL	NL	NL

Footnotes:

1. The range is approximate as it depends on both absolute and relative quantities of quartz and interferent present.
2. L no lines in region
3. No published data for barian microcline. It is assumed to be close to microcline
4. Content in ore not given. It is asumed to be 1% and interference to be negligible.
5. The subtractive region consists of the two portions between the central additive region and the two boundaries shown.

X-RAY ANALYSIS OF ORE SAMPLES

The mine supplied samples of four types of ore described as: muscovite rich, biotite rich, microcline rich and barite rich. These four types differ markedly in visual appearance and fracture properties.

Samples on filters suitable for X-ray diffraction analysis have been prepared by a wet crushing, suspension, partial settlement and filtration process previously described (1). These samples were assessed by X-ray diffraction in the quartz analysis regions using a wider than normal peak assessment program both before and after spiking with pure quartz dust.

The results are shown in Table 2. The entries are the intensity of the diffraction peak given as equivalent quartz mass in micrograms and the peak position given as a 'd' spacing. An estimate of quartz recovery is made by subtracting the before spiking intensity from the after spiking intensity.

TABLE 2 – Analysis of Ore Samples

	Quartz Miller indices		1,0,0		1,0,1		1,1,2		2,1,1		quartz[1] estimate	
		wght.	Int.	Posit.	Int.	Posit.	Int.	Posit.	Int.	Posit.		
	Ore Type	µg	µg[2]	Å	µg	Å	µg	Å	µg	Å	µg	%
	quartz			4.26		3.34		1.817		1.541		
	Ores as received											
1	Muscovite rich	1060	*548*	4.25	*633*	3.33	315	1.815	*950*	*1.538*	315	31
2	Biotite rich	910	1211	4.26	441	3.35	237	1.817	243	1.542	220	24
3	Microcline rich	1110	*552*	4.24	145	3.35	*659*	*1.809*	104	1.540	100	9
4	Barite rich	870	107	4.25	245	3.33	22	1.817	*763*	*1.534*	22	1
	after spiking with quartz	Q.wt.									Quartz recovery	
5	Muscovite rich		1290	4.23	1290	3.32	1220	1.813	1112	*1.536*		
	Difference 5-1	620	*742*		657		*905*		662		660	106
6	Biotite rich		883	4.26	1175	3.34	797	1.817	791	1.542		
	difference 6-2	540	*671*		*734*		560		548		554	103
7	Microcline rich		1255	4.26	854	3.34	591	1.817	833	1.542		
	Difference 7-2	620	708		709		*-68*		729		715	115
8	Barite rich		368	4.26	764	3.34	475	1.817	1290	1.542		
	difference 8-4	440	*261*		*519*		453		*527*		453	103

Footnotes :

1. The quartz composition is subject to error due to incomplete grinding.
2. The X-ray intensity is given as the equivalent mass of quartz for each line
3. Italics signify that either the estimated quartz mass is subject to error or that the line position is off the quartz peak.

The significant points in Table 2 are that:

1. One diffraction line in each of the muscovite rich, microcline rich and barite rich ore samples is displaced significantly such that the major contribution must be non-quartz.
2. The quartz recovery on spiking with quartz is not satisfactory on 8 out of the 16 peaks examined showing that the interference modifies the assessment of the area under the peak. This is carried out by measuring the total area in the peak region and subtracting a linear background determined at either side of the peak. The background region is determined as that part without a significant rise above the slope determined in both directions towards and away from the 'quartz' peak.
3. The determination of 24 and 9% quartz in the biotite and microcline rich ores, respectively, seems satisfactory. The other two ores do not get a pair of lines to agree and the estimates of 31 and 1% are not confirmed.
4. There is not even one of the four quartz line that could be considered satisfactory for analysis of all the four ore types.

ANALYSIS OF FIELD SAMPLES

Table 3 shows the analysis of the airborne dust samples collected in the mine to date with significant X-ray diffraction indications of quartz. Only 3 peaks on 2 samples show a possibly significant displacement indicating that they have an obvious significant non-quartz component.

On 5 samples no agreement was found between the estimates on any pair of lines. On 3 samples two results were found differing by a factor of 1.5 to 2. Each result was supported by a pair of lines. The best estimate (an educated guess) differs from the agreed pair of lines, computer estimate, on 15 out of 21 samples shown, and is on average 10% lower.

DISCUSSION

The work carried out here demonstrates that interference is a significant problem in the X-ray diffraction analysis of quartz in the airborne dust samples collected in the Hemlo gold mines. It is believed that the existing system of X-ray diffraction analysis and interpretation is not reliable for this ore. Errors could be as great as 50%.

Table 3 – Quartz analysis of airborne dust samples collected in the Hemlo Mine.

X-ray Diffraction intensity given as equivalent quartz weight in μg

Samp. No.	Quartz line (Miller indices) 1,0,0	1,0,1	1,1,2	2,1,1	Computer Estimate	Educated Guess
1	NA[1]	152	89	152	152	89
2	317	159	Int.	303	310	159
3	69	57	24	52	55	24
4	257	168	205	279	260	200
5	244	166	168	244	210	167
6	678	466	290	710	fails	290
7	52	62	51	69	58	51
8	274	179	136	188	180	136
9	25	43	10	46	44	10-20
10	13	22	10	18	16	–
11	Int[d]	Int.	Int[d]	122	fails	?
12	Int.	14	Int.	14	fails	14
13	90[d]	23	Int.	21	22	23
14	177	64	76	95	70	70
15	14	13	8	18	13	10
16	119	57	58	10	84	57
17	Int.	Int.	Int.	50	fails	?
18	42	16	0	7	8	0
19	61	72	45	55	58	45
20	84	84	49	91	86	59
21	Int.	Int.	27	173	fails	27
22	35	51	33	35	34	34

Footnotes:

1. NA= not analysed.
2. superscript d refers to displaced peak position.
3. int.= Interference by close peak.

RECOMMENDATIONS

1. It would be of great interest to examine the specific varieties of the major minerals present in the ores and identify the major interferents in more detail. For this it would be useful to separate small quantities, about 0.1 gm, of each mineral variety for a more detailed examination by X-ray diffraction.

2. Analysis of samples of the ore types by other means could serve to verify, or correct, the X-ray diffraction analysis. The same samples would of course also have to be re-analyzed by XRD. This process could be used to determine an average correction value for XRD analysis for quartz but would leave substantial uncertainty in the values for individual samples because of the variations in proportions of the interfering minerals and quartz.

REFERENCES

1. **Knight, G.** Mine dust sampling system - CAMPEDS *CANMET Report 78-7, Energy, Mines and Resources Canada* April (1978).
2. **Knight, G.** CAMPEDS - A dust sampling and assessment system for use in hard rock mines *Ann. Occup. Hyg.* 30(2)139-151, (1986).
3. **The mine** Private Communication (1986)