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OF SOUTH AFRICA

ONDERWERP:
SUBJECT: FLUORESCENCE X-RAY SPECTROMETRIC ANALYSES

OF COAL ASH.

AFDELING:
DIVISION: PHYSICS

NAAM VAN AMPTENAAR:
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FUEL RESEARCH INSTITUTE OF SOUTH AFRICA.

REPORT NO. 6 OF 1968.

FLUORESCENCE X-RAY SPECTROMETRIC ANALYSES OF COAL ASH.

INTRODUCTION.

X-ray fluorescence methods are often superior to the conventional wet chemical analysis as regards the time required for the analysis. For some years this method has been successfully applied to the rapid analysis of the heavier elements (with atomic numbers above 22) e.g. in the steel and petroleum industries. The earliest application to the petroleum field, for instance, was the determination of lead and bromine in anti-knock additives in petroleum. However, silicate analysis presents some difficulties in that some of the elements present in silicate samples have atomic numbers below 22. These difficulties are due to the rapidly decreasing intensities of their characteristic lines and due to the increasing absorption in air of their radiations. These have been largely overcome by the development of the vacuum spectrometer and gas-flow proportional counters coupled with pulse-height selection.

The present study was undertaken to establish to what extent the method could be applied to analysis of coal ashes.

As a result of matrix effects it is not possible to establish standardisation curves from synthetic mixtures of the different elemental oxides present in coal ash. Therefore coal samples that had previously been analysed by conventional wet chemical methods were used as standard samples in this study. While samples of the coals were available it was unfortunately not possible to use portions of the ash prepared from them for the wet chemical analysis. It was, therefore, necessary to prepare new ash samples for this study. It is conceivable that, due to sampling and preparation errors these ash samples could differ somewhat in composition with those prepared for the wet chemical analysis. But this had to be accepted under the circumstances. One must therefore bear in mind that any experimental

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errors in the wet chemical analysis will become evident in the standardisation curves. On the other hand, there will also be the interelemental effects from the matrix on the precision of the X-ray determinations, especially if there is a wide range in chemical composition. Another factor that can influence the precision is the particle size and homogeneity of the sample. If the mineral composition differs from sample to sample one can expect a difference in particle size between samples as some minerals are comminuted more rapidly than others.

In conventional wet chemical analysis of coal ash, figures for SiO_2, Al_2O_3, Fe_2O_3, P_2O_5, TiO_2, CaO, MgO, K_2O, Na_2O and SO_3 are reported. With the X-ray apparatus available it is possible only to determine elements with atomic numbers 12 (Mg) and upwards. In other words, it was not possible to determine sodium (atomic number 11). The determination of phosphorus was not possible because the Kα radiation of phosphorus coincides with that of the Kβ (n = 2) of calcium when using the gypsum crystal available. One could overcome this difficulty by using a germanium[10] crystal which has no second order reflections, but this was impossible due to the limited angular range of the available spectrometer (11° to 83° 2θ). The angular location for phosphorus Kα radiation when using a germanium crystal is at about 140° 2θ.

A disadvantage of the fluorescence X-ray method is that one cannot determine ferrous and ferric iron separately.

SAMPLE PREPARATION.

The sample preparation consisted of ashing the minus 60 mesh coal samples according to the standard procedure for wet chemical analysis. The coal ash thus obtained was then further processed according to two different techniques.

(a) Preparing Pressed Powder Specimens.

The ash as such was too coarse for X-ray analysis and was therefore ground to pass a 325 mesh sieve. At first the grinding was done stepwise with intermediate sieving to remove undersize. It was noticed, however, that during sieving some segregation of particles took place. The originally fine and lighter coloured material passed through first and the darker material required longer grinding. As the precision of the determinations can be increased by very fine grinding and the homogeneity of the powder also has a strong influence, it was decided that in future all samples would be ground for 2 hours

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in an automatic agate mortar. To prevent any loss of light material during the grinding process, the sample was kept moist by adding a few ml. of benzol.

The sample thus prepared was pressed into a flat round cake by using a modification of the pressed-specimen die, described by A.K. Baird[3]. The specimen-die was modified in such a manner that the sample could be pressed inside a metal ring (outside diameter 38 mm) which could then easily be transferred to the sample holder of the X-ray spectrometer. Initially, the sample was pressed inside a jacket of powdered bakelite[4], as suggested by Baird, but as it was found that after a time the bakelite particles started to crumble away at the edges and the specimen became bent, it was decided to use the metal ring.

All attempts to press specimens inside the metal ring without any backing material failed. However, even when using backing material the specimens were inclined to crack if the powder contained any lumps. This was rectified by first breaking the lumps in a small agate mortar before pressing. The most suitable material for the backing proved to be powdered bakelite. After long use, these specimens did not show any bending. To provide a smooth surface for the specimen, a glass plate[4] was placed inside the die on which the sample was pressed by applying a force of 10 tons.

The pressed specimens when not in use are stored in a dessicator.

(b) Preparing Fused Discs.

In an attempt to improve the results obtained from the pressed powdered samples, tests were made using different types of mills to reduce the particle size of the samples sufficiently to eliminate the particle size effect on the precision of the measurements. Three types of mills were used:-

1. A Retsch automatic agate mortar.
2. A Siebtechnik vibrator ringmill made of a nickel-iron alloy* or tungsten carbide.
3. A Siebtechnik vibrator ballmill made of agate or tungsten carbide.

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* Colmonoy.

Of the three mills, the ringmill proved the most suitable as far as grinding time was concerned. However, another difficulty arose viz. the contamination of the sample by the grinding medium. For instance, where the nickel-iron ringmill had been used, the iron intensities were higher than in the case where the agate mill had been used and in the case of silica, the intensities were higher where the agate mortar instead of the nickel-iron ringmill had been used.

According to Volborth[5] a dual grinding method can be applied using two different grinding media and performing two complete analyses of each sample. The contamination introduced by each grinding medium can then be determined and the true composition of the sample can be calculated accordingly. This method seems very tedious, especially in the case of coal samples where double the amount of coal ash will have to be prepared.

It was thus decided rather to try the fusion technique as suggested by Claisse[6]. The idea is to dissolve the unground ash sample (thus avoiding contamination by grinding) in a suitable flux and to cast discs which can be used as such in the X-ray apparatus. One can expect a decrease in intensity of the different radiations due to the dilution of the sample. Because this is an important factor in the case of the light elements with their low radiation yield, the following conditions were chosen to promote intensity:-

1. Lithiumtetraborate ($Li_2B_4O_7$) was chosen as a flux because of its low absorption coefficient for light element radiations.
2. The ash powder and flux were mixed in a 1:1 ratio. A higher ash concentration would be impractical because according to Welday[7] et al fusions with more than 40 to 45 per cent rock powder are difficult to make.

To start off, fusions were made in platinum crucibles over a Fisher burner but it was found that on pouring the melt most of it stuck to the sides of the crucibles and not sufficient melt could be obtained to pour a disc large enough for the sample holder of the X-ray apparatus.

The next step was to use pure graphite[7 and 8] instead of platinum crucibles. There are some advantages in using these crucibles, as:-

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1. No cleaning is needed after fusion,
2. a whole batch of crucibles can be heated in one oven at the same time, and
3. the melt can be easily poured from the crucible provided that the correct type of graphite material was chosen for the manufacture of the crucibles.*

The disadvantages are that,

1. some carbon contamination is always present on the surface of the discs,
2. one cannot weigh the discs to establish any weight loss or gain during the fusion (carbon contamination), and
3. according to Norrish and Hutton[9] samples fused in graphite crucibles loose sulphur whether it is present as $SO_4^{=}$ or $S^{=}$.

