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*QUANTITATIVE X-RAY DETERMINATION OF  
QUARTZ AND PYRITE IN DUSTS*

H. MACHACEK  
MINERAL SCIENCES DIVISION  
JANUARY 1970

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QUANTITATIVE X-RAY DETERMINATION OF QUARTZ AND PYRITE  
IN DUSTS

by

H. Machacek\*

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ABSTRACT

This investigation concerns the quantitative direct X-ray determination of quartz and pyrite in airborne mine dusts. A new technique developed by Leroux and Powers has been successfully applied and some new details have been found. The method provides reproducible analyses of quartz and pyrite in dust samples. When the calibration curves of the pure components have been made and certain sources of error are kept within appropriate limits, quartz and pyrite can be determined down to  $30 \mu\text{g}/\text{cm}^2$  when deposited on appropriate filters.

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\*N. R. C. Postdoctorate Fellow assigned to the Crystal Structure Group, Mineral Sciences Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

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DÉTERMINATION RADIOLOGIQUE DE LA QUANTITÉ DE  
QUARTZ ET DE PYRITE DANS LES POUSSIÈRES

par

H. Machacek\*

RÉSUMÉ

Cette étude a trait à la détermination radiologique directe des quantités de quartz et de pyrite contenues dans les poussières atmosphériques des mines. Une nouvelle technique mise au point par Leroux et Powers a été appliquée avec succès et quelques nouveaux détails ont été décelés. La méthode en question permet d'effectuer des analyses contrôlées du quartz et de la pyrite dans des échantillons de poussière. Lorsque les courbes de calibration des composants purs ont été tracées et que certaines sources d'erreurs sont maintenues dans des limites convenables, il est possible de mesurer les quantités de quartz et de pyrite avec une précision de  $30\mu\text{g}/\text{cm}^2$  lorsqu'ils sont déposés sur des filtres appropriés.

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\* Boursier d'études post-doctorales du CNR affecté au Groupe de la structure cristalline, Division des sciences minérales, Direction des mines, ministère de l'Énergie, des Mines et des Ressources, Ottawa, Canada.

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## INTRODUCTION

The suppression of dust during mechanical mining of ore-bearing rock represents part of an extensive research program being carried out by the Mining Research Division of the Mines Branch in the Elliot Lake area. The composition of airborne dust is thought to be an important criterion for investigating the parameters influencing rock breaking and crushing processes. The use of quantitative X-ray analysis, as a means of estimating very small amounts of quartz and pyrite in dust samples, is the subject of this report.

## THEORETICAL BACKGROUND

A need often arises to analyse solid materials for their components. One of the best methods for doing this is X-ray analysis, even though a number of difficulties occur in applying the X-ray method to the quantitative determination of solids (1). In practice, quantitative X-ray analysis is performed with a sample of approximately 0.5 g, which is easily put into a sample holder. In this case we can speak about the finite thickness of the sample, and the analysis of samples of "finite thickness" has been described in many papers, e.g. (2) to (14) and references therein. Research in the field of the various causes of air pollution has recently introduced a requirement for quantitative X-ray analysis of samples weighing a few milligrams or a fraction of a milligram. This problem has been investigated for many years by Leroux (15), who has developed a method which seems to be the best one available at the present time. The subsequent presentation will show the basic ideas and mathematical expressions of the quantitative X-ray analysis of thin-layer samples deposited on special silver membrane filters. Leroux and Mahmud (20) derived an equation which took into account the geometric shape of the irradiation of the sample volume -- treated as a triangular prism of infinite thickness.

