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THE X-RAY FLUORESCENCE ANALYSIS
OF AQUEOUS ORE SLURRIES
PART 3: HEMATITE ORES

by

MRS. D. J. REED, J. L. DALTON AND A. H. GILLIESON

MINERAL SCIENCES DIVISION

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ABSTRACT

Fractions of hematite ores were analyzed as circulating slurries to determine relationships and factors for the on-stream analysis of such samples. Only iron and silicon were determined because the ore consisted largely of Fe_2O_3 and SiO_2. Radioisotopes were used to excite these elements and their characteristic radiations were measured by non-dispersive means.

The measured intensities were corrected by a backscatter factor which was based on the ratio of the scatter of the exciting radiations from the slurries to that from water. A further correction was introduced to correct for the difference in absorption of the characteristic and exciting radiations by the slurries. A regression line was established to convert counts from slurries of various densities to a 40% solids basis.

The iron content of the solids was determined with a standard error of less than 2%. The determination of silicon was not satisfactory at low concentrations due to the pronounced absorption of SiKα by all the components of the streams.

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INTRODUCTION

The second ore to be investigated for adaptability to on-stream analysis was a hematite for which only iron and silicon values were required. Because of the simplicity of this ore system, it was decided to use radio-isotope excitation and non-dispersive analysis which would greatly decrease the complexity and cost of instrumentation. By bringing the analyzer to the ore stream instead of the stream to the analyzer, plant installation would be simplified.

The counts obtained on various fractions of the ore and the derivation of analytical results therefrom are presented in this report. The ore fractions were supplied by the Iron Ore Company of Canada and were analyzed by the Analytical Chemistry Section of the Mineral Sciences Division.

EQUIPMENT

Two radioactive sources that emit essentially mono-energetic X-radiation were used to excite the samples. Fe-55, emitting MnKα, excited the silicon and Cd-109, emitting AgKα, excited the iron in the slurries. A portion of the manganese and silver radiations originating in the sources was scattered by the slurries to reach the counters. The characteristic radiations of silicon and iron arise in the slurries from the ore samples present.

FeKα and AgKα were detected using a xenon-filled proportional counter, while SiKα and MnKα were detected by a flow proportional counter, using neon as the counter gas with 3% isobutane as the quench gas. Neon was used in place of the customary argon because it does not produce a manganese escape peak to interfere with the silicon radiation.
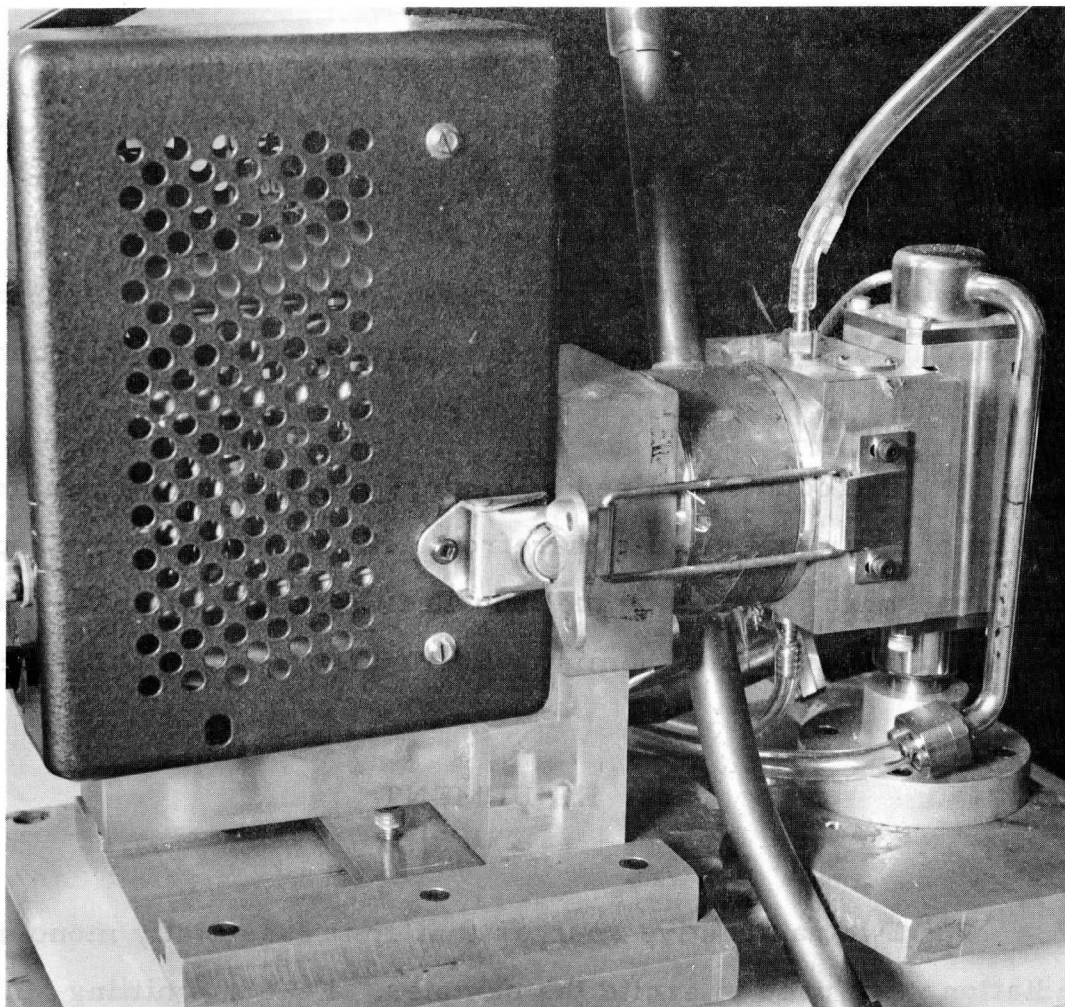
Figure 1. Slurry Cell with Spectrographic Chambers and Counters.

One face of a double-faced slurry sample cell was backed by the flow counter and the Fe-55 source which was offset at an angle of 45°. The other face was backed in a similar manner by the proportional counter and the Cd-109. This arrangement resulted in small circular chambers on each side of the slurry cell which were 1.5 inches in diameter and 1 inch deep. That between the flow counter and the cell was filled with helium because SiKa is strongly absorbed by air. The nature of the sample prohibited evacuation of the chamber. The cell windows were 50-gauge polyproplylene - Hercules B500. The slurry cell and spectrographic chambers were machined by members of the Technical Services Division of the Mines Branch. The arrangement of these components is shown in Figure 1.

The output of the counters was fed to standard Philips counting equipment and the radiations were separated by pulse-amplitude discrimination.

The slurry circuit used was similar to that used for the sulphide ores (1) but the receiving tank was enlarged.

EXPERIMENTAL PROCEDURE

After 3500 ml of water had been poured into the receiving tank, the pump was started to circulate water for about five minutes to remove the air bubbles from the sample cell. Then counts were taken on the circulating water. Successive portions of the sample were added and counts were taken after each addition. Six ten-second counts were taken for each radiation and then repeated after a two-minute interval.

The first portion of sample added weighed 875 g and resulted in a 20% solids stream. The next portion of 625 g increased the solids content to 30%. Further additions of 830 and 1170 g gave 40 and 50% slurries, respectively.

TABLE 1a

Counts for FeKα and AgKα Obtained in Series 1

Sample	% Fe$_2$O$_3$	Solids	FeKα	AgKα	B	BC cps	M	MC cps
Conc.	92.7	0	229	982	1.0000		1.000	
		20	714	620	0.6314	901	0.287	3711
		30	849	592	0.6028	1180	0.230	5895
		40	930	584	0.5947	1335	0.199	7629
		50	1004	572	0.5824	1495	0.178	9456
Conc. + Feed	86.6	0	229	990	1.0000		1.000	
		20	692	625	0.6313	867	0.303	3389
		30	806	604	0.6101	1092	0.245	5163
		40	890	590	0.5960	1264	0.212	6815
		50	963	587	0.5929	1395	0.190	8320
Feed	80.8	0	250	958	1.0000		1.000	
		20	636	625	0.6524	725	0.319	2806
		30	762	597	0.6232	979	0.260	4453
		40	833	576	0.6012	1136	0.225	5908
		50	890	568	0.5929	1251	0.203	7145
Feed rpt.		0	242	948	1.0000		1.000	
		20	633	614	0.6477	735	0.319	2822
		30	752	588	0.6202	970	0.260	4422
		40	838	567	0.5981	1159	0.225	5985
		50	902	562	0.5928	1280	0.203	7354
Conc. + Tails	60.0	0	233	982	1.0000		1.000	
		20	513	674	0.6863	514	0.442	1458
		30	620	632	0.6436	730	0.378	2315
		40	680	614	0.6353	855	0.342	2947
		50	726	606	0.6171	943	0.308	3587
Feed + Tails	54.2	0	235	980	1.0000		1.000	
		20	482	688	0.7020	452	0.458	1264
		30	577	644	0.6571	643	0.393	1999
		40	628	622	0.6347	754	0.354	2560
		50	664	613	0.6255	827	0.320	3082
Feed + Tails rpt.		0	245	942	1.0000		1.000	
		20	494	645	0.6827	476	0.458	1335
		30	577	615	0.6529	639	0.393	2004
		0	238	954	1.0000		1.000	
		40	631	593	0.6216	777	0.354	2630
		50	680	587	0.6153	867	0.320	3216
Tails	27.7	0	316	948	1.0000		1.000	
		20	416	746	0.7869	213	0.597	570
		30	451	703	0.7416	292	0.526	840
		40	498	667	0.7036	392	0.484	1146
		50	527	648	0.6835	455	0.437	1448
Tails rpt.		0	242	948	1.0000		1.000	
		20	352	720	0.7595	221	0.597	534
		30	396	678	0.7152	312	0.526	811
		40	444	642	0.6772	414	0.484	1113
		50	477	623	0.6572	484	0.437	1419