The method followed for producing the discs was by weighing 2 grams of dry coal ash and 2 grams of $Li_2B_4O_7$ which had been heated at 1000°C in a large graphite crucible to remove water of crystallisation and then finely ground in the Retsch grinder. In later experiments the $Li_2B_4O_7$ was heated overnight at 500°C to obtain the anhydrous form. The two ingredients were then thoroughly mixed inside the crucibles and placed in the oven at 1000°C. A current of nitrogen was passed through the oven to lessen the oxidation of the crucibles. After a time, each crucible was removed from the oven and swirled to aid the mixing of the ingredients and to remove any gas bubbles and was then returned to the oven. After about another 30 minutes the melt was poured onto a $\frac{1}{2}$ inch polished aluminium slab placed on top of a hotplate. The aluminium slab was provided with a thermocouple and its temperature was kept at about 470°C (Claisse[6] used a hotplate with a polished aluminium top). Immediately after pouring, an aluminium plunger was used to press the still molten material into a disc. This aluminium plunger was always placed on the aluminium slab to keep it at the temperature of the slab. This prevents the melt from cooling down too quickly when being pressed. The discs were then left on the aluminium slab for about 10 minutes and then allowed to cool on an asbestos slab which had been heated on the hotplate. This method of annealing was used to avoid cracking of the discs.

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* Obtainable from Morganite S.A. grade EY9 and CY9 and from Industrial Carbon and Graphite Co. grade S.O.

The one side of the discs were then smoothly ground on a glass plate with carborundum powder and the smooth sides were exposed to the X-ray beam when determining the intensities of the different radiations.

OPERATING CONDITIONS OF THE SPECTROMETER.

As elements with atomic numbers falling within the range of 12 to 22 had to be determined, it was necessary to work under a vacuum of about 0.02 mm mercury. One must therefore maintain this vacuum all the time during measurements, since the radiations of these elements are strongly absorbed in air.

Two analyser crystals viz. gypsum and lithium fluoride were provided with the instrument. The gypsum crystal covers the range of elements with atomic numbers 12 to 22 (i.e. magnesium to titanium) and LiF from 22 upwards. In other words, one has only to change over to the LiF crystal for the iron determination.

For the primary radiation a high power chromium X-ray tube and as detector a flow proportional counter (flowrate 5 NL/h.) were used.

After having obtained a vacuum of 0.02 mm mercury in the system and having set the right analyser crystal in position, the corresponding goniometer angle was set for the desired element. This peak position was previously determined by a stepwise recording of the intensity in the region in which the peak occurs. This position of maximum intensity is set for all subsequent determinations of the element. After other instrumental settings had been selected, e.g. for the pulse height analyser, a measurement of the time required to accumulate a set number of counts (which depends on the element being measured), were taken. Magnesium, for instance, has a low radiation yield and to use a reasonable time for a determination a lower preset number of counts will have to be selected than in the case of say, calcium. In X-ray spectography one can always expect a basic counting error originating from the random pulses received from the detector. This counting error also depends on the signal-to-noise ratio and one can thus select a counting precision within the limits of the signal-to-noise ratio and total signal to time allowed for the analyses. Thus, one must be satisfied with a higher counting error in the case of magnesium than in the case of calcium.

A background count was then taken some distance away from the peak position. In the present case the reading of the background count was taken after a definite time interval.

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Having converted the counting times to counts per minute and having subtracted the background count from the peak intensity of the element, the net peak intensity yield in counts per minute was plotted against the weight per cent of the element oxide as obtained by the wet chemical analyses. Each point on the curve represents an average of a number of repetitions of an X-ray determination of the same sample. Values of unknowns can thus be obtained by interpolation from this standardisation curve.

The operating conditions for the different elements are given in Table 1.

When adjusting the pulse height analyser for maximum pulse rate for a certain element one can either keep the window (i.e. baseline and channel) setting in a fixed position and vary the detector voltage or vice versa. In the present case the first mentioned method was applied. During periods of operation a drift of a few volts of the detector voltage occurred and to compensate for this, the voltage setting was adjusted from time to time. At the same time the intensity was checked and set if necessary by making small adjustments to the slit, which was provided with a vernier in front of the counter window. For this purpose, sample 62/712B was always used as a reference. In the case of magnesium it was necessary to use a pure magnesium oxide pressed powder specimen.

For better stability of the flow counter its temperature was kept constant by supplying it with a constant temperature plate through which water at 20°C was circulated.

THE STANDARDISATION CURVES.

The standardisation curves, obtained by the method of least squares for the different elements, are given in Figures 1 to 8 for the pressed powder specimens and Figures 9 to 15 for the fused discs. Where there are large deviations in the curves, these can be caused by matrix influences, particle size influences (in the case of the pressed powder specimens), possible experimental errors in the wet chemical analysis or sampling errors resulting from the procedure adopted.*

As was stated previously, the X-ray analysis figures were obtained by a number of repetitions on only one pressed or one fused specimen of each ash sample. To indicate the precision of these figures, the relative deviation and relative error of the repetitions are given in Tables 2, 3 and 4. It would have been better to compare the reproducibility of the sample preparation

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* (c.f. last sentences of page 1.)

technique with that of the reproducibility of the counting method. In other words, it would have been preferable to prepare, say 5 pressed powder specimens or 5 fused discs of the same sample and to compare the results of their measurements with 5 measurements, one pressed powder specimen or one fused disc of the series. Due to the lack of sufficient sample this was not possible.

An attempt was made to overcome this difficulty by preparing synthetic samples by mixing some chemically pure ingredients in the correct proportions. The fusion method was tried on these samples to overcome the particle size effect which would be present, due to the different particle sizes of the different ingredients. It was found that these synthetic samples were difficult to dissolve and broke very easily on casting discs. Although the samples were mixed for two hours in the automatic agate mortar, one was never certain whether they were really homogeneous.

Another factor which could influence the results in the case of the fused discs is that they could not be weighed to record any weight loss or gain resulting from fusion at 1000°C. The reason for this is, as already stated, that the samples were fused in carbon crucibles. This can be overcome by fusing in platinum-gold alloy crucibles* which pour cleanly so that one can weigh the discs afterwards and make corrections to the intensity measurements for any weight change.

In Tables 5 and 6, a comparison is made between the chemical analysis figures and those of the X-ray fluorescence figures obtained by interpolation from the standardisation curves (figures 1 - 8 and 9 - 15). As shall be noted, in the case of samples 5639 and 62/2127A, two fused discs were prepared of each sample. Both were measured but only one determination was used, but always of the same disc with the best surface for the interpolation of the X-ray fluorescence figures.

As the P_2O_5 and Na_2O contents could not be determined by the X-ray method, the figures of the chemical analyses were used for the summation of all the components. This also had to be done in the case of SO_3 where fused discs were prepared, due to the loss of sulphur during fusion. In Table 6 a comparison is also made between the X-ray results obtained by means of the fused discs (X-ray 1) and the pressed powder specimens (X-ray 2) in the case of the first 7 samples. The fused disc preparation method shows some definite improvement over the

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* Obtainable from Mathey & Johnson (3% Gold).

pressed powder specimens, but again due to insufficient samples a comparison could not be made in all the cases where pressed powder specimens had previously been measured.

The table also includes the sum of those oxides which were precipitated by ammonium-hydroxide. The differences between the figures of the two methods of analyses are given, as well as the differences between the two summations, viz. the total oxides and the oxides precipitated by ammonium-hydroxide.

CONCLUSION.

The results of this study indicate that the X-ray fluorescence method could be applied to the routine analysis of coal ash. Although the method is perhaps not as accurate as the conventional wet chemical method, one cannot ignore the time saving that can be achieved by applying this method. It takes about one day to determine 8 elements, repeating each determination 3 times. This does not include the time used for sample preparation. Due to a number of uncertainties one must perhaps be satisfied with a total sum of the element oxides of 100 $\pm$ 2%. Perhaps this can be improved by using the platinum-gold alloy crucibles as mentioned earlier and instead of lithiumtetraborate as flux, the Spectro fluxes (obtainable from Mathey & Johnson) having lower melting points than lithiumtetraborate.

PRETORIA.
23rd January, 1968.
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J.H. COPEMAN
Research Officer.

W.T.E. VON WOLFF.
Chief Research Officer.

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TABLE 1.

OPERATING CONDITIONS FOR FLUORESCENCE X-RAY SPECTROGRAPHIC ANALYSES.