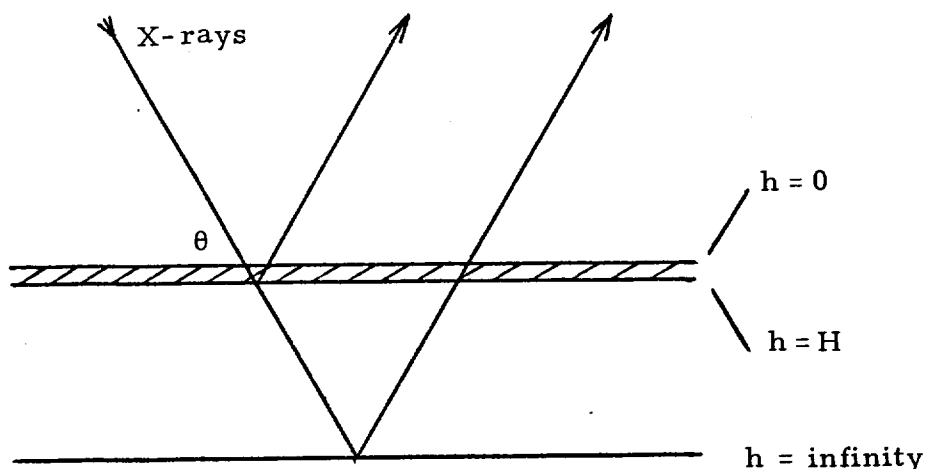


Figure 1. Irradiation of Thin Layers.

From Figure 1 it can be seen that, in the case of a thin layer, the shape of the irradiated volume of the sample can be approximated by a rectangular prism.

The energy flux per second in the diffracted beam is given by the following equation (see Ref. 16):

$$dI_i = KI_0 f_i A \exp(-2\mu_s^* \rho_s \operatorname{cosec} \theta \cdot h) dh, \quad \dots \text{(Eq. 1)}$$

where

$K$  = constant of proportionality,

$dI_i$  = intensity of X-rays diffracted from volume element  $dV = Adh$  by component  $i$  of the mixture [ergs/sec],

$I_0$  = intensity diffracted by unit volume of pure  $i$ -th component at the angle  $2\theta$  to the primary beam [ergs/sec],

$f_i$  = volume fraction occupied by  $i$ -th component,

$A$  = irradiated sample area [ $\text{cm}^2/\text{g}$ ],

$\mu_s$  = mass absorption coefficient (mac) of the sample [ $\text{cm}^2/\text{g}$ ], and

$\rho_s$  = density of the sample [ $\text{g}/\text{cm}^3$ ].



We can replace  $f_i$  in Eq. 1 by

$$f_i = \frac{x_i \rho_s}{\rho_i}, \quad \dots \text{(Eq. 2)}$$

where  $x_i$  = weight fraction of i-th component, and

$\rho_i$  = density of i-th component [ $\text{g}/\text{cm}^3$ ],

and thus we obtain the expression to be integrated within limits

$h = 0$  and  $h = H$ :

$$dI_i = KI_o A \frac{x_i \rho_s}{\rho_i} \exp(-2\mu_s^* \rho_s h \text{cosec } \theta) dh \quad \dots \text{(Eq. 3)}$$

The integration of 3 gives

$$I_i = KI_o A \frac{x_i \rho_s}{\rho_i} \cdot \frac{1 - \exp(-2\mu_s^* \rho_s H \text{cosec } \theta)}{2\mu_s^* \rho_s \text{cosec } \theta} \quad \dots \text{(Eq. 4)}$$

$$\text{Substituting } \rho_s = \frac{\bar{W}_s}{A \cdot H} \quad \dots \text{(Eq. 5)}$$

and noting that

$$\bar{W}_i = x_i \bar{W}_s, \quad \dots \text{(Eq. 6)}$$

where  $\bar{W}_s$  = total weight of the sample [g],

$\bar{W}_i$  = total weight of the i-th component [g], and

H = thickness of the deposited sample [cm],

we obtain

$$I_i = KI_o \frac{\bar{W}_i}{\rho_i} \cdot \frac{1 - \exp(-2\mu_s^* W_s \text{cosec } \theta)}{2\mu_s^* W_s \text{cosec } \theta}, \quad \dots \text{(Eq. 7)}$$

$$\text{where } W_s = \frac{\bar{W}_s}{A} \quad (\text{g}/\text{cm}^2). \quad \dots \text{(Eq. 8)}$$