Low SiKα counts had been anticipated because of the low fluorescent yield of the element and of the strong absorption of the radiation by all the stream components. After the first counts taken for silicon were examined, it was decided to repeat the counts using a stronger isotope source. Thus, two series of samples were run about a year apart. For the second series, a new spectrographic chamber was required to house the new source which had different dimensions from the first. It was also necessary to have the new ore fractions analyzed chemically. The first Fe-55 source used had a flux of 15 and the second of 102 mCi. The same Cd-109, with an original flux of 3 mCi, was used for both series of counts. In Series 2, the 50% slurry was omitted to reduce sample consumption. In Series 1, 35 and 45% slurries were also counted and provided extra values for the determination of the factors used. However, for clarity they have been omitted from the tables of results because they served only to verify the validity of the factors and did not change them in any way.

RESULTS

Tables 1a and 1b present the FeKα and AgKα counts taken in Series 1 and 2 respectively, and the counts for SiKα and MnKα are given in Tables 2a and 2b. On the first day of Series 2, the silicon and manganese counts were so low that they have been omitted, although the corresponding iron counts are retained. The cause of the low counts has not been pinpointed, but it may have been poor counter response after a long period of disuse.

The scatter counts, especially MnKα, do not always decrease steadily with increasing solids content. The iron counts increase with slurry solids, but the silicon counts remain almost stationary in some cases and, in others, even decrease. For FeKα, it is possible to get net counts by subtracting the water count from that of the slurries; but, in the case of SiKα, such counts would be negative, except for the last three samples listed in Table 2b.

TABLE 1b

Counts for FeKα and AgKα Obtained in Series 2

Sample	% Fe_2O_3	Solids	FeKα	AgKα	B	BC cps	M	MC cps
Conc.	92.0	0	126	524	1.0000		1.000	
		20	469	282	0.5382	745	0.289	2889
		30	556	270	0.5153	953	0.232	4525
		40	616	258	0.4924	1125	0.200	6129
Conc. rpt.		0	138	554	1.0000		1.000	
		20	479	307	0.5542	726	0.289	2853
		30	576	292	0.5271	955	0.232	4572
		40	649	281	0.5072	1142	0.200	6260
Feed	82.5	0	130	515	1.0000		1.000	
		20	418	298	0.5786	592	0.315	1885
		30	506	278	0.5398	807	0.253	3165
		40	572	265	0.5146	981	0.222	4877
Feed rpt.		0	143	555	1.0000		1.000	
		20	413	332	0.5982	547	0.314	2056
		30	507	307	0.5532	773	0.255	3451
		40	573	289	0.5207	957	0.222	4814
Conc. + Tails	59.9	0	138	556	1.0000		1.000	
		20	355	335	0.6025	451	0.443	1192
		30	424	306	0.5504	632	0.379	1895
		40	486	289	0.5198	797	0.342	2596
Tails	27.7	0	141	560	1.0000		1.000	
		20	231	381	0.6982	190	0.597	413
		30	251	372	0.6643	237	0.526	577
		40 /	278	357	0.6375	295	0.484	760
SiO_2	0.0	0	128	526	1.0000		1.000	
		20	113	458	0.8707	2	1.049	-4
		30	112	426	0.8099	10	1.065	2
		40	106	406	0.7719	9	1.078	-1

TABLE 2a

Counts for SiKα and MnKα Obtained in Series 1

Sample	% SiO$_2$	Solids	SiKα	MnKα	B	BC cps	M	MC cps
Conc.	2.2	0	167	750	1.0000		1.000	
		20	126	536	0.7147	9	0.959	17
		30	133	516	0.6880	26	0.945	38
		40	128	514	0.6853	20	0.933	33
		50	120	512	0.6827	9	0.923	23
Conc. + Feed	8.6	0	165	750	1.0000		1.000	
		20	128	534	0.7120	14	0.938	28
		30	129	520	0.6933	21	0.918	39
		40	122	520	0.6933	11	0.901	30
		50	124	512	0.6827	17	0.886	40
Feed	15.0	0	152	768	1.0000		1.000	
		20	118	552	0.7188	12	0.918	27
		30	120	542	0.7057	18	0.891	39
		40	121	528	0.6875	24	0.869	50
		50	130	511	0.6654	43	0.850	78
Feed rpt.		0	132	754	1.0000		1.000	
		20	106	546	0.7241	14	0.918	27
		30	106	536	0.7109	17	0.891	35
		40	112	518	0.6870	31	0.869	55
		50	116	512	0.6790	39	0.850	69
Conc. + Tails	34.4	0	148	768	1.0000		1.000	
		20	116	561	0.7305	11	0.865	36
		30	118	547	0.7122	18	0.816	55
		40	116	532	0.6927	19	0.780	67
		50	120	528	0.6875	26	0.752	84
Feed + Tails	40.8	0	154	754	1.0000		1.000	
		20	124	542	0.7188	18	0.844	50
		30	135	522	0.6923	41	0.790	83
		40	144	508	0.6737	60	0.748	132
		50	147	496	0.6578	69	0.715	158
Feed + Tails rpt.		0	138	756	1.0000		1.000	
		20	108	546	0.7228	11	0.844	39
		30	119	532	0.7037	31	0.790	76
		0	130	767	1.0000		1.000	
		40	119	520	0.6780	46	0.748	105
		50	122	518	0.6754	51	0.715	122
Tails	66.7	0	157	744	1.0000		1.000	
		20	132	546	0.7339	23	0.771	76
		30	141	529	0.7110	41	0.688	131
		40	150	512	0.6882	61	0.626	191
		50	157	496	0.6667	78	0.580	249
Tails rpt.		0	142	755	1.0000		1.000	
		20	114	552	0.7311	14	0.771	60
		30	120	524	0.6940	31	0.688	109
		40	133	504	0.6675	57	0.626	176
		50	149	500	0.6622	83	0.580	245

TABLE 2b

Counts for SiKα and MnKα Obtained in Series 2

Sample	% SiO$_2$	Solids	SiKα	MnKα	B	BC cps	M	MC cp.
Conc.	2.9	0	169	1265	1.0000		1.000	
		20	155	1075	0.8498	13	0.956	22
		30	160	1044	0.8253	25	0.941	37
		40	163	1048	0.8285	28	0.929	43
Feed	13.4	0	184	1175	1.0000		1.000	
		20	176	1056	0.8987	12	0.924	28
		30	180	1041	0.8860	19	0.898	42
		40	182	1025	0.8723	25	0.876	54
Tails	66.7	0	205	1231	1.0000		1.000	
		20	209	1080	0.8773	33	0.771	104
		30	217	1054	0.8562	48	0.688	163
		40	226	1039	0.8440	63	0.626	223
SiO$_2$	100.0	0	207	1261	1.0000		1.000	
		20	220	1107	0.8747	44	0.676	165
		30	227	1068	0.8470	61	0.576	258
		40	241	1050	0.8327	82	0.498	374
Feed + SiO$_2$	56.8	0	202	1224	1.0000		1.000	
		20	202	1089	0.8897	25	0.800	82
		30	210	1054	0.8611	41	0.737	129
		40	213	1052	0.8595	46	0.687	159

The decrease in scatter from water for AgKα from a mean of 964 for Series 1 to 541 for Series 2 is due to the decay of the Cd-109. The corresponding scatter at the FeKα energy level decreased from 238, omitting the high 316 value, to 135. The new Fe-55 source caused the MnKα scatter counts to increase from a mean of 755 to one of 1231.

BACKSCATTER CORRECTIONS

The counts for iron from a given slurry consist of the FeKα radiation excited by the Cd-109 from the iron component of the stream plus the degraded silver radiation scattered at the iron wavelength. The FeKα counts for water are from the degraded radiation only. The AgKα counts, in all cases, are due to Rayleigh scatter which causes no change in energy.

Scatter varies inversely with the mean atomic number of the scatterer and hence, in slurry work, with the composition of the stream. Therefore, scatter counts provide a good first correction for the character-istic radiation counts and also a basis for the estimation of solids content if the composition of the solids does not vary too widely.