Element	Crystal	Pulse Height Analyser		Detector Voltage Approx.	Tube-Rating		Soller Slit	Attenuation	Line	Peak Location	Background Location	Preset Peak Counts	Intensity*** Adjustment to 62/712B in counts/min.
		Baseline V	Channel V	V	KV	mA	degrees			2θ	2θ		
Mg	gypsum	11	8.8	1950	45	30	0.4	X40	Kα	81° 12.5'	79° 0'	*	55600**
Al	gypsum	11	8.8	1920	45	30	0.4	X40	Kα	66° 32'	67° 30'	$2x10^5$	63800
Si	gypsum	11	8.8	1905	35	30	0.4	X40	Kα	55° 52'	54° 30'	$4x10^5$	87000
S	gypsum	12	8.4	1890	45	30	0.4	X40	Kα	41° 21'	40° 30'	$1x10^5$	53500
K	gypsum	12	7.2	1845	45	30	0.4	X40	Kα	28° 27.5'	27° 30'	$2x10^5$	59000
Ca	gypsum	12	6.0	1935	20	6	0.4	X100	Kα	25° 32'	24° 30'	$2x10^5$	178500
Ti	gypsum	12	7.2	1920	35	30	0.4	X100	Kα	20° 49'	22° 0'	$4x10^5$	151000
Fe	LiF	12	5.4	1875	20	20	0.4	X100	Kα	57° 32'	56° 30'	$4x10^5$	248300

* In the case of magnesium a preset time of 10 minutes was used for the peak as well as the background count and for all other elements a preset time of 2 minutes for the background.

** In the case of magnesium a pure magnesium oxide pressed powder specimen was used.

*** Intensity adjustment done by means of the adjustable slit in front of the flow counter.

TABLE 2.*

RANGE OF COMPONENT CONCENTRATION AND RANGE OF RELATIVE DEVIATION.

Components	Range of Concentration, %		Range of Relative Deviation,** %	
	Pressed Powder Specimens	Fused Discs	Pressed Powder Specimens	Fused Discs
MgO	0.54 - 4.28	0.93 - 3.74	0.12 - 7.83	0.33 - 2.60
Al_2O_3	20.00 - 38.60	19.4 - 42.1	0.10 - 2.60	0.03 - 0.37
SiO_2	32.80 - 68.20	25.0 - 56.7	0.05 - 1.64	0.03 - 0.41
SO_3	0.47 - 6.60	- - -	0.15 - 1.87	- - -
K_2O	0.30 - 2.78	0.23 - 2.78	0.02 - 2.05	0.10 - 0.41
CaO	0.9 - 15.3	1.1 - 20.7	0.06 - 0.72	0.10 - 0.67
TiO_2	1.03 - 2.81	0.76 - 2.81	0.05 - 1.72	0.07 - 0.78
Fe_2O_3	2.9 - 14.7	3.1 - 22.3	0.003 - 1.26	0.07 - 0.29

* See also Tables 3 and 4.

** Relative Deviation is the standard deviation of a single measurement.

TABLE 4 (Continued)

Sample No.	No. of Determinations	Counting Method: Preset Count	Counting Method: Preset Time in min.	Average Count per Minute	Relative: Deviation *	Relative: Error **	Chem. Anal. %
		SiO_2					
65/1585B	3	$4x10^5$		78583	0.07	0.04	43.8
65/1551A	3	$4x10^5$		77538	0.34	0.19	42.1
65/1551B	3	$4x10^5$		96026	0.07	0.04	50.6
63/814C	3	$4x10^5$		85451	0.03	0.02	46.5
63/931D	4	$4x10^5$		74675	0.39	0.20	41.3
62/584B	3	$4x10^5$		94404	0.05	0.03	52.2
5639 (a	3	$4x10^5$		100647	0.17	0.10	56.7
(b		$4x10^5$		103903	0.08	0.04	-
62/585B	3	$4x10^5$		105851	0.21	0.12	56.1
63/461D	4	$4x10^5$		77491	0.14	0.07	43.9
63/584D	4	$4x10^5$		97327	0.11	0.05	53.0
65/1551C	4	$4x10^5$		92639	0.19	0.09	50.7
65/1880C	4	$4x10^5$		64286	0.15	0.08	36.6
65/1886C	4	$4x10^5$		82289	0.14	0.07	46.0
63/1399C	4	$4x10^5$		78208	0.19	0.10	43.8
65/2127A (a	4	$4x10^5$		62191	0.18	0.09	32.6
(b	2	$4x10^5$		61786	0.21	0.10	-
65/210B	4	$4x10^5$		76244	0.12	0.06	41.1
64/390B6	4	$4x10^5$		46214	0.13	0.07	25.0
11/63 7/8"x⅜"	4	$4x10^5$		70502	0.14	0.07	41.0
62/1168A	4	$4x10^5$		88095	0.18	0.09	49.1
65/1585A	4	$4x10^5$		70771	0.15	0.08	44.0
11/63 6"x7/8"	4	$4x10^5$		62879	0.41	0.21	36.6
		K_2O					
65/1585B	4	$2x10^5$		62379	0.41	0.21	0.45
65/1551A	4	$2x10^5$		35652	0.27	0.13	0.30
65/1551B	4	$2x10^5$		59600	0.13	0.06	0.50
63/814C	4	$2x10^5$		196634	0.15	0.08	1.78
63/931D	4	$2x10^5$		54170	0.13	0.07	0.46
62/584B	4	$2x10^5$		112136	0.15	0.09	0.99
5639 (a	4	$2x10^5$		297440	0.40	0.20	2.78
(b		$2x10^5$		296805	0.17	0.08	-
62/585B	4	$2x10^5$		127190	0.19	0.10	1.12
63/461D	4	$2x10^5$		59922	0.30	0.15	0.46
63/584D	4	$2x10^5$		121841	0.20	0.10	1.11
65/1551C	4	$2x10^5$		43922	0.22	0.11	0.40
65/1880C	4	$2x10^5$		48905	0.10	0.06	0.41
65/1886C	4	$2x10^5$		48345	0.27	0.16	0.38
63/1399C	4	$2x10^5$		51078	0.18	0.10	0.41
65/2127A (a	3	$2x10^5$		101613	0.09	0.05	0.64
(b	4	$2x10^5$		102646	0.18	0.09	-
65/210B	3	$2x10^5$		135521	0.15	0.09	1.14
64/390B6	4	$2x10^5$		29268	0.24	0.12	0.23
11/63 7/8"x⅜"	5	$2x10^5$		63589	0.13	0.06	0.55
62/1168A	4	$2x10^5$		170166	0.26	0.13	1.60
65/1585A	4	$2x10^5$		88701	0.41	0.20	0.75
11/63 6"x7/8"	4	$2x10^5$		56926	0.19	0.09	0.47

* Relative Deviation is the standard deviation of a single determination
** Relative Error is the standard deviation of the arithmetic mean.

