TR17_175_183

R.630 Sieb-technik grinding and its effect on tin, tungsten assays by XRF.

The original objective of this project was to study the sizing of the products made by the various Sieb grinding vessels from different types of rock, for it had been observed that soft materials like limestone did not behave like harder materials such as quartz.

Because one of the main uses of the Sieb mill is in the preparation of powders for pressing into XRF discs and because the degree of grinding affected the XRF results for tin and tungsten assay, the study was extended to cover the effect of grinding on the apparent tin and tungsten assays. The XRF method used is given in Appendix 1.

The three steel Sieb vessels calibrated in this investigation can be described as follows:

(*a*) 10 cm^3 net capacity - one grinding stone only.
(*b*) 100 cm^3 net capacity - one grinding stone and one grinding ring.
(*c*) 250 cm^3 net capacity - one grinding stone and two grinding rings.

The aim of the investigation was to:

(1) calibrate the three Sieb mills using quartzite, granite and limestone,
(2) find the sample particle size requirement of XRF tin analysis (cassiterite),
(3) find the sample particle size requirement of XRF tungsten analysis (wolfram),
(4) examine the Sieb grinding characteristics of Sample 711956.

SAMPLES

The following samples were used in the calibration of the three Sieb mills:

(*a*) Quartzite from Investigation R.628/N2 bin 1.
(*b*) Granite from Samples 700151, 702721 and 702864.
(*c*) Limestone from Sample 711546.

The samples were crushed and ground to -710 µm and mixed thoroughly. A sieve analysis of the prepared samples was carried out and the results are presented in Table 1.

Alluvial tin samples from north-eastern Tasmania which consisted mainly of quartz grains with minor amounts of cassiterite were used to determine the sample particle size requirement for XRF tin analysis. The samples used were as follows:

Registered Number	*% Sn*
711951	1.11
711956	2.40
711973	0.85
711991	0.36
712210	4.20
712223	6.35

Table 1. SEIVE ANALYSIS OF PREPARED SAMPLES

Particle Size µm	Fraction (BSS#)	Quartzite (*a*) % Wt	Quartzite (*a*) % Cum. Wt	Granite (*b*) % Wt	Granite (*b*) % Cum. Wt	Limestone (*c*) % Wt	Limestone (*c*) % Cum. Wt
>600	+25	0.4	0.4	2.7	2.7	4.4	4.4
>420	+36	1.4	1.8	28.6	31.3	25.6	30.0
>300	+52	2.8	4.6	15.7	47.0	11.9	41.9
>180	+85	19.9	24.5	18.8	65.8	12.4	54.3
>150	+100	9.8	34.3	5.1	70.9	3.1	57.4
>105	+150	14.4	48.7	7.3	78.2	4.9	62.3
>75	+200	12.0	60.7	6.4	84.6	4.7	67.0
>44*	C/S1	2.8	63.5	1.8	86.4	2.6	69.6
>35*	C/S2	10.1	73.6	3.2	89.6	3.6	73.2
>24*	C/S3	7.9	81.5	3.3	92.9	4.0	77.2
>15*	C/S4	6.1	87.6	2.4	95.3	4.0	81.2
>12*	C/S5	2.7	90.3	1.0	96.3	2.2	83.4
<12*	O/F	9.7	100.0	3.7	100.0	16.6	100.0
Head		100.0	-	100.0	-	100.0	-