A scatter correction - called B, for backscatter - may be calculated for slurries using the counts from water as reference. B is defined as the scatter from the slurry divided by the scatter from water. For a 20% solids stream, the value of B to be applied to the FeKα counts is the silver counts from the 20-% stream divided by the silver counts from water or AgKα (20%)/AgKα (0%). For general application, this becomes $B = AgK\alpha (x\%)/AgK\alpha (0\%)$. Similarly, the correction for silicon counts is MnKα (x%)/MnKα (0%).

To determine the B-corrected counts per second for a stream, the counts from the stream are divided by the appropriate B and the counts from water are subtracted from the result, i. e., $\dfrac{FeK\alpha\ (x\%)}{B\ (x\%)} - FeK\alpha\ (0\%)$. The values for B and these BC cps are included in the tables of results.
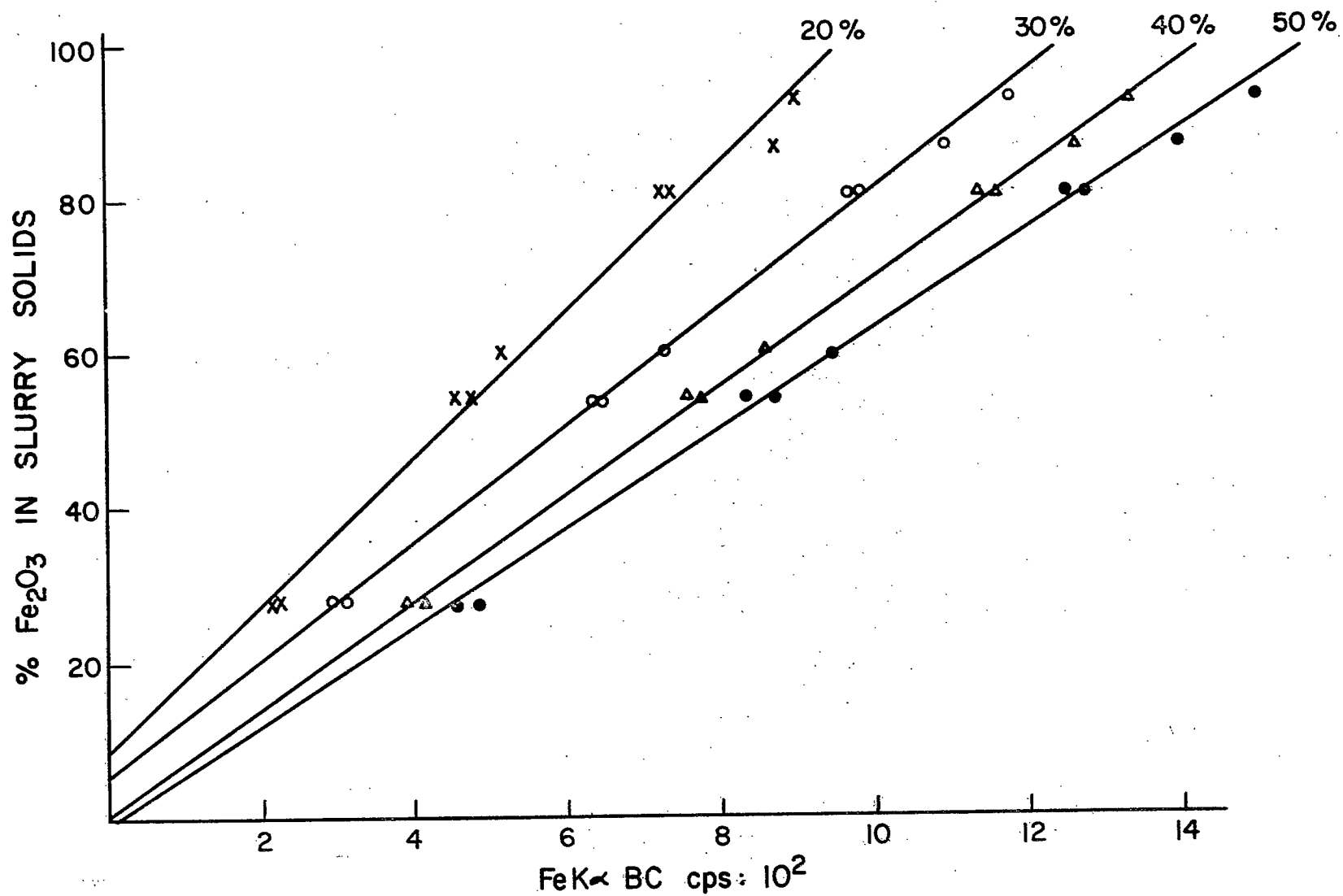
Figure 2. Effect of Slurry Density on Iron BC Regression Lines

In making this correction it is assumed that the degraded radiation scattered at the iron wavelength is affected by the slurry to the same extent that the Rayleigh scatter is, and that FeKα is absorbed by the slurry in a like manner to AgKα. From the results in Tables 1a and 1b, it is evident that this B correction is satisfactory for the determination of iron from the FeKα counts. However, the silicon BC counts in Tables 2a and 2b are not satisfactory.

The BC cps for FeKα for Series 1 are plotted in Figure 2 for the four streams. They show that counts increase significantly with solids content as would be expected. The equations for these lines are:

$$20\% \text{ solids: } Y = 0.09442X + 9.1978$$
$$30\% \text{ solids: } Y = 0.07547X + 5.4108$$
$$40\% \text{ solids: } Y = 0.06949X + 0.3100$$
$$50\% \text{ solids: } Y = 0.06380X - 1.0349$$

where Y is $\%Fe_2O_3$ and X is BC cps for FeKα.

ABSORPTION COEFFICIENT CORRECTIONS

In using B correction, it is assumed that iron and silicon radiations are absorbed by the slurries to a similar extent as those of silver and manganese, respectively. A comparison of the mass absorption coefficients listed in Table 3 (2, 3) shows that such is not the case. All components of the streams absorb SiKα much more strongly than the other radiations. Therefore, it would seem that B is not a large enough correction for this radiation.

Absorption is additive and the mass absorption coefficients of the compounds are calculated by summing the product of the weight fraction of each element present in the compound and that element's absorption coefficient for the radiation in question. The calculation of the water coefficient for the four radiations of interest is shown in Table 3.

TABLE 3

Mass Absorption Coefficients of Slurry Components for Radiations Counted

Radiation:	$SiK\alpha$	$MnK\alpha$	$FeK\alpha$	$AgK\alpha$
λ	7.13	2.10	1.94	0.56
keV	1.74	5.90	6.40	22.16
Component				
H	5.25	0.5	0.48	0.37
O	965.6	30.9	24.5	0.75
Fe	2502.1	89.4	71.4	20.0
Si	327.9	145.0	115.5	3.3
H_2O	860	27.56	21.86	0.71
SiO_2	666	84.53	67.27	1.95
Fe_2O_3	2040	71.85	57.33	14.22

Calculation of Water Coefficients: H_2O 11% H and 89% O by weight

For $SiK\alpha$: $0.11 \times 5.25 + 0.89 \times 965.6 = 859.96$

$MnK\alpha$: $0.11 \times 0.50 + 0.89 \times 30.9 = 27.56$

$FeK\alpha$: $0.11 \times 0.48 + 0.89 \times 24.5 = 21.86$

$AgK\alpha$: $0.11 \times 0.37 + 0.89 \times 0.75 = 0.71$

The absorption correction factor, M, was calculated for silicon with respect to manganese for each ore fraction, as shown in Table 4 for the concentrate. The ratio of coefficients of the elements for water is taken as unity and the ratio for the slurry streams is compared with it. Again, the absorption for the slurries is calculated by summing the products of the weight fractions of the components and the absorption coefficients listed in Table 3. The MC-corrected counts are obtained by dividing the counts from the stream by the product of the appropriate B and M and subtracting the water counts from the result: $MC \ cps = \dfrac{SiK\alpha(x\%)}{B(x\%) \cdot M(x\%)} - SiK\alpha(0\%)$. The calculated factors and the MC cps for silicon are included in Tables 2a and 2b.

The correction could not be applied to silicon without also applying it to iron to check its validity. The calculation of M for iron in ore tailings is included in Table 4 and the factors and MC cps in the tables of iron counts. It is interesting to note that the absorption corrections are larger for the iron radiation than they are for that of silicon - the relative absorption effect is masked at first sight by the much larger silicon coefficients.

BC and MC counts are compared with net counts for iron in ore fractions in streams having 30% solids in Figure 3a, and for silicon in SiO_2 at three slurry densities in Figure 3b. There is a pronounced increase in counts when the absorption factor is used. The exponential nature of the response is due to the fact that absorption coefficients are derived as exponentials and are applied in a linear manner. The response becomes linear when logarithms of the counts are used.

Absorption correction factors were calculated including absorption of the primary X-ray beam as well as the secondary, but these have not been included because their use made no significant change in results.