CaO...../

TABLE 4 (Continued)

Sample No.	No. of Determinations	Counting Method: Preset Count	Counting Method: Preset Time in min.	Average Count per Minute	Relative: Deviation *	Relative: Error **	Chem. Anal. %
		CaO					
65/1585B	4	$2x10^5$		111732	0.17	0.08	11.6
65/1551A	4	$2x10^5$		115693	0.30	0.15	11.1
65/1551B	4	$2x10^5$		94157	0.27	0.13	9.3
63/814C	4	$2x10^5$		71581	0.25	0.12	7.2
63/931D	4	$2x10^5$		125675	0.11	0.05	12.5
62/584B	4	$2x10^5$		36351	0.23	0.11	3.5
5639 (a	3	$2x10^5$		11976	0.31	0.18	1.1
5639 (b	3	$2x10^5$		10835	0.63	0.36	-
62/585B	4	$2x10^5$		31597	0.34	0.17	3.1
63/461D	3	$2x10^5$		52740	0.37	0.21	4.9
63/584D	3	$2x10^5$		47257	0.10	0.06	4.5
65/1551C	4	$2x10^5$		92965	0.29	0.15	9.0
65/1880C	3	$2x10^5$		126891	-	-	12.3
65/1886C	3	$2x10^5$		98444	0.25	0.14	9.5
63/1399C	3	$2x10^5$		75224	0.30	0.17	7.3
65/2127A (a	3	$2x10^5$		155093	0.28	0.16	15.6
65/2127A (b	4	$2x10^5$		155150	0.40	0.20	-
65/210B	3	$2x10^5$		93992	0.44	0.25	9.6
64/390B6	4	$2x10^5$		223450	0.67	0.34	20.7
11/63 7/8"x3/8"	4	$2x10^5$		69289	0.13	0.07	7.1
62/1168A	3	$2x10^5$		27407	0.14	0.08	2.8
65/1585A	4	$2x10^5$		137697	0.14	0.07	10.9
11/63 6"x7/8"	4	$2x10^5$		87182	0.20	0.10	8.9
		TiO_2					
65/1585B	3	$4x10^5$		141053	0.08	0.05	1.48
65/1551A	3	$4x10^5$		118261	0.11	0.06	1.28
65/1551B	3	$4x10^5$		136315	0.20	0.12	1.44
63/814C	3	$4x10^5$		141021	0.04	0.02	1.54
63/931D	3	$4x10^5$		95285	0.12	0.07	1.03
62/584B	4	$4x10^5$		162612	0.19	0.09	1.64
5639 (a	4	$4x10^5$		161979	0.17	0.10	1.61
5639 (b	4	$4x10^5$		159914	0.78	0.39	-
62/585B	4	$4x10^5$		145524	0.07	0.03	2.11
63/461D	4	$4x10^5$		267503	0.19	0.10	2.81
63/584D	4	$4x10^5$		205982	0.14	0.07	2.11
65/1551C	4	$4x10^5$		124904	0.07	0.03	1.34
65/1880C	3	$4x10^5$		152265	0.08	0.05	1.64
65/1886C	3	$4x10^5$		165993	0.17	0.10	1.80
63/1399C	3	$4x10^5$		174978	0.22	0.13	1.84
65/2127A (a	3	$4x10^5$		148230	0.12	0.07	1.66
65/2127A (b	2	$4x10^5$		146771	0.16	0.11	-
65/210B	3	$4x10^5$		131887	0.12	0.07	1.38
64/390B6	4	$4x10^5$		61954	0.16	0.08	0.76
11/63 7/8"x3/8"	8	$4x10^5$		164751	0.50	0.18	1.65
62/1168A	4	$4x10^5$		154264	0.25	0.12	1.42
65/1585A	4	$4x10^5$		144287	0.14	0.07	1.51
11/63 6"x7/8"	4	$4x10^5$		132289	0.12	0.06	1.41

* Relative Deviation is the standard deviation of a single determination
** Relative Error is the standard deviation of the arithmetic mean.

Fe_2O_3...../

TABLE 4 (Continued)

Sample No.	No. of Determinations	Counting Method: Preset Count	Counting Method: Preset Time in min.	Average Count per Minute	Relative: Deviation *	Relative: Error **	Chem. Anal. %
	Fe_2O_3						
65/1585A	4	$4x10^4$		170038	0.14	0.07	5.2
65/1551A	4	$4x10^4$		444496	0.25	0.12	13.6
65/1551B	4	$4x10^4$		290072	0.15	0.08	8.6
63/814C	4	$4x10^4$		294242	0.22	0.11	9.3
63/931D	4	$4x10^4$		319714	0.18	0.09	10.3
62/584B	4	$4x10^4$		473230	0.20	0.10	14.4
5639 (a	4	$4x10^4$		235458	0.11	0.06	6.5
5639 (b	4	$4x10^4$		231832	0.07	0.04	
62/585B	4	$4x10^4$		418806	0.18	0.09	12.5
63/461D	4	$4x10^4$		178729	0.14	0.07	5.3
63/584D	4	$4x10^4$		296101	0.11	0.06	8.7
65/1551C	4	$4x10^4$		223494	0.21	0.10	6.4
65/1880C	3	$4x10^4$		150793	0.13	0.08	4.5
65/1886C	3	$4x10^4$		107907	0.09	0.06	3.3
63/1399C	3	$4x10^4$		55990	0.25	0.15	1.5
65/2127A (a	3	$4x10^4$		127029	0.07	0.04	4.3
65/2127A (b	4	$4x10^4$		136964	0.10	0.05	
65/210B	3	$4x10^4$		366722	0.09	0.05	11.5
64/390B6	4	$4x10^4$		553804	0.29	0.15	22.3
11/63 7/8"x⅜"	4	$4x10^4$		237129	0.19	0.09	3.1
62/1168A	4	$4x10^4$		345769	0.08	0.04	11.0
65/1585A	4	$4x10^4$		159273	0.21	0.10	5.2
11/63 6"x7/8"	4	$4x10^4$		384001	0.22	0.11	6.5

* Relative Deviation is the standard deviation of a single determination.
** Relative Error is the standard deviation of the arithmetic mean.

Table 5...../

TABLE 3 (Continued)

Sample No.		No. of Determi-nations	Counting Method		Average Count per Minute
-325 Mesh	Ground for 2 hours		Preset Count	Preset time in min.	
			Al_2O_3		
62/712B		5	$2x10^5$		62747
		11		2	62825*)
62/1194A		5	$2x10^5$		75252
		10		2	73360*)
62/1261F		5	$2x10^5$		70245
		10		2	68098*)
63/723C		5	$2x10^5$		74743
		10		2	72553*)
5647		5	$2x10^5$		65687
		5	$2x10^5$		66775*)
	62/712D	5	$2x10^5$		54928
		5	$2x10^5$		55106*)
	63/879C	5	$2x10^5$		58876
		7	$2x10^5$		59332*)
	63/930D	5	$2x10^5$		51014
		7	$2x10^5$		52085*)
	5639	5	$2x10^5$		58730
		6	$2x10^5$		59176*)
	63/931D	5	$2x10^5$		50858
		6	$2x10^5$		51851*)
	65/1551A	5	$2x10^5$		38482
	65/1551B	5	$2x10^5$		42981
		5	$2x10^5$		41053
	65/1585A	5	$2x10^5$		55783
		4	$2x10^5$		55549
	62/585B	5	$2x10^5$		45114
	62/585D	5	$2x10^5$		44078
	64/1178A	4	$2x10^5$		62330
	64/741A	5	$2x10^5$		55015
	63/559B	5	$2x10^5$		45114
	63/559D	5	$2x10^5$		42945
	63/461D	5	$2x10^5$		69310
	64/761C	5	$2x10^5$		72580
	64/750C	5	$2x10^5$		56923
	62/748B	5	$2x10^5$		61743

* Relative Deviation is the standard deviation of a s

** Relative Error is the standard deviation of the ari

*) Previous measurements (see page 13).

TABLE 3 (Continued)