It is worth noting the fundamental difference between  $W_s$  and  $\bar{W}_s$  in Eq. 7, which has been introduced for the following reason: The average thickness of thin deposited layers used in this technique is less than  $5\mu\text{m}$ , which means that the absorption effect is so negligible that the thickness H need not be taken into consideration. A more useful quantity has been introduced and it can be called surface area weight concentration (SAWC), as given by Eq. 8.

A convenient unit used in practice is  $\mu\text{g}/\text{cm}^2$ .

Leroux and Powers (15) have introduced the following notation in relation to Eq. 7:

$$T = \exp(-2\mu_s^* W_s \operatorname{cosec}\theta) \quad \dots \text{(Eq. 9)}$$

$$-\ln T = 2\mu_s^* W_s \operatorname{cosec}\theta \quad \dots \text{(Eq. 10)}$$

Combining equations 7, 9 and 10, the final working formula for intensity evaluation of X-ray diffraction on thin layers of dust samples deposited on silver membranes can be written in this form:

$$\begin{aligned} I_i &= KI_o \frac{\bar{W}_i}{\rho_i} \left[ \frac{1 - T}{-\ln T} \right] \\ &= KI_o \frac{\bar{W}_i}{\rho_i} f(T) \quad \dots \text{(Eq. 11)} \end{aligned}$$

Equation 11 is the basic expression used in quantitative X-ray analysis of thin-layer samples whose weights per squared centimetre do not exceed 250  $\mu\text{g}$  for  $\text{MoK}_\alpha$  radiation.

A more convenient formula may be derived from Eq. 11. If we designate the diffracted intensity of pure i-th component  $(I_i)_o$ , the weight of pure i-th component  $(\bar{W}_i)_o$ , and the transmittance function as  $f(T_o)$ , we get

$$(I_i)_o = KI_o \frac{(\bar{W}_i)_o}{\rho_i} f(T_o) \quad \dots \text{(Eq. 12)}$$

Dividing equation 11 by equation 12 we obtain the more useful expression:

$$\frac{\bar{W}_i}{(\bar{W}_i)_o} = \frac{I_i}{(I_i)_o} \cdot \frac{f(T_o)}{f(T)} \quad \dots \text{(Eq. 13)}$$

From the relation of Eq. 7, it follows that when the values of  $W_s$  and  $\mu_s^*$  are less than certain critical values, which can be experimentally determined and quantitatively assessed from the equation 9, the ratio of  $f(T_o)/f(T)$  may be considered to be unity and we can assume a linear dependence between the SAWC and the corresponding diffracted intensity.

Therefore

$$\frac{\bar{W}_i}{(\bar{W}_i)_o} = C \frac{I_i}{(I_i)_o}, \quad \dots \text{ (Eq. 14)}$$

where the constant  $C$  has a value near unity (the value is given by statistical evaluation of experimental data), and in routine work it can be put equal to unity. A thorough discussion of the properties of the equation 14 is given by Leroux and Powers (15), and the reader is referred to this work for further details. The importance of the equation 14 is due to the fact that the rapid analysis for component  $i$  consists in measuring the intensities of an unknown sample and those of a standard pure component  $i$ , respectively. The comparison of measured intensities with the calibration graph yields the SAWC of the component  $i$ . In order to use this calibration graph it would be necessary to measure the intensity of a pure quartz sample for every analysis, in order to place the intensity at the time of measurement on the same scale as at the time the calibration graph was plotted. However, this may be done equally well with another material, and, because of the delicate nature of the deposits on the FLOTRONICS filters, an  $Al$  slab was chosen for this continual measurement and the intensity of the peak at  $2\theta = 17.42^\circ$  was so measured.