* Limiting quartz particle size

Table 2. GRINDING CHARACTERISTICS FOR GRANITE IN SIEB VESSELS

Cyclosizer Fractions	100 cm^3 Vessel (2 g charge) 3 minutes % Wt	3 minutes % Cum. Wt	4 minutes % Wt	4 minutes % Cum. Wt	100 cm^3 Vessel (20 g charge) 3 minutes % Wt	3 minutes % Cum. Wt	4 minutes % Wt	4 minutes % Cum. Wt	250 cm^3 Vessel (50 g charge) 2 minutes % Wt	2 minutes % Cum. Wt	3 minutes % Wt	3 minutes % Cum. Wt
C/S1	0.5	0.5	0.5	0.5	1.0	1.0	0.5	0.5	0.2	0.2	0.6	0.6
C/S2	0.5	1.0	0.5	1.0	1.0	2.0	0.5	1.0	0.4	0.6	0.8	1.4
C/S3	2.5	3.5	2.5	3.5	3.5	5.5	2.5	3.5	4.0	4.6	2.8	4.2
C/S4	8.5	12.0	8.0	11.5	8.5	14.0	7.0	10.5	9.0	13.6	7.2	11.4
C/S5	7.5	19.5	6.5	18.0	6.0	20.0	5.5	16.0	6.6	20.2	5.4	16.8
O/F	80.5	100.0	82.0	100.0	80.0	100.0	84.0	100.0	79.8	100.0	83.2	100.0

Four samples from Investigation R.628 (Mt Pelion wolfram ore) were used to determine the sample particle size requirement for XRF tungsten analysis. The R.628 ore consisted of wolfram in quartz veins with siliceous country rock. The samples used were as follows:

R.628 Sample	% WO_3	% *Sn*
-3 in +1¼ in S/K	0.79	0.12
-⅞ in +½ in S/K	1.25	0.13
-½ in +⅜ in S/K	3.17	0.16
-⅜ in ore	4.18	0.07

Sample 711956 was a further alluvial tin sample from north-eastern Tasmania composed of grains of quartz with minor amounts of cassiterite.

PROCEDURE

Various grinding times, materials, and mill charges were used with each Sieb mill as will be shown in a later section.

Mill discharge was sized in a Warman Cyclosizer in each case.

(1) Calibration of 100 cm^3 with quartzite.
   (*a*) Loads of 5, 10, 15, 20, 25, 30, 35, 40, 50, 60, 70, 80, 90, 100 and 120 g were ground for periods of 4 minutes.
   (*b*) Charges of 20 g were ground for periods of 0.25, 0.5, 0.75, 1, 2, 3, 3.5, 4, 5, 6, 7, 8, 9 and 10 minutes.

(2) Calibration of 100 cm^3 vessel with granite.
   (*a*) Loads of 20, 30, 40, 50 and 60 g were ground for periods of 4 minutes.
   (*b*) Charges of 20 g were ground for periods of 0.5, 1, 2, 3, 4, 5, 6 and 7 minutes.

(3) Calibration of 100 cm^3 vessel with limestone.
   (*a*) Loads of 20, 30, 40, 50 and 60 g were ground for periods of 4 minutes.
   (*b*) Charges of 20 g were ground for periods of 0.5, 1, 2, 3, 4, 5 and 6 minutes.

(4) Calibration of 250 cm^3 vessel with granite.
   (*a*) Loads of 50 g were ground for periods of 1, 2, 3 and 4 minutes.

(5) Calibration of 10 cm^3 vessel with granite.
   (*a*) Loads of 5 g were ground for periods of 2, 3 and 4 minutes.
   (*b*) Loads of 4 g were ground for periods of 2, 3 and 4 minutes.
   (*c*) Loads of 2 g were ground for periods of 2, 3 and 4 minutes.

To determine the sample particle size requirement for XRF tin analysis, the six alluvial samples (described previously) were separately ground in 20 g charges for periods of time ranging from 0.25 to 5 minutes in 0.25 minute intervals. Grinding was carried out only in the 100 cm^3 vessel, and mill discharge was thoroughly mixed prior to submission for tin analysis.

To determine the sample particle size requirement for XRF tungsten analysis, the four samples (described previously) were separately ground in 20 g charges for varying periods of time from 0.25 to 5 minutes in 0.25 minute intervals. Grinding was carried out only in the 100 cm^3 vessel and mill discharge was thoroughly mixed prior to submission for tin analysis.