Sample No.		No. of Determinations	Counting Method		Average Count per Minute	Relative		Chem. Anal. %
-325 Mesh	Ground for 2 hours		Preset Count	Preset time in min.		Deviation *	Error **	
		Al_2O_3						
62/712B		5	$2x10^5$		62747	0.13	0.06	31.1
		11		2	62825*)	0.19	0.06	-
62/1194A		5	$2x10^5$		75252	2.60	1.16	35.7
		10		2	73360*)	0.33	0.10	-
62/1261F		5	$2x10^5$		70245	0.16	0.07	34.5
		10		2	68098*)	0.43	0.14	-
63/723C		5	$2x10^5$		74743	0.25	0.11	38.4
		10		2	72553*)	0.44	0.14	-
5647		5	$2x10^5$		65687	0.18	0.08	32.3
		5	$2x10^5$		66775*)	0.57	0.26	-
	62/712D	5	$2x10^5$		54928	0.25	0.11	24.6
		5	$2x10^5$		55106*)	0.47	0.21	-
	63/879C	5	$2x10^5$		58876	0.30	0.13	30.2
		7	$2x10^5$		59332*)	0.32	0.12	-
	63/930D	5	$2x10^5$		51014	0.19	0.09	27.4
		7	$2x10^5$		52085*)	0.26	0.10	-
	5639	5	$2x10^5$		58730	1.56	0.70	28.7
		6	$2x10^5$		59176*)	0.53	0.22	-
	63/931D	5	$2x10^5$		50858	0.10	0.04	26.8
		6	$2x10^5$		51851*)	0.62	0.25	-
	65/1551A	5	$2x10^5$		38482	0.13	0.06	20.00
	65/1551B	5	$2x10^5$		42981	0.18	0.08	20.10
		5	$2x10^5$		41053	0.17	0.08	-
	65/1585A	5	$2x10^5$		55783	0.18	0.08	28.10
		4	$2x10^5$		55549	0.21	0.10	-
	62/585B	5	$2x10^5$		45114	0.13	0.06	21.70
	62/585D	5	$2x10^5$		44078	0.28	0.13	21.00
	64/1178A	4	$2x10^5$		62330	0.14	0.07	30.90
	64/741A	5	$2x10^5$		55015	0.12	0.06	27.50
	63/559B	5	$2x10^5$		45114	0.13	0.06	21.40
	63/559D	5	$2x10^5$		42945	0.20	0.09	21.00
	63/461D	5	$2x10^5$		69310	0.29	0.13	36.60
	64/761C	5	$2x10^5$		72580	0.42	0.19	38.60
	64/750C	5	$2x10^5$		56923	0.31	0.14	30.20
	62/748B	5	$2x10^5$		61743	0.20	0.09	34.60

* Relative Deviation is the standard deviation of a single determination.

** Relative Error is the standard deviation of the arithmetic mean.

*) Previous measurements (see page 13).

SiO_2...../

TABLE 3.

REPRODUCIBILITY OF COUNTING.

(Pressed Powder Specimens).

Sample No.		No. of Determinations	Counting Method		Average Count per Minute	Relative		Chem. Anal. %
-325 Mesh	Ground for 2 hours		Preset Count	Preset time in min.		Deviation *	Error **	
		MgO						
62/712B		3		10	762	0.87	0.50	1.80
		8		10	855*)	0.24	0.09	-
62/1194A		2		10	291	-	-	1.29
		7		10	312*)	6.39	2.41	-
62/1261F		2		10	706	-	-	1.77
		10		10	756*)	1.46	0.46	-
63/723C		3		10	1139	0.15	0.09	-
		8		10	1217*)	0.98	0.35	2.71
5647		2		10	593	-	-	-
		5		10	611*)	1.17	0.52	1.47
	62/712D	2		10	455	-	-	-
		5		10	459*)	1.73	0.77	1.00
	63/879C	2		10	300	-	-	-
		5		10	337*)	1.80	0.81	0.86
	63/930D	2		10	466	-	-	-
		4		10	479*)	3.59	1.80	1.14
	5639	2		10	346	-	-	-
		5		10	361*)	2.03	0.91	0.93
	63/931D	2		10	1008	-	-	-
		5		10	1011*)	0.96	0.43	2.4
	65/1551A	2		10	1073	-	-	2.58
	65/1551B	3		10	1008	1.16	0.67	2.97
		2		10	985	-	-	-
	65/1585A	2		10	1534	-	-	4.28
		2		10	1610	-	-	-
	62/585B	2		10	569	-	-	1.42
	62/585D	2		10	647	-	-	1.56
	64/1178A	2		10	579	-	-	1.24
	64/741A	3		10	768	7.83	4.52	1.47
	63/559B	3		10	228	3.11	1.80	0.54
	63/559D	3		10	716	0.12	0.07	1.55
	63/461D	2		10	1277	-	-	2.64
	64/761C	2		10	1145	-	-	2.71
	64/750C	2		10	1172	-	-	2.64
	62/748B	2		10	1341	-	-	3.20

* Relative Deviation is the standard deviation of a single determination.
** Relative Error is the standard deviation of the arithmetic mean.
*) Previous measurements (see J.H. Copeman and W.T.E. von Wolff, Fluorescence X-Ray Spectrometric Analysis of Coal Ash. Technical Memorandum No. 21 of 1965).

Al_2O_3...../

TABLE 3 (Continued)

Sample No.		No. of Determinations	Counting Method		Average Count per Minute	Relative		Chem. Anal. %
-325 Mesh	Ground for 2 hours		Preset Count	Preset time in min.		Deviation *	Error **	
		SiO_2						
62/712B		5	$4x10^5$		85422	0.13	0.06	34.0
		10		2	84970*)	0.30	0.09	-
62/1194A		5	$4x10^5$		99282	0.17	0.07	40.0
		11		2	97421*)	0.26	0.08	-
62/1261F		5	$4x10^5$		98624	0.19	0.08	39.1
		10		2	97550*)	0.27	0.09	-
63/723C		5	$4x10^5$		95804	0.16	0.07	38.0
		10		2	95306*)	0.31	0.10	-
5647		5	$4x10^5$		130959	0.26	0.12	49.1
		6	$4x10^5$		128453*)	0.31	0.13	-
	62/712D	5	$4x10^5$		189145	0.22	0.10	64.6
		6	$4x10^5$		187532*)	0.91	0.37	-
	63/879C	5	$4x10^5$		124898	0.10	0.05	47.5
		9	$4x10^5$		125694*)	0.75	0.25	-
	63/930D	5	$4x10^5$		130105	0.13	0.06	47.0
		6	$4x10^5$		129079*)	0.53	0.22	-
	5639	5	$4x10^5$		150542	0.32	0.15	56.7
		5	$2x10^5$		148609*)	0.80	0.36	-
	63/931D	5	$4x10^5$		109412	0.19	0.09	41.3
		6	$4x10^5$		108565*)	0.33	0.13	-
	65/1551A	5	$4x10^5$		121003	0.21	0.09	42.10
	65/1551B	5	$4x10^5$		151728	0.17	0.08	50.60
		4	$4x10^5$		147724	0.10	0.05	-
	65/1585A	5	$4x10^5$		103692	0.13	0.06	44.00
		5	$4x10^5$		102029	0.15	0.07	-
	62/585B	5	$4x10^5$		169003	0.42	0.19	56.10
	62/585D	5	$4x10^5$		176438	0.20	0.09	58.30
	64/1178A	5	$4x10^5$		151205	1.64	0.73	53.30
	64/741A	5	$4x10^5$		183281	0.05	0.02	61.90
	63/559B	5	$4x10^5$		219259	0.29	0.13	68.20
	63/559D	5	$4x10^5$		196020	0.09	0.04	63.60
	63/461D	5	$4x10^5$		112906	0.20	0.09	43.90
	64/761C	5	$4x10^5$		101715	0.15	0.07	39.90
	64/750C	5	$4x10^5$		125083	0.08	0.04	46.40
	62/748B	5	$4x10^5$		83723	0.09	0.04	32.80

* Relative Deviation is the standard deviation of a single determination.

** Relative Error is the standard deviation of the arithmetic mean.

*) Previous measurements (see page 13).