TABLE 4

Calculation of Absorption Factors

% Solids	SiKα M for concentrate - 92% Fe_2O_3 and 3% SiO_2 Composition (%)			μSiKα	μMnKα	Si/Mn	M
0	100 H_2O			860	27.56	31.20	1,000
20	80 "	+ 18.4 Fe_2O_3	+ 0.6 SiO_2	1067	35.78	29.82	0.956
30	70 "	+ 27.6 "	+ 0.9 "	1171	39.88	29.36	0.941
40	60 "	+ 36.8 "	+ 1.2 "	1275	43.99	28.98	0.929

% Solids	FeKα M for tails - 66.7% SiO_2 and 27.7% Fe_2O_3 Composition (%)			μFeKα	μAgKα	Fe/Ag	M
0	100 H_2O			21.86	0.71	30.7887	1.000
20	80 "	+ 5.5 Fe_2O_3	+ 13.3 SiO_2	29.59	1.61	18.3857	0.597
30	70 "	+ 8.3 "	+ 20.0 "	33.51	2.07	16.2095	0.526
40	60 "	+ 11.0 "	+ 26.7 "	37.38	2.51	14.8877	0.484
50	50 "	+ 14.8 "	+ 33.4 "	41.88	3.11	13.4623	0.437
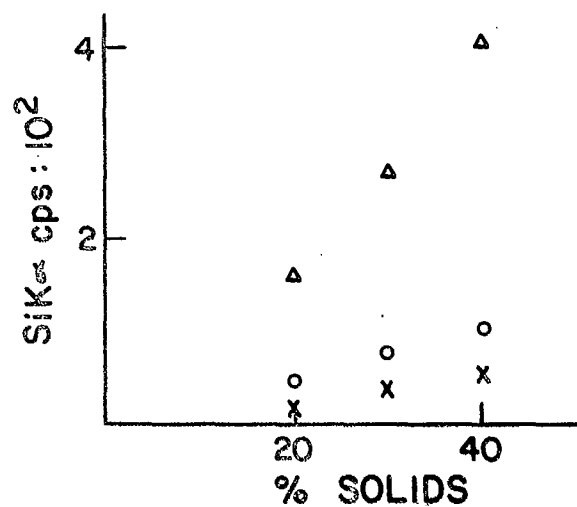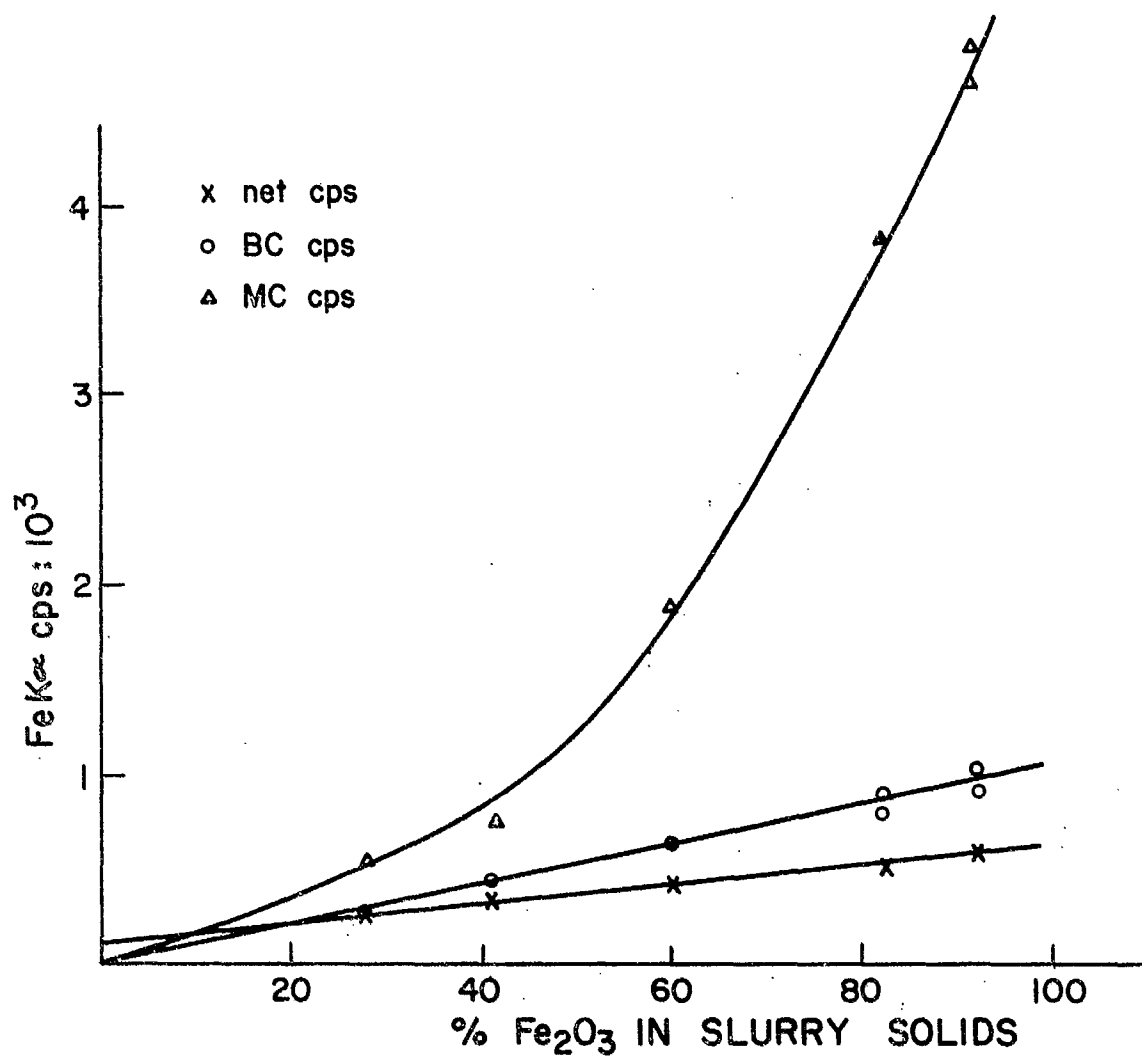
Figure 3. Effect of Corrections on Countrates
  a. FeKα in 30% Slurries
  b. SiKα at Three Slurry Densities.

TABLE 5

Equations for the Determination of Percent Solids, Series 1

Unknown	Known	X	Y	Equation	r°	se*	Sample
% solids a	Ag B's	B	reciprocal of square root of % solids	$Y = 0.70608X - 0.3292$	+0.941	0.012	tails
				$Y = 1.72595X - 0.8635$	+0.936	0.007	conc
				$Y = 1.37444X - 0.6695$	+0.988	0.005	feed
% solids b	Mn B's	B	"	$Y = 1.09764X - 0.5855$	+0.955	0.009	tails
				$Y = 1.01717X - 0.5404$	+0.952	0.005	feed
% solids c	B's	Ag B x MnB	"	$Y = 0.61419X - 0.1296$	+0.955	0.009	tails
				$Y = 1.11124X - 0.2984$	+0.929	0.010	conc & feed
% solids 1	Mn/Fe	ratio	10/% solids	$Y = 0.5756\ X - 0.4241$	+0.983	0.020	tails
				$Y = 1.2504\ X - 0.4357$	+0.999	0.006	conc
				$Y = 1.0156\ X - 0.3837$	+0.998	0.006	feed
% solids 2	Mn/Fe	ratio	cube root of % solids	$Y = 5.6747 - 1.9022X$	−0.964	0.061	tails
				$Y = 6.6675 - 5.8798X$	−0.997	0.019	conc
				$Y = 5.9430 - 4.0050X$	−0.993	0.027	feed
% solids 3	$(Mn/Fe)_x$ x $(Mn/Fe)_0$	ratio	10/% solids	$Y = 0.1511\ X - 0.2951$	+0.995	0.005	tails
				$Y = 0.4166\ X - 0.4983$	+0.998	0.004	conc
				$Y = 0.3006\ X - 0.3306$	+0.997	0.004	feed

*results are ± this figure which is in units of Y;
°perfect correlation is 1.000.

DETERMINATION OF SOLIDS CONTENT

As mentioned above, B is a measure of the solids content of a stream, provided the composition of the solids does not vary greatly. In the former investigation[1], it was possible to determine the cube root of the solids content directly from B. As had been noted in the present tests, B did not always decrease steadily with the stream density. However, with the two values for B available, an attempt was made to correlate B and per cent solids. Because B was relatively insensitive to slurry changes, the product of the two factors was also used. The matrix of the sample had a pronounced effect on the relationship and it was necessary to establish separate lines for the different ore-fractions. The equations of these lines are listed in Table 5 for Series 1 counts.

The absorption coefficients of SiO_2, Fe_2O_3, and H_2O for $MnK\alpha$ and $FeK\alpha$ are similar, as Table 3 shows, so the effect of the slurries on these radiations should be comparable. Both radiations are affected by the solids composition and solids content of the slurry, but in different ways. Both suffer similar absorption from silica and water; but, though $MnK\alpha$ is absorbed by hematite, $FeK\alpha$ originates in the hematite and varies in intensity with its concentration in the stream. Therefore, for samples of similar composition, the ratio between these radiations should vary with the iron content and, hence, with the solids content. The percent solids was found to vary linearly with the Mn/Fe ratio but the simple solids value could not be used. The response was linear and direct if the inverse of the solids was used and linear, but inverse, if the cube root of the solids was considered. The equations for these lines are also included in Table 5.