To examine the Sieb grinding characteristics of Sample 711956, 20 g charges were ground for periods of 2, 3 and 4 minutes and physically examined. Samples ground for 2 and 4 minutes were cyclosized. One sample prior to a 4 minute grind was further dried under infrared light for a period of 35 minutes.

RESULTS

Calibration of Sieb Vessels.

As a large number of results were obtained, only the significant results have been included in this report in the form of graphs (fig. 43, 44) and also in tabular form (table 2).

A graph of Sieb grinding in the 100 cm^3 vessel with cumulative weight percentage versus limiting particle size (μm) is given in Figure 43. The graph shows the effect of grinding quartzite, granite and limestone at a constant charge of 20 g for various grinding times from 0.25 to 8 minutes.

Figure 44 is a graph of Sieb grinding in the 100 cm^3 vessel with cumulative weight percentage versus limiting particle size (μm). The graph shows the effect of grinding quartzite, granite, and limestone for a constant time of 4 minutes with various charges from 10 - 80 g.

The grinding characteristics for granite in the 10, 100 and 250 cm^3 Sieb vessels are given in Table 2.

The nature of the Sieb grinding mechanism is such that materials (especially with a low hardness) are initially ground quickly, then compacted into lumps. These lumps prove quite solid and cannot be readily broken. The position of limestone plots on Figures 43 and 44 are indicative of this, and even extended granite grinding times showed slight increases in the coarser fractions on sizing.

Sieb grinding prior to XRF tin analysis.

A graph of apparent XRF tin values versus Sieb grinding time (100 cm^3 vessel) is shown in figure 45. From the graph it can be seen that the required grinding time increases slightly as the value of tin in a sample increases.

Sieb grinding prior to XRF tungsten analysis.

A graph of apparent XRF tungsten values versus Sieb grinding time (100 cm^3 vessel) is shown in figure 46. From the graph it can be seen that all samples require a minimum grinding time of 3.5 minutes to approximate the maximum apparent tungsten value.

Examination of the grinding characteristics of Sample 711956.

(1) A 20 g sample ground for 4 minutes, became very compacted with the formation of lumps and adherance to the grinding barrel and ring. This made cleaning out the Sieb vessel to retain all the sample very difficult.

(2) A 20 g sample dried under infra-red lamps for 35 minutes prior to 4 minutes grinding gave the same result.

(3) A 20 g sample ground for 3 minutes was not quite as badly compacted as for a 4 minute grind but the sample adhered strongly to the barrel bottom and sides.