SO_3...../

TABLE 3 (Continued)

Sample No.		No. of Determinations	Counting Method		Average Count per Minute	Relative		Chem. Anal. %
-325 Mesh	Ground for 2 hours		Preset Count	Preset time in min.		Deviation *	Error **	
			SO_3					
62/712B		4	$1x10^5$		49627	0.17	0.08	3.14
		10		2	49018*)	0.31	0.10	-
62/1194A		4	$1x10^5$		34544	0.34	0.17	2.30
		10		2	35580*)	1.61	0.72	-
62/1261F		4	$1x10^5$		41104	0.48	0.24	2.14
		10		2	42992*)	1.35	0.43	-
63/723C		4	$1x10^5$		52755	0.31	0.15	3.81
		9		2	53011*)	0.36	0.12	-
5647		5	$1x10^5$		16814	0.33	0.15	1.18
		8	$1x10^5$		18154*)	1.87	0.66	-
	62/712D	4	$1x10^5$		12630	0.15	0.08	0.90
		6	$1x10^5$		13922*)	1.79	0.73	-
	63/879C	4	$1x10^5$		11189	0.67	0.33	0.54
		6	$1x10^5$		13062*)	2.02	0.82	-
	63/930D	4	$1x10^5$		35205	0.20	0.10	2.70
		7	$1x10^5$		35675*)	1.24	0.47	-
	5639	4	$1x10^5$		9833	1.40	0.70	0.47
		6	$1x10^5$		11853*)	1.98	0.81	-
	63/931D	4	$1x10^5$		54453	0.86	0.43	3.88
		6	$1x10^5$		53982*)	1.26	0.51	-
	65/1551A	4	$2x10^5$		82797	0.32	0.16	6.60
	65/1551B	4	$1x10^5$		61262	0.24	0.12	5.13
		4	$1x10^5$		61759	0.27	0.13	-
	65/1585A	4	$1x10^5$		49647	0.23	0.12	3.46
	62/585B	4	$1x10^5$		28094	0.18	0.09	2.35
	62/585D	4	$1x10^5$		30803	0.80	0.40	2.36
	64/1178A	4	$1x10^5$		23991	0.56	0.28	1.59
	64/741A	4	$1x10^5$		20684	0.26	0.13	1.41
	63/559B	4	$1x10^5$		8796	0.61	0.30	0.58
	63/559D	4	$1x10^5$		24049	0.42	0.21	2.13
	63/461D	4	$1x10^5$		46989	0.26	0.13	2.83
	64/761C	4	$1x10^5$		35287	0.38	0.19	2.43
	64/750C	5	$1x10^5$		35517	0.80	0.36	2.21
	62/748B	4	$1x10^5$		52603	0.24	0.12	3.54

* Relative Deviation is the standard deviation of a single determination.

** Relative Error is the standard deviation of the arithmetic mean.

*) Previous measurements (see page 13).

K_2O...../

TABLE 3 (Continued)

Sample No.		No. of Determinations	Counting Method		Average Count per Minute	Relative		Chem. Anal. %
-325 Mesh	Ground for 2 hours		Preset Count	Preset time in min.		Deviation *	Error **	
		K_2O						
62/712B		4	$2x10^5$		51895	1.15	0.58	0.34
		6		2	49951*)	0.58	0.24	-
62/1194A		4	$2x10^5$		53569	0.17	0.09	0.33
		6	$2x10^5$		52376*)	0.22	0.09	-
62/1261F		4	$2x10^5$		83729	0.21	0.10	0.55
		7	$2x10^5$		82848*)	0.60	0.23	-
63/723C		4	$2x10^5$		104341	0.26	0.13	0.62
		6	$2x10^5$		102306*)	0.15	0.06	-
5647		4	$4x10^5$		337692	2.05	1.03	2.54
		7	$2x10^5$		326972*)	1.31	0.50	-
	62/712D	4	$4x10^5$		206358	0.23	0.11	1.67
		6		2	200888*)	0.21	0.09	-
	63/879C	4	$4x10^5$		236745	0.08	0.04	1.32
		6	$2x10^5$		233914*)	0.63	0.26	-
	63/930D	4	$2x10^5$		77005	0.22	0.11	0.49
		8	$2x10^5$		75666*)	0.63	0.22	-
	5639	4	$4x10^5$		367489	0.36	0.18	2.78
		6	$2x10^5$		360107*)	0.24	0.10	-
	63/931D	4	$2x10^5$		63734	0.28	0.14	0.46
		6	$2x10^5$		62407*)	0.31	0.13	-
	65/1551A	4	$2x10^5$		40520	0.12	0.06	0.30
	65/1551B	4	$2x10^5$		73017	0.23	0.12	0.50
		4	$2x10^5$		71517	0.10	0.05	-
	65/1585A	4	$2x10^5$		102292	0.22	0.11	0.75
		4	$2x10^5$		102702	0.37	0.19	-
	62/585B	4	$2x10^5$		152592	0.08	0.04	1.12
	62/585D	4	$2x10^5$		151589	0.02	0.01	1.10
	64/1178A	4	$2x10^5$		96948	0.14	0.07	0.70
	64/741A	4	$4x10^5$		208671	0.25	0.13	1.56
	63/559B	4	$2x10^5$		136868	0.22	0.11	0.99
	63/559D	4	$2x10^5$		126617	0.22	0.11	0.91
	63/461D	4	$2x10^5$		69615	0.04	0.02	0.46
	64/761C	4	$2x10^5$		57784	0.16	0.08	0.41
	64/750C	4	$2x10^5$		83837	0.09	0.05	0.64
	62/748B	4	$2x10^5$		47085	0.13	0.07	0.39

* Relative Deviation is the standard deviation of a single determination.

** Relative Error is the standard deviation of the arithmetic mean.

*) Previous measurements (see page 13).

CaO...../

TABLE 3 (Continued)

Sample No.		No. of Determinations	Counting Method		Average Count per Minute	Relative		Chem. Anal. %
-325 Mesh	Ground for 2 hours		Preset Count	Preset time in min.		Deviation *	Error **	
		CaO						
62/712B		4	$4x10^5$		180631	0.19	0.09	15.3
		10		2	176712*)	0.23	0.07	-
62/1194A		4	$2x10^5$		141675	0.24	0.12	11.9
		10		2	138902*)	0.27	0.09	-
62/1261F		4	$2x10^5$		89464	0.12	0.06	7.2
		11		2	87362*)	0.23	0.07	-
63/723C		4	$2x10^5$		131324	0.72	0.36	10.6
		10		2	129282*)	0.28	0.09	-
5647		4	$1x10^5$		21424	0.10	0.05	1.8
		6	$2x10^5$		21595*)	0.27	0.11	-
	62/712D	4	$1x10^5$		15421	0.24	0.12	1.4
		6	$2x10^5$		15734*)	0.29	0.12	-
	63/879C	4	$1x10^5$		16732	0.22	0.11	1.5
		6	$2x10^5$		16965*)	0.57	0.23	-
	63/930D	4	$4x10^5$		94135	0.15	0.08	7.2
		6	$2x10^5$		92357*)	0.59	0.24	-
	5639	4	$1x10^5$		13437	0.54	0.27	1.1
		5	$2x10^5$		13261*)	0.68	0.30	-
	63/931D	4	$2x10^5$		153422	0.30	0.15	12.5
		6	$4x10^5$		151638*)	0.12	0.05	-
	65/1551A	4	$2x10^5$		138077	0.21	0.11	11.1
	65/1551B	4	$2x10^5$		113882	0.12	0.06	9.3
		4	$2x10^5$		118213	0.35	0.17	-
	65/1585A	4	$2x10^5$		167322	0.11	0.05	10.9
		4	$2x10^5$		168154	0.29	0.14	-
	62/585B	4	$1x10^5$		38887	0.31	0.16	3.1
	62/585D	4	$1x10^5$		48127	0.06	0.03	3.9
	64/1178A	4	$1x10^5$		68186	0.39	0.19	5.2
	64/741A	4	$1x10^5$		30889	0.13	0.06	2.5
	63/559B	4	$1x10^5$		10982	0.31	0.16	0.9
	63/559D	4	$1x10^5$		45232	0.12	0.06	3.4
	63/461D	4	$1x10^5$		63375	0.29	0.15	4.9
	64/761C	4	$2x10^5$		108100	0.14	0.07	8.4
	64/750C	4	$2x10^5$		133525	0.16	0.08	10.3
	62/748B	4	$2x10^5$		159339	0.13	0.07	12.4

* Relative Deviation is the standard deviation of a single determination.

** Relative Error is the standard deviation of the arithmetic mean.

*) Previous measurements (see page 13).