On an atomic basis, slurries have much larger water contents than they do on a weight basis. In X-radiation studies, we are concerned with atomic phenomena, so it was considered probable that the Mn/Fe

TABLE 6

Percent Solids Results for Series 1

| Sample | Expt'l | Percent Solids | | | | | |
| | | by B factors | | | by S_{Mn}/I_{Fe} | | |
		a	b	c	1	2	3
Conc	20	19.6		24.2	19.9	(11.4)	19.0
	30	31.9		37.9	30.9	29.6	30.3
	40	37.6		42.0	39.2	39.9	39.2
	50	49.6		48.6	49.5	49.4	50.8
Feed	20	19.4	(27.5)	20.1	20.1	(15.0)	21.2
	30	28.6	31.9	27.7	29.5	29.6	30.6
	40	40.6	39.6	38.6	38.4	39.5	39.3
	50	47.6	54.0	51.0	50.2	48.3	50.1
Feed rpt.	20	20.5	(26.0)	20.1	20.3	(15.4)	20.9
	30	29.9	29.8	27.1	29.4	29.4	29.7
	40	42.7	40.1	40.1	41.0	41.7	40.3
	50	47.6	44.5	45.0	51.8	49.4	49.8
Tails	20	19.5	20.6	22.5	20.9	26.5	(22.5)
	30	26.5	26.3	32.6	29.6	31.5	30.4
	40	35.4	34.6	45.7	43.7	43.5	41.6
	50	42.8	46.9	52.6	55.9	49.9	50.2
Tails rpt.	20	23.3	21.2	19.7	20.7	19.1	(23.5)
	30	32.3	32.3	26.5	26.8	28.1	29.5
	40	45.0	46.2	35.4	37.9	39.3	39.8
	50	54.9	50.2	44.5	49.8	47.0	49.8

Points for 20% slurries lay off the line in some instances. These were not used to calculate the equation. Values for these slurries were calculated, however, as a matter of interest and appear in parentheses in the table.

ratios would be enhanced and made more significant if they were multiplied by the corresponding ratio for water. This product was related to the inverse of the solids and the relationship is included in Table 5.

The values obtained for the percent solids, back-calculated by these methods, are given in Table 6. It is evident that the ratios give better results than does B.

Similar relationships were found for Series 2 counts, but the Mn/Fe ratio line for solids had a more pronounced slope because the MnKα flux was greatly increased by the new Fe-55 source and because the FeKα counts were reduced by the loss in intensity from the Cd-109 in the interval between the tests. The response of the ratio and of B to percent solids is shown graphically for this series in Figure 4.

DENSITY FACTORS

It was understood that, in the plant process, slurry streams would be maintained close to 40% solids, so it was to this basis that counts were converted. The correction to be applied to the counts from a slurry to make them equivalent to the counts that a 40% slurry would give was originally calculated by dividing the B counts from the 40% slurry by those from slurries of the other solids contents. This result was then divided by the corresponding inverse ratio of the scatter counts to give D, the density factor:

$$\text{D for 30\% solids} = \frac{\text{Fe cps (40\%)/Fe cps (30\%)}}{\text{Ag cps (30\%)/Ag cps (40\%)}}$$

The D values for slurries with less than 40% solids are naturally greater than unity, and those for slurries with more than 40% are less than unity.

Lines were calculated for each type of sample for the variation of D with solids content for Series 1:
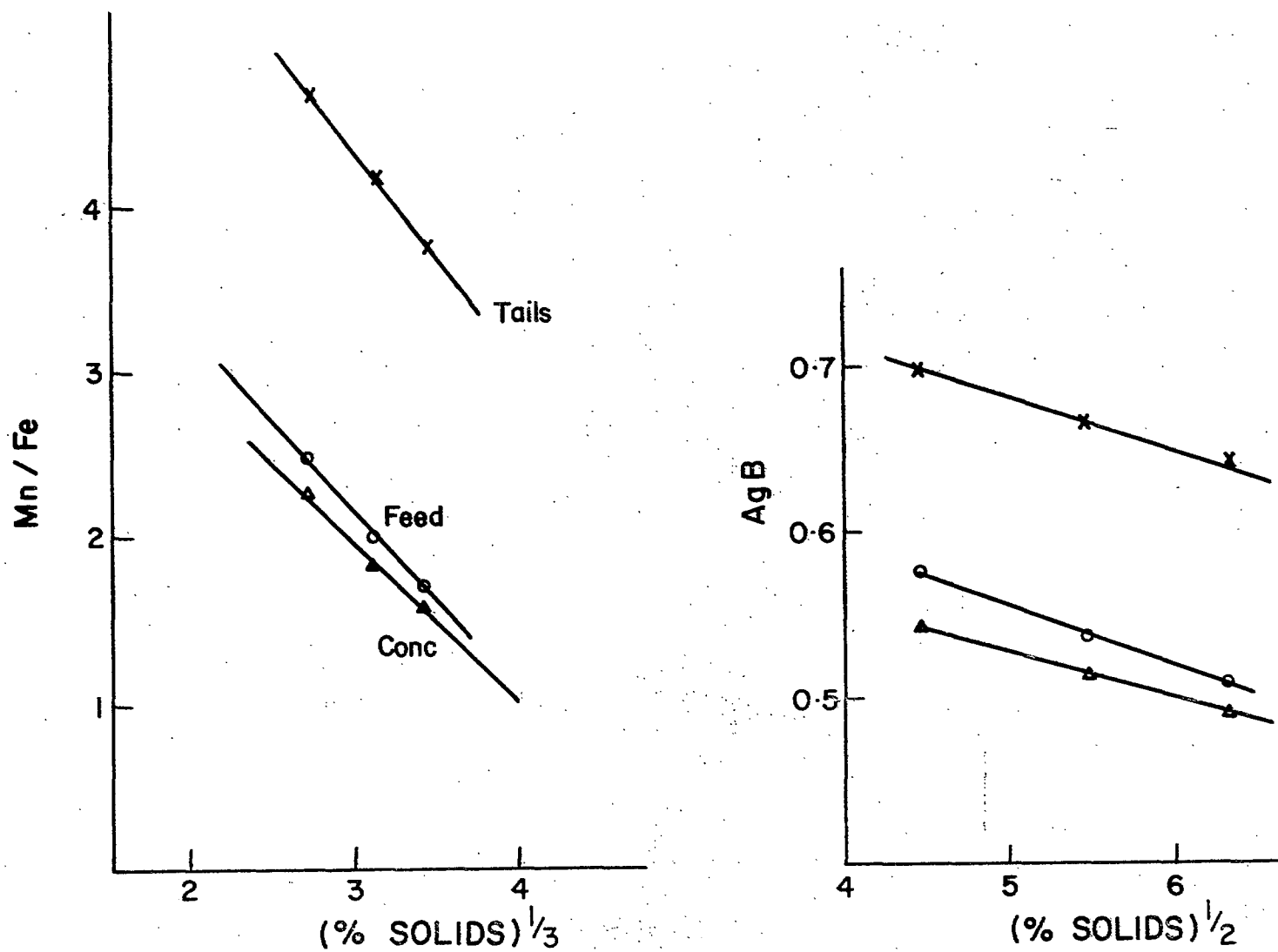
Figure 4.   Determination of Solids Content of Slurries, Series 2.
    (a)   Relationship between Percent Solids and Mn/Fe;
    (b)   Relationship between Percent Solids and AgB's.

$$\text{for conc:} \quad Y = 1.5962X + 0.5936$$
$$\text{for feed:} \quad Y = 1.7788X + 0.5547$$
$$\text{for tails:} \quad Y = 2.5965X + 0.3696$$

where $Y = D$ and $X = 10/\%$ solids. The slopes of the lines vary significantly with the iron content of the sample as would be expected.

Because of the simple nature of the ore, which is composed mainly of two oxides, it seemed reasonable to effect the transition from the slurry density of the stream under investigation to 40% solids by means of regression rather than separate factors for each type of ore fraction. As the first step in the establishment of such a regression, BC and MC cps for FeKα were tabulated separately and, taking the 40% counts in each case as Y and those of the other densities in turn as X, lines for the conversion of counts from each slurry density used to 40% were established:

	Line for BC cps	Line for MC cps
Series 1, for 20%:	$Y = 1.3617X + 126.22$	$Y = 2.0728X - 38.53$
30%:	$Y = 1.0855X + 73.83$	$Y = 1.3137X - 8.65$
50%:	$Y = 0.9188X - 20.04$	$Y = 0.8183X - 3.63$
Series 2, for 20%:	$Y = 1.5104X + 64.42$	$Y = 2.2156X - 34.48$
30%:	$Y = 1.1680X + 34.70$	$Y = 1.3708X - 2.50$

The intercepts for the MC lines are small compared with the MC count-rates and may be ignored; for the BC counts, they vary inversely with stream density.