If  $(I_a/I_c)_{Al}$  is the ratio intensities of the  $Al$  peak at the time of the analysis and at the time of calibration respectively, equation 14 becomes

$$\bar{W}_i = I_i \left( \frac{(W_i)_o}{(I_i)_o} \right) \cdot \left( \frac{I_a}{I_c} \right)_{Al}, \quad \dots \text{ (Eq. 15)}$$

where  $(W_i)_o/(I_i)_o$  is the reciprocal of the slope of the calibration graph in  $\mu\text{g}/\text{cm}^2/\text{mm}$ .

## EXPERIMENTAL TECHNIQUE

### Sample Preparation

The standard samples of pure quartz and pure pyrite were deposited on silver membrane filters (FLOTRONICS metal membrane filters) by using the device and technique for thin dust coatings which have been designed by Leroux (17). Quartz and pyrite were ground to give particle sizes under  $3\ \mu\text{m}$ . Each of the FLOTRONICS filters deposited with quartz or pyrite was cut into 4 quadrants, which were afterwards glued and centred to rectangular glass slides (these were used as sample holders in the X-ray diffractometer). We used Glyptal cement, diluted in amylacetate, as a glueing agent.

### Diffraction Measurement

The instrument used was a PHILIPS X-ray diffractometer. The molybdenum X-ray tube was operated at 50 kV and 20 mA, and the diffractometer scanning speed was 0.25 degree per minute, with a receiving slit of 0.006 inch. The reflected intensities were measured with a scintillation counter, and recorded with a strip chart recorder. The strongest reflections for quartz and pyrite are located for  $\text{MoK}_\alpha$  radiation at  $2\theta$  angles of  $12.14^\circ$  ( $d = 3.34$ ) and  $15.10^\circ$  ( $d = 2.70$ ), respectively. All diffracted intensities were compared with the intensity diffracted by an aluminum slab.

### Choice of Molybdenum Radiation

Even though it is less commonly used in general, molybdenum radiation has proved to be superior to other radiations for this technique. Using  $\text{MoK}_\alpha$  radiation, the fluorescence effect which occurs with other radiations is negligible. The most important reason for employing this radiation, however, is that the mass absorption coefficients of the elements in the samples are considerably smaller than for other radiation. Low

absorption by the analysed materials is the main prerequisite for the successful application of equation 14. Table I shows how the mass absorption coefficients vary for the elements of the SiO<sub>2</sub>-FeS<sub>2</sub> system, for three commonly used radiations.

TABLE I

The Values (mac) of the Elements Forming the SiO<sub>2</sub>-FeS<sub>2</sub> System

Element	Mass Absorption Coefficient [cm <sup>2</sup> /g] <sup>18</sup>		
	MoK <sub>α</sub>	CuK <sub>α</sub>	CoK <sub>α</sub>
O	1.31	11.5	17.8
Si	6.44	60.6	93.3
S	9.55	89.1	136.0
Fe	38.50	308.0	52.8

The (mac) of a chemical compound or of a mixture of n components is given by:

$$\mu_C^* = \sum_{i=1}^n a_i \mu_i^* , \quad \dots \text{ (Eq. 16)}$$

where  $\mu_C^*$  = (mac) of the compound (or of the mixture),

$a_i$  = the weight fraction of the i-th pure component, and

$\mu_i^*$  = (mac) of the i-th pure component.

The values of (mac) for quartz and pyrite for various radiations are shown in Table II.

TABLE II

The Values (mac) of Quartz and Pyrite for Three Radiations

Mineral	Mass Absorption Coefficient [ $\text{cm}^2/\text{g}$ ]		
	MoK $_{\alpha}$	CuK $_{\alpha}$	CoK $_{\alpha}$
SiO $_2$	3.72	36.4	53.1
FeS $_2$	22.04	191.1	97.2

From this table the advantage of MoK $_{\alpha}$  radiation for the analysed system is quite evident.