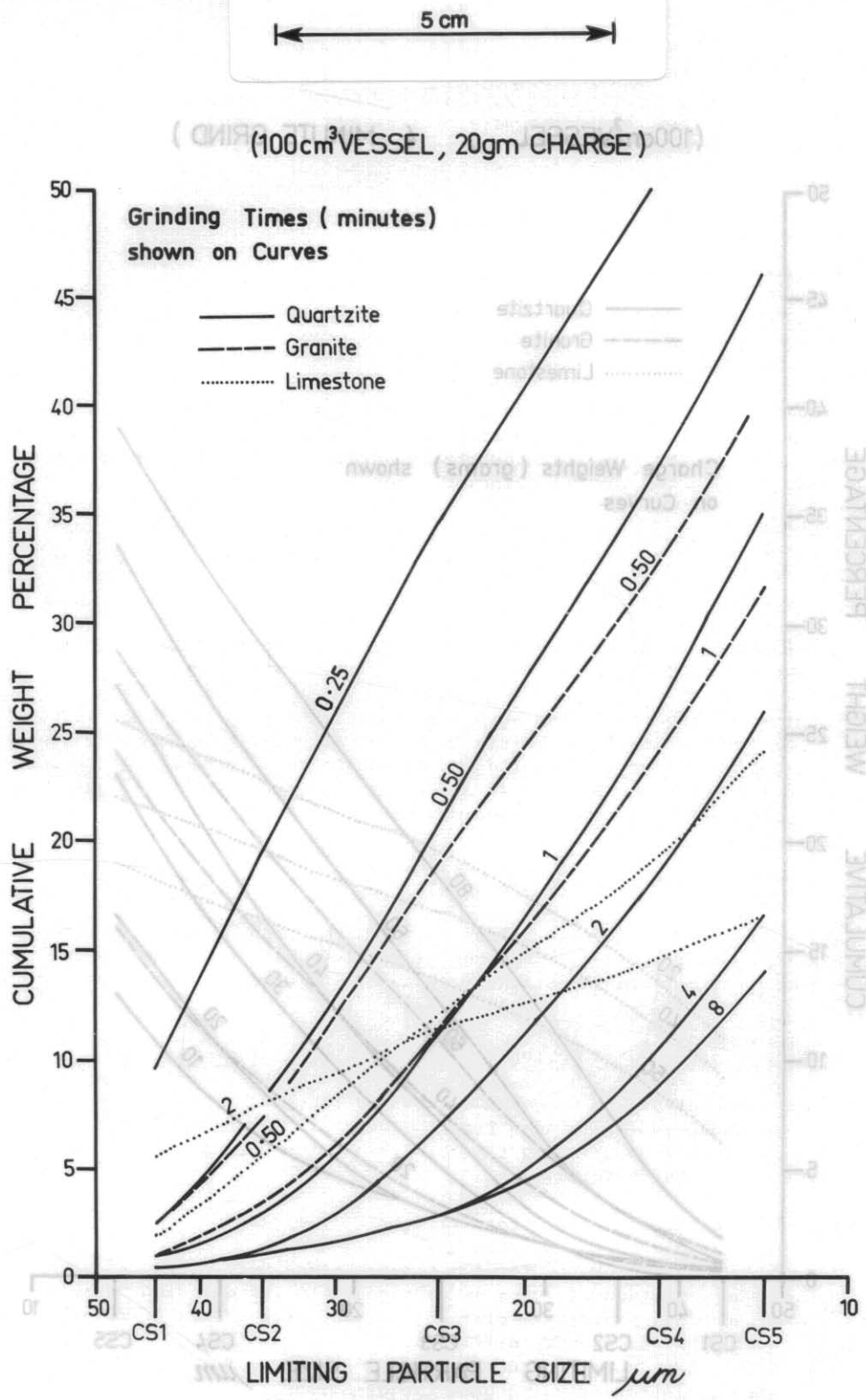


Figure 43. *R.630. Sieb grinding in 100 cm³ vessels with cumulative wt% versus limiting particle size (constant charge).*