TiO_2...../

TABLE 3 (Continued)

Sample No. -325 Mesh	Sample No. Ground for 2 hours	No. of Determinations	Counting Method: Preset Count	Counting Method: Preset time in min.	Average Count per Minute	Relative: Deviation *	Relative: Error **	Chem. Anal. %
		TiO_2						
62/712B		4	$4x10^5$		148943	0.11	0.05	1.32
		10		2	147145*)	0.42	0.13	-
62/1194A		4	$4x10^5$		205926	0.05	0.02	1.83
		10		2	202082*)	0.54	0.17	-
62/1261F		4	$4x10^5$		205707	0.13	0.06	1.69
		10		2	203077*)	0.41	0.13	-
63/723C		4	$4x10^5$		199977	0.07	0.03	1.36
		10		2	195123*)	0.28	0.09	-
5647		4	$4x10^5$		184409	0.23	0.11	1.84
		5	$4x10^5$		185435*)	0.54	0.24	-
	62/712D	4	$4x10^5$		156480	0.25	0.12	1.28
		6	$4x10^5$		162475*)	1.00	0.41	-
	63/879C	4	$4x10^5$		154450	0.17	0.08	1.18
		6	$4x10^5$		154569*)	1.06	0.43	-
	63/930D	4	$4x10^5$		124263	0.27	0.13	1.61
		8	$4x10^5$		126859*)	0.71	0.25	-
	5639	4	$4x10^5$		186228	0.10	0.05	1.03
		6	$4x10^5$		186000*)	1.72	0.70	-
	63/931D	4	$4x10^5$		108607	0.16	0.08	1.61
		6	$4x10^5$		109005*)	0.28	0.11	-
	65/1551A	4	$4x10^5$		130760	0.10	0.05	1.28
	65/1551B	4	$4x10^5$		153432	0.65	0.02	1.44
		4	$4x10^5$		155382	0.21	0.11	-
	65/1585A	4	$4x10^5$		158659	0.09	0.04	1.51
		4	$4x10^5$		161203	0.21	0.11	-
	62/585B	4	$4x10^5$		167374	0.08	0.04	1.39
	62/585D	4	$4x10^5$		257387	0.42	0.21	2.40
	64/1178A	4	$4x10^5$		94576	0.15	0.08	1.59
	64/741A	4	$4x10^5$		142560	0.11	0.06	1.11
	63/559B	4	$4x10^5$		196401	0.14	0.07	1.74
	63/559D	4	$4x10^5$		271886	0.06	0.03	1.79
	63/461D	4	$4x10^5$		305746	0.34	0.17	2.81
	64/761C	4	$4x10^5$		196572	0.16	0.08	1.70
	64/750C	4	$4x10^5$		153867	0.09	0.05	1.28
	62/748B	4	$4x10^5$		152702	0.11	0.06	1.35

* Relative Deviation is the standard deviation of a single determination.

** Relative Error is the standard deviation of the arithmetic mean.

*) Previous measurements (see page 13).

Fe_2O_3...../

TABLE 3 (Continued)

Sample No.		No. of Determinations	Counting Method		Average Count per Minute	Relative		Chem. Anal. %
-325 Mesh	Ground for 2 hours		Preset Count	Preset time in min.		Deviation *	Error **	
		Fe_2O_3						
62/712B		4	$4x10^5$		269480	0.49	0.24	8.3
		10		2	245800*)	0.07	0.02	-
62/1194A		4	$4x10^5$		147347	0.11	0.05	4.3
		10		2	144219*)	0.03	0.01	-
62/1261F		4	$4x10^5$		358272	0.40	0.20	10.8
		10		2	350208*)	0.17	0.05	-
63/723C		4	$4x10^5$		104728	0.15	0.08	3.4
		10		2	105451*)	0.06	0.02	-
5647		4	$4x10^5$		295716	0.33	0.17	8.1
		8	$4x10^5$		296072*)	1.26	0.45	-
	62/712D	4	$4x10^5$		146418	0.14	0.07	3.60
		6	$4x10^5$		146433*)	0.39	0.16	-
	63/879C	4	$4x10^5$		539739	0.003	0.001	14.7
		7	$4x10^5$		542594*)	0.67	0.25	-
	63/930D	4	$4x10^5$		450702	0.04	0.02	12.9
		6	$4x10^5$		449930*)	0.59	0.24	-
	5639	4	$4x10^5$		265820	0.17	0.09	6.5
		6	$4x10^5$		262002*)	0.86	0.35	-
	63/931D	4	$4x10^5$		341051	0.28	0.14	10.3
		6	$4x10^5$		341082*)	0.37	0.15	-
	65/1551A	4	$4x10^5$		472269	0.48	0.24	13.6
	65/1551B	4	$4x10^5$		307461	0.30	0.15	8.6
		4	$4x10^5$		314834	0.004	0.002	-
	65/1585A	4	$4x10^5$		174294	0.65	0.33	5.2
		4	$4x10^5$		172054	0.07	0.04	-
	62/585B	4	$4x10^5$		469426	0.38	0.19	12.5
	62/585D	4	$4x10^5$		320883	0.29	0.14	8.6
	64/1178A	4	$4x10^5$		200407	0.08	0.04	5.0
	64/741A	4	$4x10^5$		121297	0.19	0.09	2.9
	63/559B	4	$4x10^5$		233532	0.11	0.05	5.9
	63/559D	4			218785	0.22	0.11	5.5
	64/461D	4	$4x10^5$		198275	0.23	0.12	5.3
	64/761C	4	$4x10^5$		145849	0.17	0.08	5.2
	64/750C	4	$4x10^5$		199595	0.12	0.06	5.6
	62/748B	4	$4x10^5$		308111	0.88	0.44	9.5

* Relative Deviation is the standard deviation of a single determination.

** Relative Error is the standard deviation of the arithmetic mean.

*) Previous measurements (see page 13).

Table 4...../

TABLE 4.

REPRODUCIBILITY OF COUNTING (FUSED DISCS).

Sample No.	No. of Determinations	Counting Method		Average Count per Minute	Relative		Chem. Anal. %
		Preset Count	Preset Time in min.		Deviation *	Error **	
		MgO					
65/1585B	3		10	873	0.55	0.32	3.74
65/1551A	3		10	582	0.33	0.19	2.85
65/1551B	3		10	589	1.35	0.78	2.97
63/814C	3		10	307	0.90	0.52	1.18
63/931D	3		10	552	0.47	0.27	2.4
62/584B	3		10	347	0.52	0.30	1.44
5639 (a	3		10	200	0.77	0.45	0.93
5639 (b	4		10	186	2.43	1.22	-
62/585B	4		10	331	1.47	0.73	1.42
63/461D	3		10	669	0.89	0.52	2.64
63/584D	4		10	438	2.26	1.13	1.81
65/1551C	3		10	598	1.51	0.87	2.95
65/1880C	3		10	593	0.56	0.32	2.72
65/1886C	3		10	325	2.74	1.58	1.36
63/1399C	3		10	151	1.28	0.74	0.70
65/2127A (a	3		10	678	1.05	0.61	2.71
65/2127A (b	3		10	677	1.33	0.77	-
67/210B	3		10	443	2.60	1.50	2.08
64/390B6	3		10	638	0.36	0.21	3.27
11/63 7/8"x3/8"	3		10	301	0.51	0.29	1.34
62/1168A	3		10	262	0.53	0.30	1.23
65/1585A	3		10	807	1.12	0.64	4.28
11/63 6"x7/8"	4		10	344	0.81	0.40	1.63
		Al_2O_3					
65/1585B	3	$2x10^5$		32247	0.03	0.02	28.8
65/1551A	3	$2x10^5$		23256	0.27	0.16	20.0
65/1551B	3	$2x10^5$		25126	0.34	0.19	20.1
63/814C	3	$2x10^5$		31817	0.23	0.13	28.2
63/931D	3	$2x10^5$		30886	0.15	0.09	26.8
62/584B	3	$2x10^5$		25186	0.05	0.03	22.8
5639 (a	3	$2x10^5$		33334	0.18	0.10	28.7
5639 (b	4	$2x10^5$		34264	0.17	0.08	-
62/585B	4	$2x10^5$		24361	0.27	0.13	21.7
63/461D	3	$2x10^5$		40477	0.07	0.04	36.6
63/584D	5	$2x10^5$		29133	0.20	0.09	26.2
65/1551C	3	$2x10^5$		29370	0.14	0.08	24.8
65/1880C	4	$2x10^5$		40183	0.22	0.11	37.2
65/1886C	4	$2x10^5$		39441	0.23	0.11	34.8
63/1399C	4	$2x10^5$		46027	0.34	0.17	42.1
65/2127A (a	4	$2x10^5$		38926	0.16	0.08	36.6
65/2127A (b	4	$2x10^5$		37689	0.21	0.11	-
65/210B	4	$2x10^5$		30014	0.34	0.17	28.0
64/390B6	3	$2x10^5$		22693	0.27	0.15	19.4
11/63 7/8"x3/8"	4	$2x10^5$		42048	0.25	0.13	41.0
62/1168A	4	$2x10^5$		33648	0.13	0.07	29.5
65/1585A	4	$2x10^5$		32814	0.37	0.19	28.1
11/63 6"x7/8"	4	$2x10^5$		37318	0.16	0.08	39.6

* Relative Deviation is the standard deviation of a single determination.
** Relative Error is the standard deviation of the arithmetic mean.