### EXPERIMENTAL RESULTS

#### Pure Quartz Calibration Curve

Using Leroux's method (17) of preparing the fine dust deposits, a set of ten standard samples of quartz ranging from 0.70 to 4.5 mg was prepared (particle size under 3  $\mu\text{m}$ ). The diameter of FLOTRONICS filters is 47 mm, but that of the coated area is only 35 mm which corresponds to an area of 9.62  $\text{cm}^2$ . Each of the four quadrants of a FLOTRONICS filter was scanned 3 times, so that 12 measurements of each sample were made. The weights of the standard samples were determined with fairly high precision, so that only the intensity measurements (the intensities are given as the peak heights measured from the average background) displayed significant statistical scattering. The averaged values of measured intensities (I) are plotted against  $W_Q$  (SAWC) coordinates in Figure 2. The intensity of the aluminum reflection was found to be 94 mm for the particular X-ray diffractometer set-up, and it represents the mean value of 40 intensity measurements. The data from which Figure 2 was constructed are listed in Table III, and the slope and  $A \ell$  intensity are shown with Figure 2. The

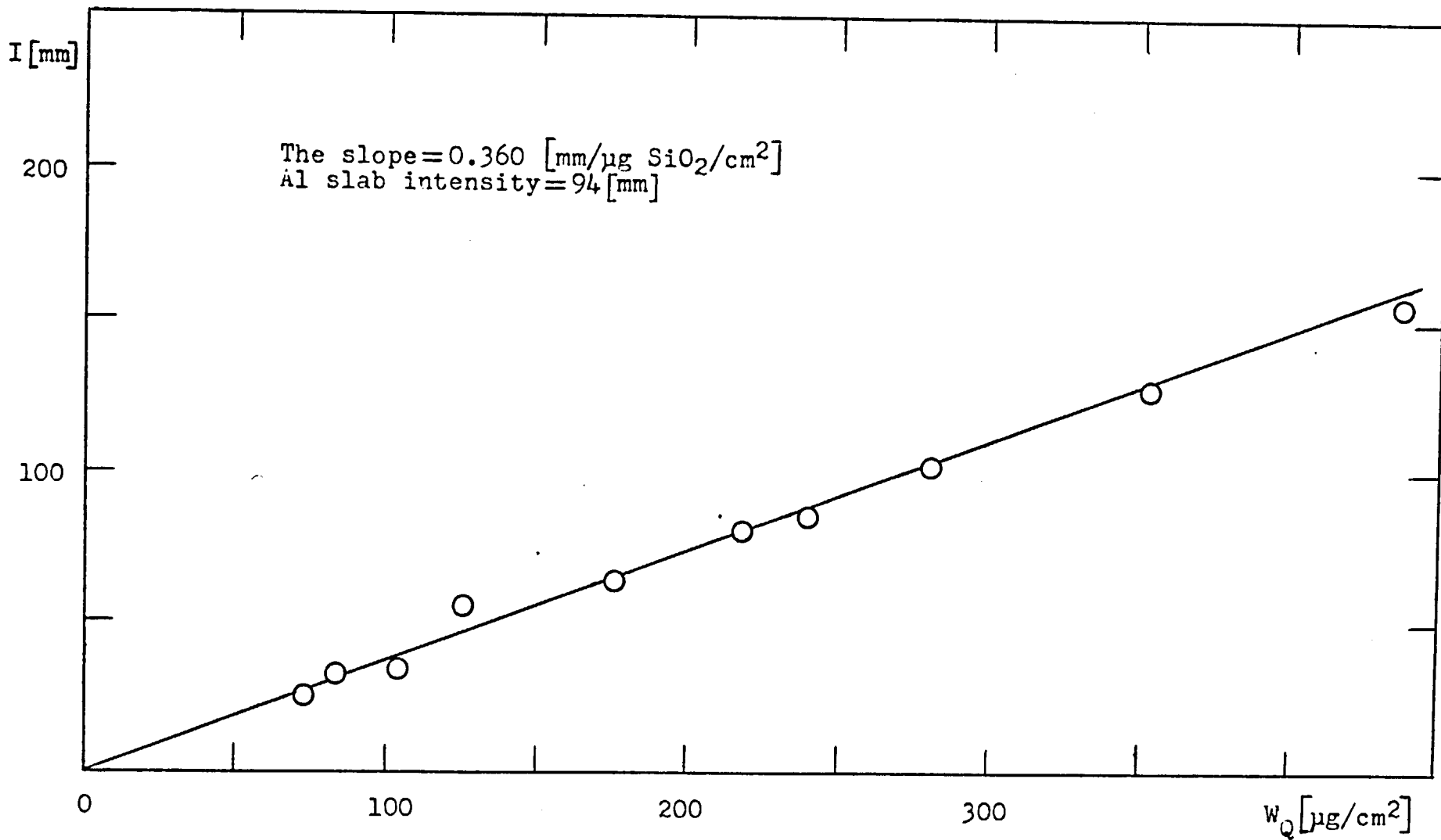


Figure 2. Intensity of Quartz vs Weight of Quartz on Filter.

square root of the estimate of the variance has yielded the values  $s_{y.x} = \pm 4.06$  ( $y$  for intensity  $I$  and  $x$  for SAWC  $W_Q$ ).

TABLE III

Intensity of Quartz vs Weight of Quartz on Filter

Sample No.	Weight of SiO <sub>2</sub> [ $\mu\text{g}/\text{cm}^2$ ]	Intensity [mm]
1	218	81
2	280	102
3	125	55
4	177	62
5	104	33
6	437	155
7	83	32
8	353	127
9	239	85
10	73	27

In general, quartz occurs in mixtures containing a number of other minerals. Provided that no superposition of reflections from different minerals takes place (e. g. micas or clay minerals), or that only a small amount of these minerals is present, the quartz content in the mixture is readily determined in weight per cent by the following formula:

$$\% \text{ Quartz} = \frac{\bar{W}_Q}{\bar{W}_M} \cdot 100, \quad \dots \text{ (Eq. 20)}$$

where  $\bar{W}_M$  = total weight of the multicomponent sample [ $\mu\text{g}/\text{cm}^2$ ]

Again, a low value of the (mac) of the mixture is necessary to keep within the linear range of the calibration graph, and preliminary qualitative calculations of this quantity should always be done to avoid this type of error.



Pure Pyrite Calibration Curve

The same procedure as for the quartz calibration curve determination was used to get the pyrite calibration curve. The plot of I vs  $W_P$  is shown in the Figure 3; however, in this case there is a much shorter linear range. This is due to the fact that the mass absorption coefficient of pyrite is approximately 6 times that of quartz. This fact is in good agreement with Leroux (19) who has found the linear range of I vs  $W_Q$  to extend up to  $1500 \mu\text{g SiO}_2/\text{cm}^2$  with  $\text{MoK}_\alpha$  radiation. The intensity of the aluminum reflection was  $I = 133 \text{ mm}$ , which is again within the linear portion of the curve. The data from which the Figure 3 was constructed are listed in Table IV.

TABLE IV

Intensity of Pyrite vs Weight of Pyrite on Filter

Sample No.	Weight of $\text{FeS}_2$ [ $\mu\text{g}/\text{cm}^2$ ]	Intensity [mm]
1	353	194
2	156	105
3	333	179
4	249	158
5	156	101
6	73	60
7	123	72

Regression analysis of the data in the Table IV supplied the square root of variance  $s_{y,x} = \pm 7.5$ . Even though the  $s_{y,x}$  for pyrite is nearly twice as large as that for quartz, the relative error of pyrite determination does not differ much from that of quartz, because the intensities are approximately twice as large.