As the second step in the establishment of the regression factors, the equation for 40% counts ($Y = 1.00X + 0.0$) was included with those above (see Figure 5a). The values of the slopes, b, varied linearly with the slurry density for both BC and MC cps. (This relationship for the MC cps is shown in Figure 5b.) Thus it was possible to establish a further equation for each for the response of the slope of the conversion line to slurry density:
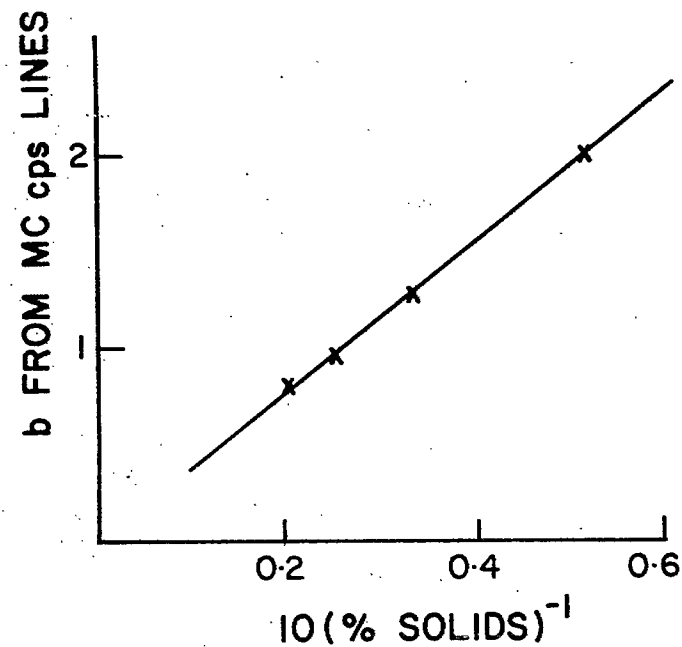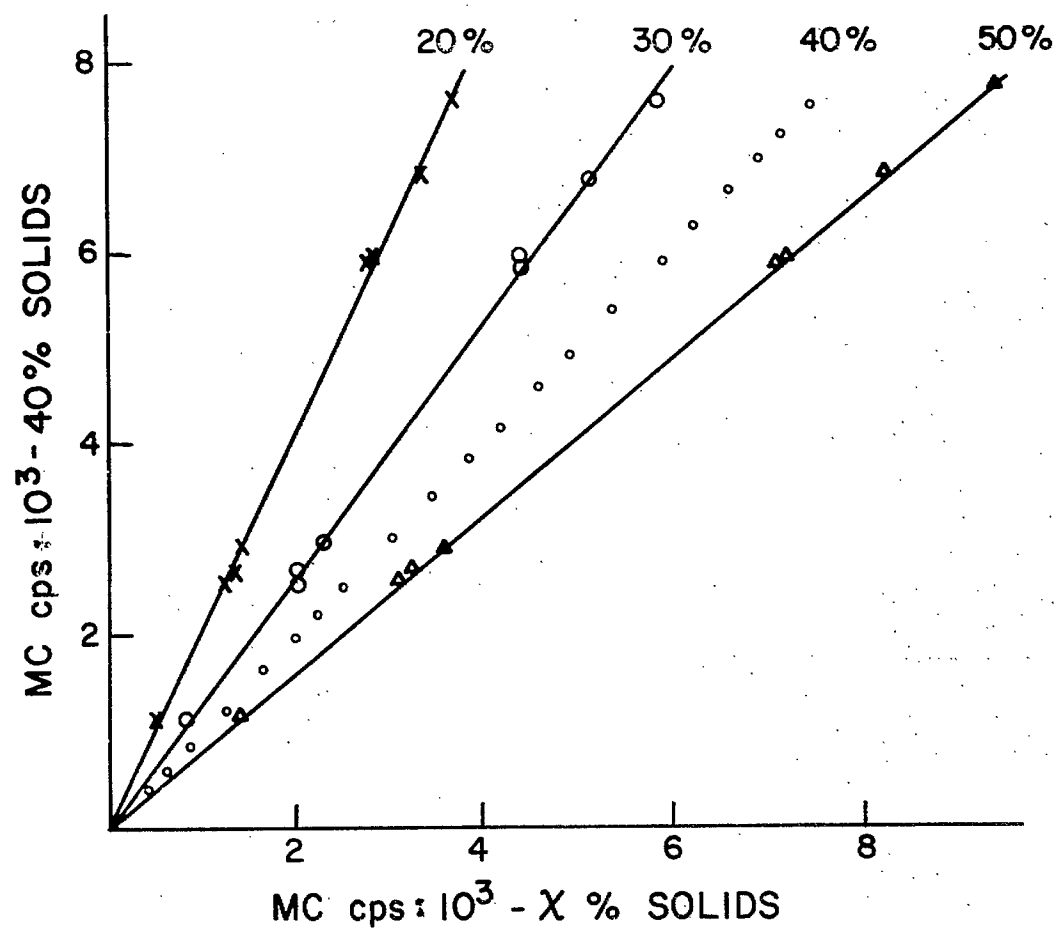
Figure 5. Conversion of FeKα cps to 40% Solids.
(a) Relationship of 40% Solids Counts to those of other Densities;
(b) Response of Slopes of MC cps Lines to Slurry Density.

	Line for BC cps		Line for MC cps	
Series 1:	$Y = 1.4615X + 0.6238$	(B1)	$Y = 4.2094X - 0.0458$	(M1)
Series 2:	$Y = 2.0373X + 0.4927$	(B2)	$Y = 4.8791X - 0.2277$	(M2)

where Y = the slope of the line for conversion to 40% solids and X = 10/% solids.
The intercept, a, for the conversion line for the BC cps varied inversely
with the cube root of the percent solids:

$$\text{Series 1:} \quad Y = 560.5051 - 159.5372X \quad \text{(B3)}$$
$$\text{Series 2:} \quad Y = 312.1070 - 90.5960X \quad \text{(B4)}$$

Thus, once the percent solids in a stream is established,
substitution in the appropriate equation will give the slope and intercept,
if required, for the conversion of the iron counts to a 40% solids basis.
The actual D values for each type of sample are no longer needed. For
example, if N B-corrected counts are recorded from a stream of x%
solids, x is substituted in Equation B1 to give Yb and in B3 to give Ya;
then N is substituted in the equation using these values of slope and
intercept to determine the equivalent counts for a 40% solids stream:
$Y = Yb.N + Ya$.

Similar results were obtained for the treatment of SiKα cps,
the equations for the conversion of MC counts to 40% solids being:

	Series 1 Lines	Series 2 Lines
20%	$Y = 2.9672X - 22.81$	$Y = 2.4616X - 54.47$
30%	$Y = 1.5885X - 13.86$	$Y = 1.4262X - 18.36$
50%	$Y = 0.7568X \times 6.91$	

For these lines the intercept is large enough to be significant compared to
the low countrates. Fortunately, it had a linear response to solids content.
This response, and that of b as well, are shown in Figure 6 for Series 1.
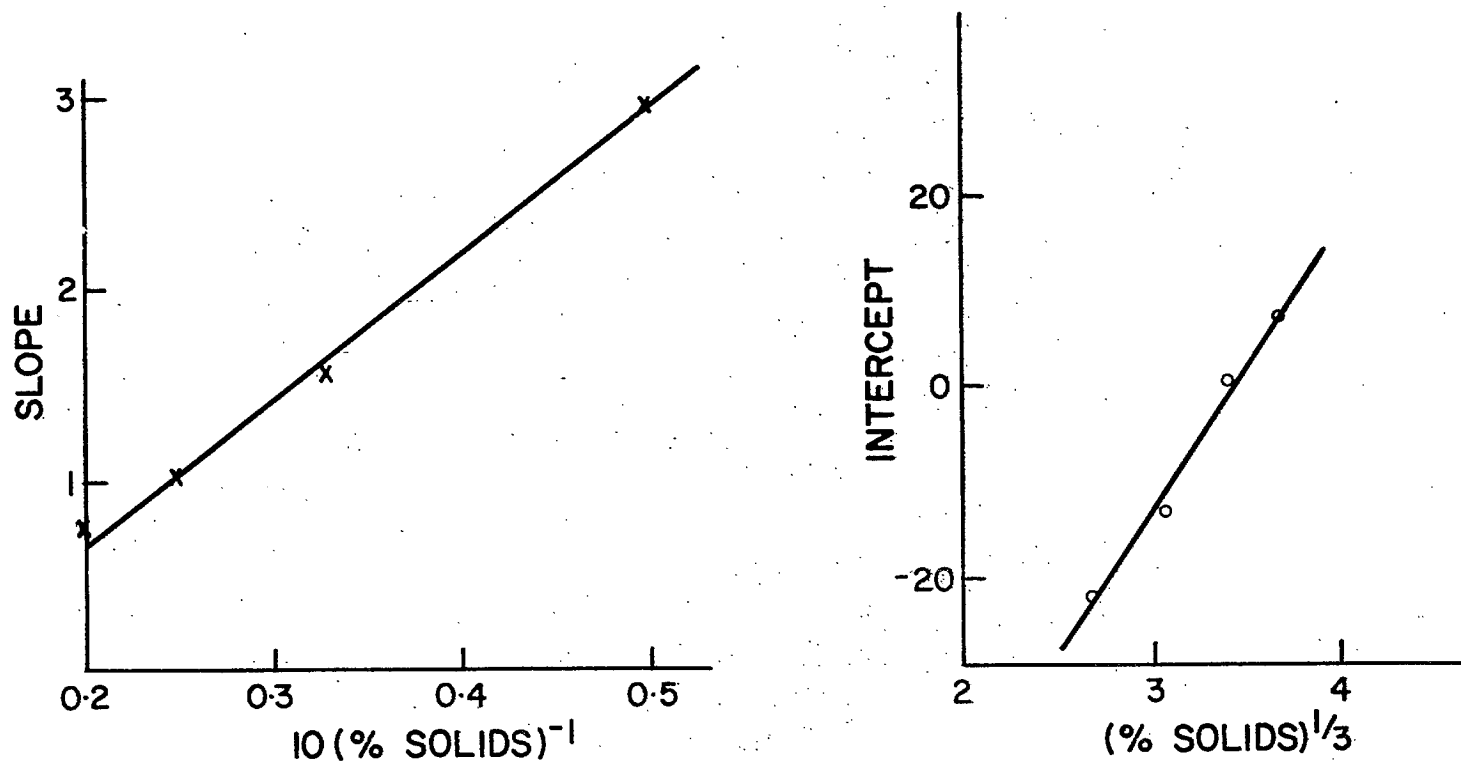The equations for both series were:

Figure 6. Determination of Conversion Factors to 40% Solids for SiKa.

    (a) Response of Slopes to Slurry Densities;

    (b) Response of Intercepts to Slurry Density.

Series 1, slope : Yb = 7.3096X - 0.7756
intercept: Ya = 31.8006X - 110.1944

Series 2, slope : Yb = 5.8754X - 0.4825
intercept: Ya = 77.7651X - 263.8174

For the slopes, X is 10/% solids and, for the intercepts, it is the cube root of the solids.

DETERMINATION OF SAMPLE COMPOSITION

The FeKα counts obtained on the ore fractions were all calculated to 40% solids and equations were derived from them for the iron present in the sample. The equations were:

BC cps	MC cps
Series 1: $Y = 0.07004X - 0.0079$	$Y = 76.01276X - 204.7365$
Series 2: $Y = 0.07000X + 7.2838$	$Y = 72.0445X - 182.6995$

where Y is % Fe_2O_3 in the solids and X is BC cps in one case and the logarithm of MC cps in the other. The standard errors of prediction were 2.0 and 2.7% for BC cps, Series 1 and 2, and 1.4 and 1.7% for the MC'cps

The logarithms of the MC cps for SiKα gave the equations:

Series 1: $Y = 96.6018X - 156.0199$
Series 2: $Y = 102.3197X - 164.4513$

with corresponding errors of 4.2 and 3.5%. In these equations, the counts from the iron concentrates, containing less than 3% SiO_2, were not used.

For both elements the MC lines have similar slopes. For iron, the BC lines have identical slopes. The increase in flux for SiKα, due to the new source, and the loss in flux for FeKα, caused by the aging of the Cd-109, have little effect on the response of counts to composition when they are compared at the 40% solids level, although the number of counts changes significantly.

The steps in the calculation of composition are as follows:

1. Divide the AgKα and MnKα counts from the stream by those obtained from water to determine B.

2. From Mn/Fe, or from B, determine the solids content of the stream.

3. From the solids content determine Yb and Ya, if needed, for both radiations.

4. Divide FeKα and SiKα counts by their B's and subtract the water counts to determine the BC cps for these elements.

5. Substitute the BC cps in equations using Ya as intercept and Yb as slope to determine BC cps at 40% solids.

6. Substitute the BC cps from Step 5 in the appropriate equation to determine the approximate percentage of Fe_2O_3 and SiO_2.

7. From the approximate composition, determine the value of M using the appropriate absorption coefficient ratio - Si/Mn or Fe/Ag.

8. Apply the M's along with the B's to the original counts and subtract the water scatter to determine the MC cps.

9. Calculate the MC cps to 40%-solids basis as in Step 5, using the proper values for Ya and Yb.

10. Substitute the MC cps from Step 9 in the proper equations and determine Fe_2O_3 and SiO_2 percentages.

11. Reiterate Steps 7 to 10 until there is no significant change in the results.

DISCUSSION

The use of radioisotopes for the excitation of slurry streams offers several advantages, but also suffers one serious drawback. The source is decaying and the flux of the exciting radiation decreases at a rate dependent upon the half-life of the isotope. This means that factors and regression lines must be revised continually. Cd-109 has a half-life of 1.3 years and Fe-55 of 2.7 years, that is, these isotopes will lose half of their activity in these periods of time. The lapse of time has a greater significance on the measurement of FeKα than that of SiKα.

The same cadmium source was used to excite the iron in the samples of both series. The mean value of the AgKα scatter was 964 cps for the first and 541 for the second series, whereas the countrates of the scatter at the FeKα wavelength were 238, omitting the high result, and 135. For the second series, the countrates were 56% and 57% of those of the first series, comparing well with the reduction in the flux of the isotope to 58% of its original value. However, if the counts for Series 2 from the slurries containing the conc + tails mixture are divided by the Series 1 counts, the mean result for AgKα is 0.48 and for FeKα 0.69. From these values, it is concluded that neither half-life nor scatter counts can be used to compensate for the loss in counts with time. If they could, then the B factors from similar streams would be the same for both series, allowing for counting errors, and they are not. Therefore, standards must be used to check the effect of isotope decay on significant variables.

The effect of flux on countrates was investigated experimentally on synthetic streams and the results are presented in Appendix A.

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   of Aqueous Ore Slurries, Part 1: Copper-Zinc Sulphide Ores",
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CONCLUSIONS

Radioisotopes may be used for on-stream analysis of ore slurries when isotopes suitable for the excitation of the components of the ore are available. A different isotope might be required for each element to be determined depending on the energies involved.

Because silicon has a poor fluorescent yield and its radiation is strongly absorbed by water, the determination of this element in concentrations below 10% in the stream solids is not satisfactory.

The effect of the decay of the exciting isotopes on significant variables must be revised constantly.

ACKNOWLEDGEMENTS

TABLE A1

Response of Scatter to Flux - MnKα cps from Synthetic Slurries

Flux	a	b	c	d
1	262	233	224	220
2	522	467	443	432
3	831	742	718	671
4	1177	1061	1064	960

TABLE A2

Response of SiKα Countrates from Synthetic Streams to Flux

Flux	a	b	c	d
1	22	60	69	87
2	60	132	169	203
3	99	203	261	321
4	135	290	375	452

TABLE A3

BC Countrates for SiKα from Synthetic Samples

Flux	b	c	d
1	45	59	82
2	88	151	199
3	128	203	298
4	203	296	435

APPENDIX A

The Effect of Flux on Countrate

During the analysis of the counts from the slurry streams, it was noticed that scatter and fluorescent counts did not respond to differences in the activity of the sources in a comparable manner. The scatter from Cd-109 was roughly proportional to the loss in activity with time, but the FeKα countrates were not. Similarly, the increase in SiKα countrates with the second Fe-55 source did not correspond to the increase in MnKα counts. This observation led to a decision to investigate the response of counts from slurries to flux.

The second Fe-55 source was in the form of two bars, approximately 1.25 by 0.5 inch. When these were placed in the chamber, the exposed area was 1 by 0.5 inch. The bars were positioned one each side of the counter window at an angle of 45° to the sample surface. It was possible to remove one of the bars and to absorb the radiation from half of a bar by covering it with a square of platinum. This made irradiation of the samples by four different fluxes from Fe-55 relatively simple. It was not feasible to dismount the equipment during a slurry run to position and remove the platinum; hence, solid samples had to be used for this investigation.