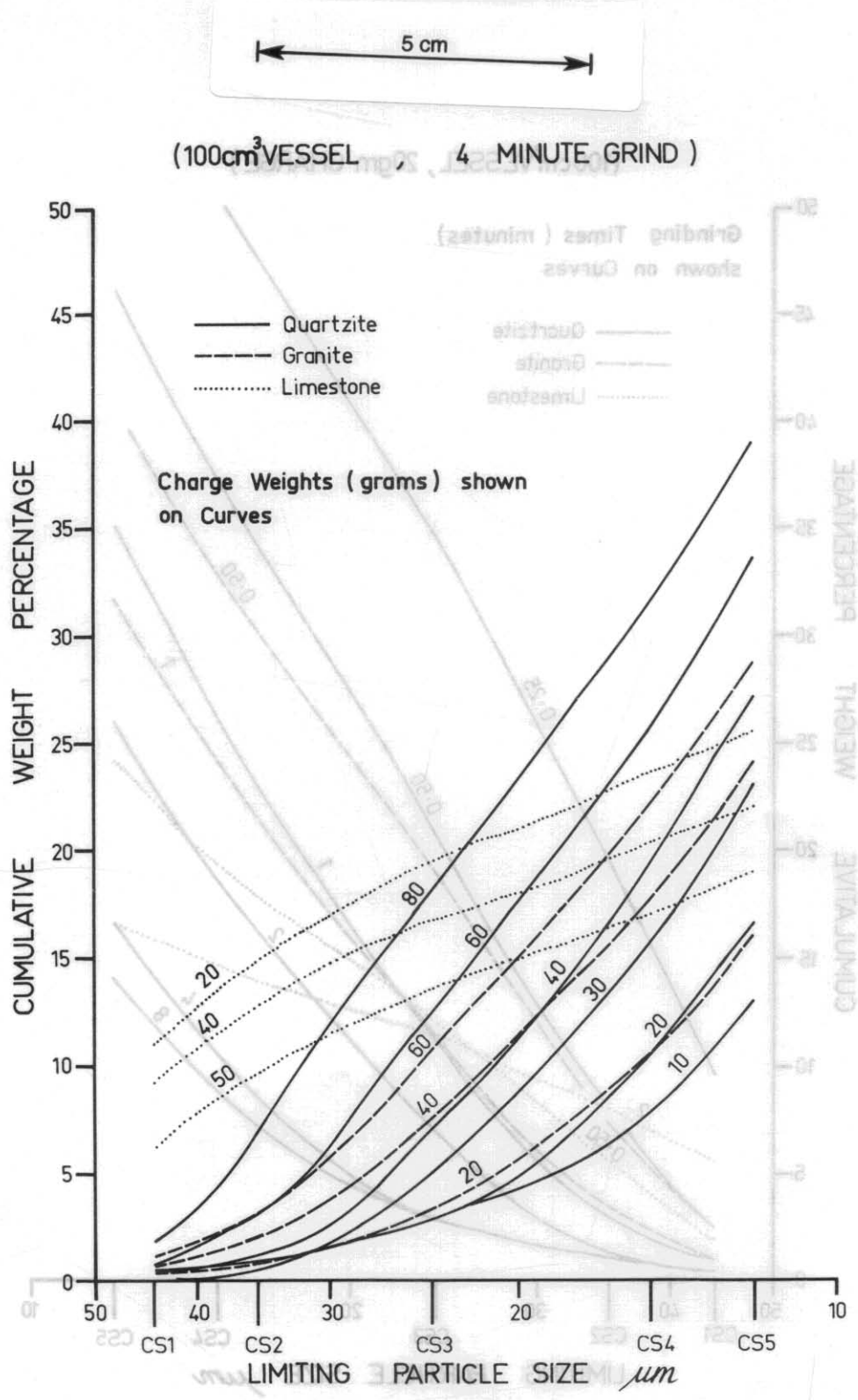


Figure 44. *R.630. Sieb grinding in 100 cm³ vessels with cumulative wt % versus limiting particle size (constant time).*