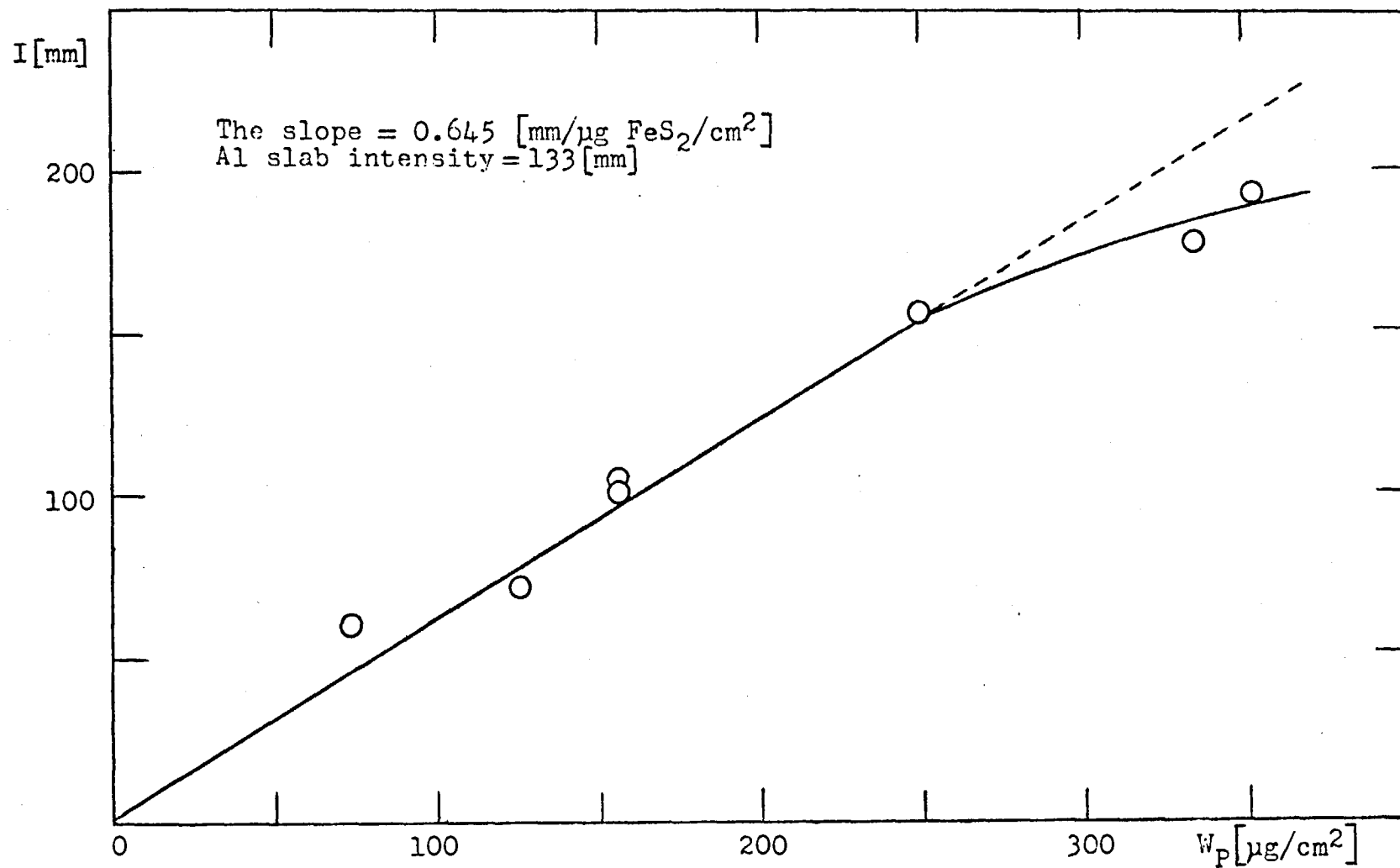


Figure 3. Intensity of Pyrite vs Weight of Pyrite on Filter.