Tartaric acid, ground to pass 100 mesh, was chosen to represent the water of the slurries and synthetic samples were prepared by mixing Fe_2O_3 and SiO_2 with it. To represent a sample containing 60% SiO_2 and 20% Fe_2O_3 in slurries of 20, 30 and 40% solids, the following mixtures were prepared:
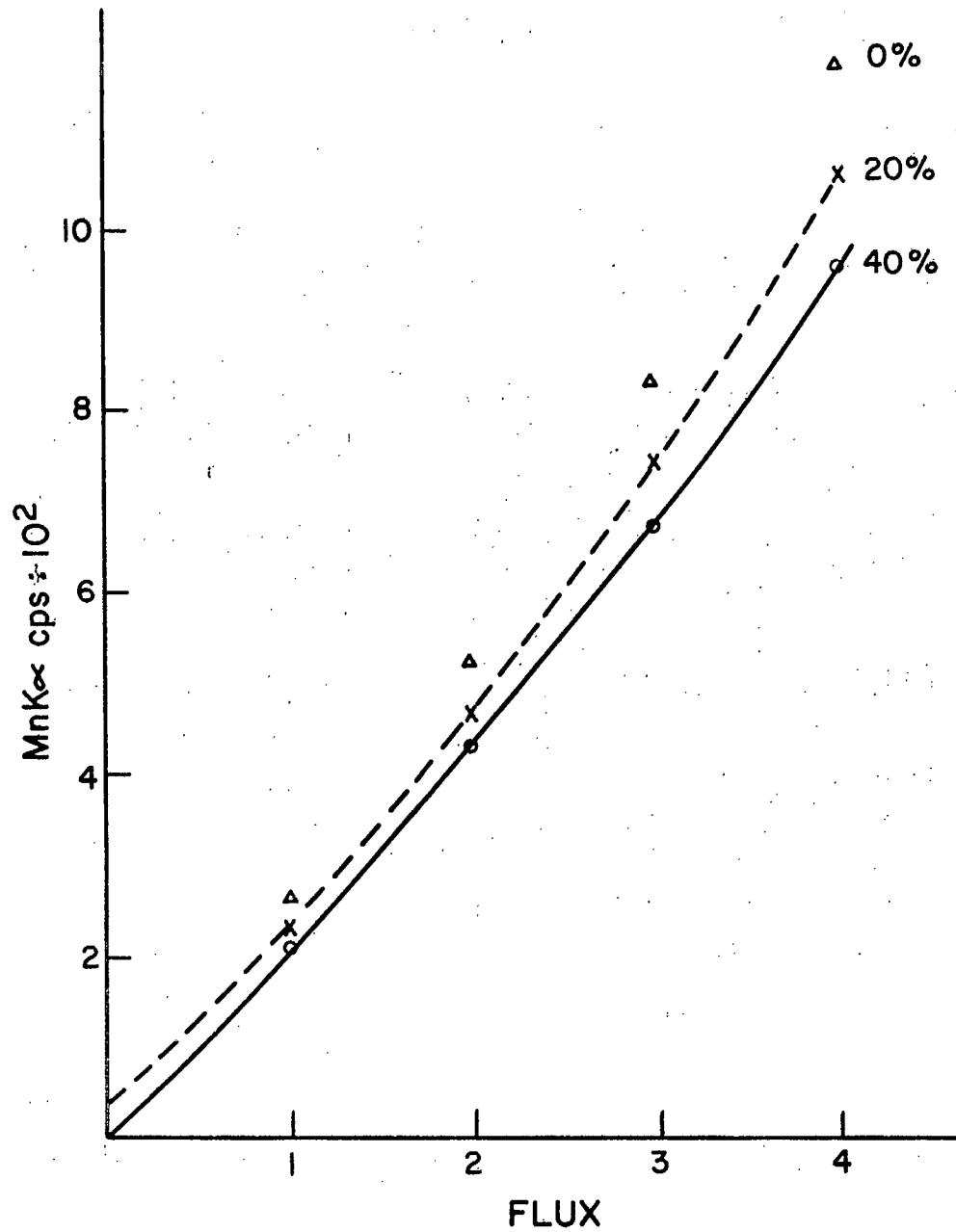
Figure A1. Response of the Scatter from Synthetic Slurries to Flux.

	Tartaric Acid	SiO_2	Fe_2O_3
a	10 g		
b	8 g	1.6 g	0.4 g
c	7 g	2.4 g	0.6 g
d	6 g	3.2 g	0.8 g

The counts obtained after these samples were packed in a holder and while they were exposed to 0.5, 1, 1.5 and 2 bars of the source (Flux 1, 2, 3 and 4) are presented in Tables A1 and A2 for the scatter and fluorescent radiation, respectively. There is an obvious discrepancy in the scatter counts for Flux 4 - the counts from the 30% sample are too high. All the samples were exposed to one flux; then the flux was varied and all were again exposed, so that samples were repacked for each flux and packing is a variable to be considered.

The response of the scatter is shown graphically in Figure A1 and lines are shown for two densities. The 30% countrates are omitted because they are close to the 20% results. The response decreases with solids content and is not linear, although the curvature is slight. The equation for the 20% line is $Y = 42.5 + 169.4X + 21.34X^2$ where X represents the flux. The 40% line is represented by the equation $Y = 201X + 9.3X^2 - 1.51$.

The linear response of SiKα to flux is shown in Figure A2 for the original counts. The BC cps listed in Table A3 had a similar response. The equations calculated for both types of countrates were:

	Original cps	BC cps
a	$Y = 33.0X - 7.5$	
b	$Y = 76.1X - 19.0$	$Y = 51.4X - 12.5$
c	$Y = 101.0X - 34.0$	$Y = 76.3X - 13.5$
d	$Y = 121.3X - 37.5$	$Y = 115.8X - 36.0$

Although the response is linear, the counts do not relate to the flux on a one-to-one basis. To do this, the slope of the line relating counts to flux would have to be 1.00. It is the lack of a 45° response that makes isotope decay factors not relevant as count corrections.
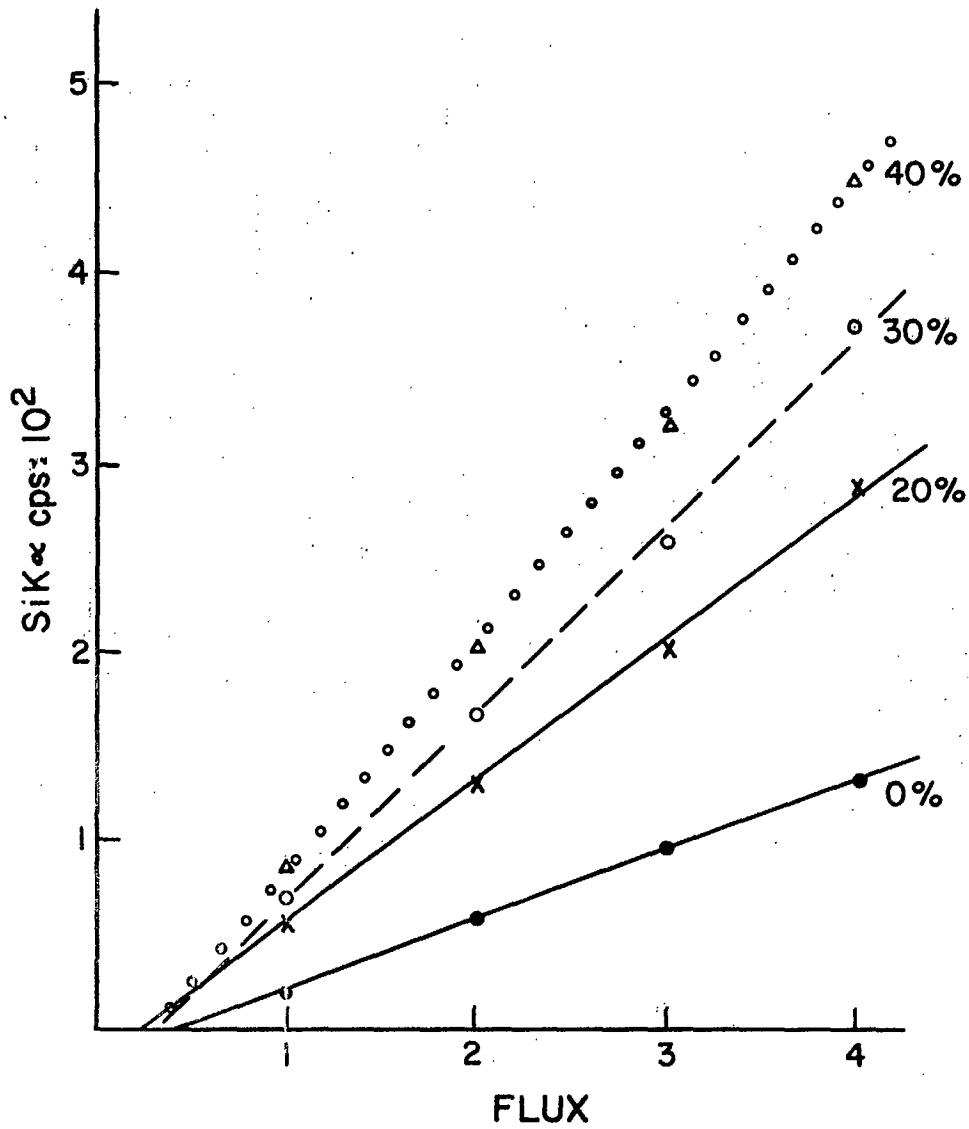
Figure A2. Response of SiKα to Flux.

Another way of showing that flux is not applicable is to take the counts from the lowest flux as unity and relate the other counts to them. When this is done for the BC cps, the results are as follows:

	1	2	3	4
b	1.00	1.96	2.85	4.81
c	1.00	2.56	3.44	5.02
d	1.00	2.43	3.63	5.30

Similar figures are obtained if the counts in Table A2 are treated the same way. Thus, standards are necessary to check the effect of time on all the factors and relationships used for calculation of results.