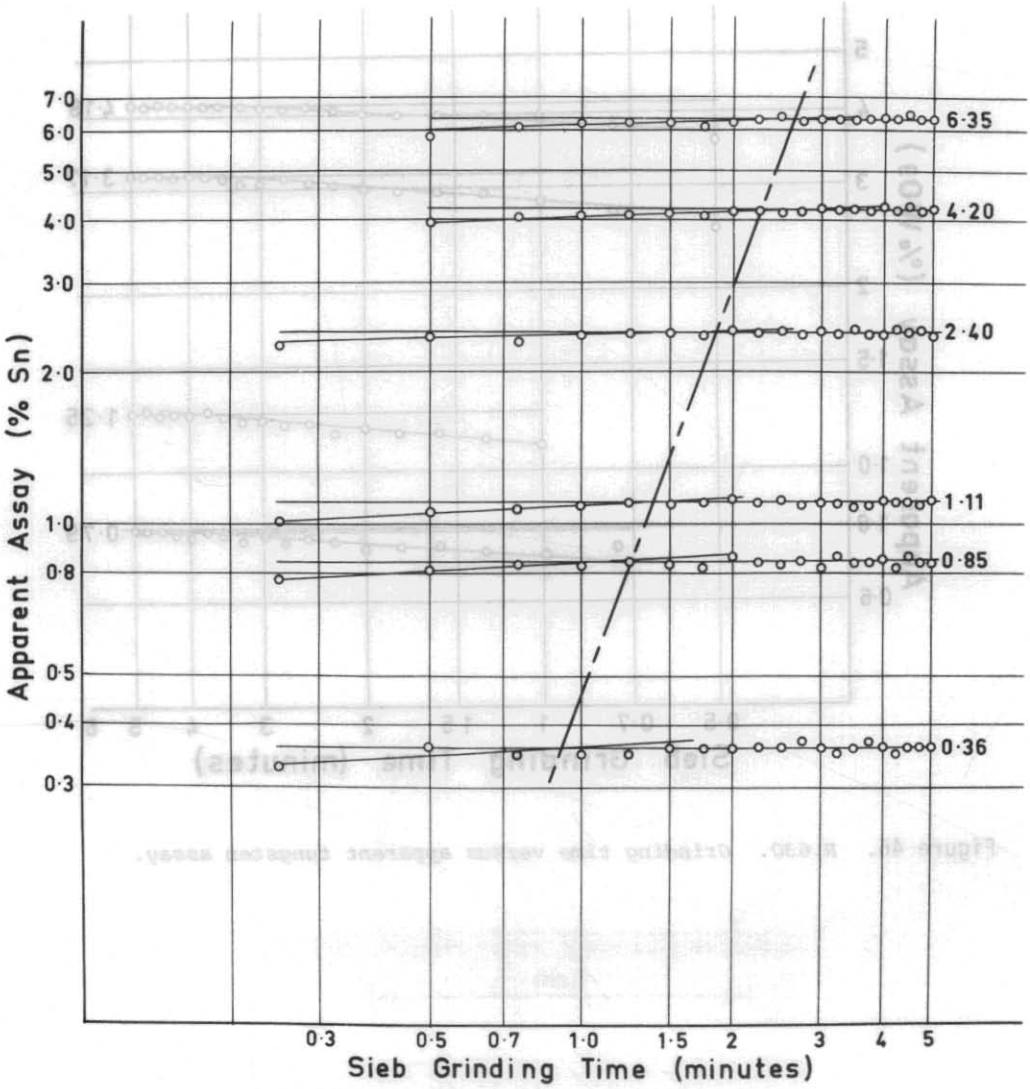


Figure 45. *R.630. Grinding time versus apparent tin assay.*

5 cm

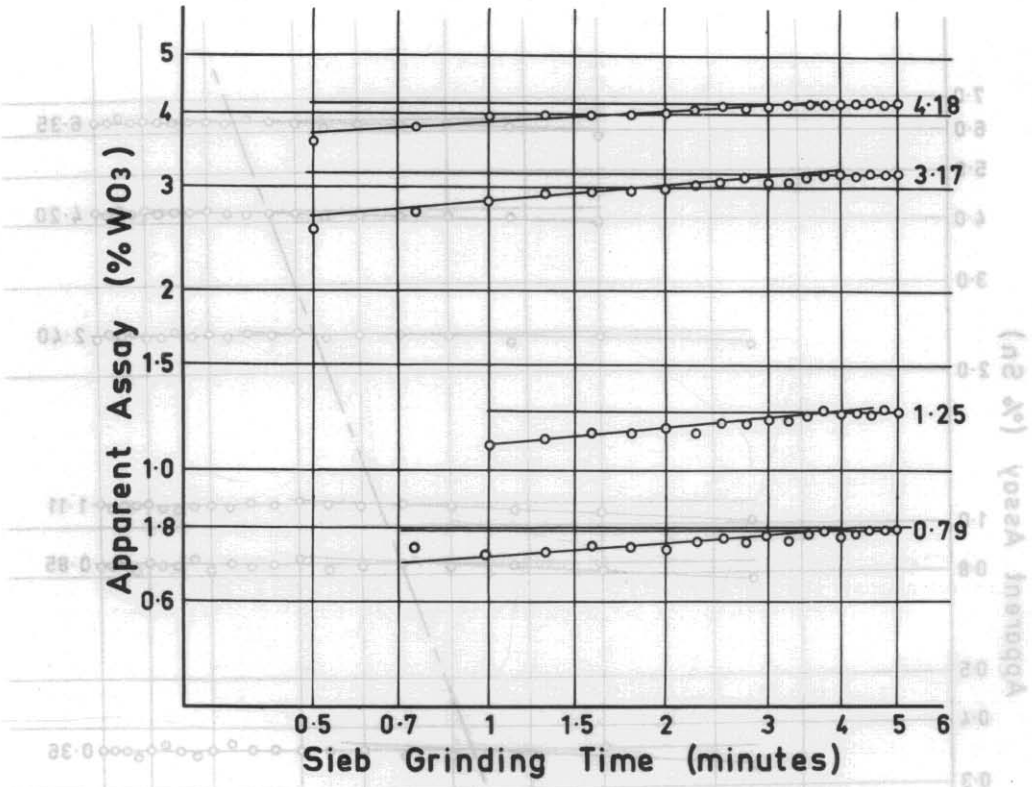
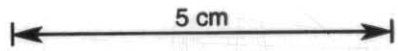


Figure 46. *R.630. Grinding time versus apparent tungsten assay.*



(4) A 20 g sample ground for 2 minutes, was not very lumpy and did not adhere to the grinding barrel or ring and the vessel was relatively easy to clean out.

(5) The results of the 2 and 4 minute grinds were as follows:

Cyclosizer Fractions	*2 minutes (100 cm^3 vessel)*		*4 minutes (100 cm^3 vessel)*	
	% Wt	*% Cum. Wt*	*% Wt*	*% Cum. Wt*
C/S1	Trace	Trace	0.5	0.5
C/S2	0.5	0.5	0.5	1.0
C/S3	3.0	3.5	2.0	3.0
C/S4	10.0	13.5	7.0	10.0
C/S5	7.5	21.0	6.0	16.0
O/F	79.0	100.0	84.0	100.0

CONCLUSION

The three Sieb vessels grind a hard material to a similar short size range product when the following conditions are met.

Vessel	*Charge (g)*	*Grinding time (min.)*
10 cm^3	2	3, 4
100 cm^3	20	3, 4
250 cm^3	50	2, 3

The Sieb feed size is immaterial, as long as it can be correctly placed in a Sieb barrel at a reasonable vessel load. Original particle size is destroyed almost instantaneously. Soft materials like limestone require only a very short grinding time or a large charge weight otherwise agglomeration occurs.

The Sieb grinding mills achieve reasonably quickly, and with little contamination, a product suitable for XRF tin or tungsten analysis. However, the following factors should be considered.

(1) A uniform grinding time of not less than 3.5 minutes is required for all wolfram samples.

(2) Grinding time for tin samples increases as the percentage of tin increases.

(3) Extended grinding times may result in the formation of lumps and a heterogeneous sample which is difficult to clean from the Sieb grinding barrel and ring, giving losses in sample weight.

(4) All samples require at least a one minute Sieb grind to produce a suitable size range for pressing into discs for XRF analysis.

APPENDIX 1

The XRF determinations were carried out on a Philips 1540 instrument, with a molybdenum tube, LiF 200 crystal and a scintillation counter. The $SnK\alpha$ and $WL\alpha$ lines response was measured and the known content of Sn and WO_3 equated with the peak counts. Other counts were expressed as apparent content of Sn and WO_3.