## Quantitative Determination of Quartz and Pyrite in the NORDIC Ore Samples

From the mineralogical examination it is known that NORDIC ore contains four main components:

1. Quartz
2. Feldspars
3. Mica minerals
4. Pyrite

Preliminary qualitative X-ray analyses of NORDIC ore samples (on FLOTRONICS filters) which were used in breaking mechanism studies did not indicate the presence of mica minerals and, therefore, possible superposition of lines was not considered.

During the breaking mechanism tests the dust samples were taken by a dust-sampling instrument in the rock-breaking laboratory. As the criteria for dust collecting on FLOTRONICS filters by such sampling instruments are only just being tested, not all the analysed samples were found suitable for the X-ray microanalysis, and evenly distributed dust deposition outside a dust chamber, or without the use of a dust-coating device (Leroux, 17), remains a difficulty at present. The unevenness of the dust coating causes the results of the X-ray analyses to be unreliable and irreproducible. To obtain good results, one has to be very careful when collecting dust samples on the FLOTRONICS filters in the field.

Results of the analyses of two runs of dust samples collected during breaking tests are shown in Tables V and VI.

## Factors that Influence the Reliability of X-Ray Determination on the FLOTRONICS Filters

A number of other factors which influence the accuracy of X-ray determination of components deposited in thin layer samples, especially those which have been collected outside a laboratory, must be borne in mind, namely:

TABLE V

Quantitative X-Ray Determination of Quartz and Pyrite in NORDIC Ore Samples - Run A

Sample No.	Total Weight of the Sample [ $\mu\text{g}/\text{cm}^2$ ]	Determined Weight of $\text{SiO}_2$ [ $\mu\text{g}/\text{cm}^2$ ]	Weight % of $\text{SiO}_2$	Determined Weight of $\text{FeS}_2$ [ $\mu\text{g}/\text{cm}^2$ ]	Weight % of $\text{FeS}_2$
A-11	255	153	60.0	45	17.6
A-12	261	200	77.4	43	16.5
A-13	229	71	31.1	50	21.8
A-14	242	73	30.2	51	21.0
A-15	121	50	41.1	48	39.7
A-16	152	66	43.2	52	34.2
A-17	449	91	20.2	34	7.7
A-18	354	131	37.1	40	11.3
A-19	283	118	41.7	44	15.5
A-22	94	62	47.9	44	46.8

TABLE VI

Quantitative X-Ray Determination of Quartz and Pyrite in NORDIC Ore Samples - Run B

Sample No.	Total Weight of the Sample [ $\mu\text{g}/\text{cm}^2$ ]	Determined Weight of $\text{SiO}_2$ [ $\mu\text{g}/\text{cm}^2$ ]	Weight % of $\text{SiO}_2$	Determined Weight of $\text{FeS}_2$ [ $\mu\text{g}/\text{cm}^2$ ]	Weight % of $\text{FeS}_2$
B-11	224	146	64.2	58	25.9
B-12	224	137	61.2	51	22.8
B-13	184	124	67.6	43	23.3
B-14	168	120	75.5	39	23.2
B-16	120	84	70.0	34	28.3
B-18	240	123	51.3	36	15.0
B-19	171	127	74.0	28	16.7
B-22	95	57	59.5	14	14.7

1. Unpredictable size distribution of dust particles
2. Presence of interfering minerals
3. Weighing errors
4. Adsorption of water vapour from the air in the pores of the FLOTRONICS filters
5. Possibility of damaging the samples during handling and transportation

Some of these factors can be suppressed and their common effect kept to a minimum, when careful experimentation is carried out. In analysing the dust samples of Elliot Lake, no attention has been paid to the possible presence of mica and clay minerals because the preliminary qualitative work on NORDIC ore did not show any presence of these minerals. The above-mentioned factors should always be estimated before a particular application of this method is undertaken so that the possible error can be kept within reasonable limits.

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