SiO_2...../

COMPARATIVE NORMAL CHEMICAL AND X-RAY FLUORI

(where a dash is given in the difference columns, the che

Sample No.	62/1194A**			62/712B**			62/	
Components	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X
SiO_2	40.00	39.20	-0.80	34.00	34.00	0.00	39.14	3
Al_2O_3	35.70	37.33	1.63	31.10	31.50	0.40	34.52	3
Fe_2O_3	4.30	4.10	-0.20	8.30	7.40	-0.90	10.80	1
P_2O_5	1.37	1.37	-	3.69	3.69	-	1.44	
TiO_2	1.83	1.84	0.01	1.32	1.32	0.00	1.69	
CaO	11.90	11.10	-0.80	15.30	14.20	-1.10	7.19	
MgO	1.29	0.70	-0.59	1.80	1.90	0.10	1.77	
K_2O	0.33	0.37	0.04	0.34	0.36	0.02	0.55	
Na_2O	0.22	0.22	-	0.19	0.19	-	0.25	
SO_3	2.30	2.42	0.12	3.14	3.40	0.26	2.14	
Total Sum	99.24	98.65	-0.59	99.18	97.96	-1.22	99.49	9
Sum of Al_2O_3, Fe_2O_3, P_2O_5 and TiO_2	43.20	44.64	1.44	44.41	43.91	-0.50	48.45	4

Sample No.	63/930D**			5639**			63/	
Components	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X
SiO_2	47.00	48.70	1.70	56.70	53.80	-2.90	41.30	4
Al_2O_3	27.40	25.75	-1.65	28.70	29.60	0.90	26.80	2
Fe_2O_3	12.90	12.80	-0.10	6.50	7.50	1.00	10.30	
P_2O_5	0.08	0.08	-	0.13	0.13	-	0.09	
TiO_2	1.18	1.11	-0.07	1.61	1.67	0.06	1.03	
CaO	7.20	7.40	0.20	1.10	1.10	0.00	12.50	1
MgO	1.14	1.11	-0.03	0.93	0.83	-0.10	2.40	
K_2O	0.49	0.55	0.06	2.78	2.75	-0.03	0.46	
Na_2O	0.10	0.10	-	0.59	0.59	-	0.60	
SO_3	2.70	2.44	-0.26	0.47	0.73	0.26	3.88	
Total Sum	100.19	100.04	-0.15	99.51	98.70	-0.81	99.36	9
Sum of Al_2O_3, Fe_2O_3, P_2O_5 and TiO_2	41.56	39.74	-1.82	36.94	38.90	1.96	38.22	3

Sample No.	65/1585A			62/585B			62	
Components	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X
SiO_2	44.00	40.80*	-3.20	56.10	58.40	2.30	58.30	6
Al_2O_3	28.10	27.80*	-0.30	21.70	22.50	0.80	21.00	2
Fe_2O_3	5.20	4.90*	-0.30	12.50	13.35	0.85	8.60	
P_2O_5	1.07	1.07	-	0.12	0.12	-	0.17	
TiO_2	1.51	1.43*	-0.08	1.39	1.49	0.10	2.40	
CaO	10.90	13.30*	2.40	3.10	3.10	0.00	3.90	
MgO	4.28	3.69*	-0.59	1.42	1.34	-0.08	1.56	
K_2O	0.75	0.75*	0.00	1.12	1.13	0.01	1.10	
Na_2O	0.61	0.61	-	0.13	0.13	-	0.13	
SO_3	3.46	3.41	-0.05	2.35	1.93	-0.42	2.36	
Total Sum	99.88	97.76	-2.12	99.93	103.49	3.56	99.52	10
Sum of Al_2O_3, Fe_2O_3, P_2O_5 and TiO_2	35.88	35.20	-0.68	35.71	37.46	1.75	32.17	3

* The average of two or more results.

** These samples had been determined earlier and the average of t
(see J.H. Copeman and W.T.E. von Wolff, Fluorescence X-Ray Sp

TABLE 5.

SCENCE ANALYSES OBTAINED FROM PRESSED POWDER SPECIMENS.
mically determined value has been used in the fluorescence column).

1261F**		62/712D**			63/723C**			63/879C**		
-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.
9.20	0.06	64.60	62.60	-2.00	38.00	38.20	0.20	47.50	47.50	0.00
4.75	0.23	24.60	27.60	3.00	38.40	37.05	-1.35	30.20	29.60	-0.60
0.10	-0.70	3.60	4.15	0.55	3.40	2.95	-0.45	14.70	15.40	0.70
1.44	-	0.07	0.07	-	0.41	0.41	-	0.07	0.07	-
1.84	0.15	1.36	1.42	0.06	1.89	1.77	-0.12	1.28	1.38	0.10
7.00	-0.19	1.40	1.25	-0.15	10.60	10.30	-0.30	1.50	1.35	-0.15
1.72	-0.05	1.00	1.07	0.07	2.71	2.76	0.05	0.86	0.75	-0.11
0.60	0.05	1.67	1.52	-0.15	0.62	0.76	0.14	1.32	1.76	0.44
0.25	-	0.17	0.17	-	0.35	0.35	-	1.38	1.38	-
2.90	0.76	0.90	0.94	0.04	3.81	3.63	-0.18	0.54	0.83	0.29
9.80	0.31	99.37	100.79	1.42	100.19	98.18	-2.01	99.35	100.02	0.67
8.13	-0.32	29.63	33.24	3.61	44.10	42.18	-1.92	46.25	46.45	0.20

931D**		5647**			65/1551A			65/1551B		
-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.
2.70	1.40	49.10	48.80	-0.30	42.10	46.30	4.20	50.60	53.80*	3.20
5.65	-1.15	32.30	33.20	0.90	20.00	19.10	-0.90	20.10	20.93*	0.83
9.70	-0.60	8.10	8.40	0.30	13.60	13.40	-0.20	8.60	8.80*	0.20
0.09	-	0.17	0.17	-	1.57	1.57	-	0.73	0.73	-
0.96	-0.07	1.61	1.65	0.04	1.28	1.16	-0.12	1.44	1.38*	-0.06
2.10	-0.40	1.80	1.70	-0.10	11.10	10.90	-0.20	9.30	9.20*	-0.10
2.38	-0.02	1.47	1.42	-0.05	2.85	2.52	-0.33	2.97	2.34*	-0.63
0.45	-0.01	2.54	2.50	-0.04	0.30	0.28	-0.02	0.50	0.52*	0.02
0.60	-	1.41	1.41	-	0.61	0.61	-	0.40	0.40	-
3.74	-0.14	1.18	1.21	0.03	6.60	5.70	-0.90	5.13	4.24*	-0.89
8.37	-0.99	99.68	100.46	0.78	100.01	101.54	1.53	99.77	102.34	2.57
6.40	-1.82	42.18	43.42	1.24	36.45	35.23	-1.22	30.87	31.84	0.97

/585D		64/1178A			64/741A			63/559B		
-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.
0.00	1.70	53.30	54.20	0.90	61.90	61.40	-0.50	68.20	68.60	0.40
1.90	0.90	30.90	31.25	0.35	27.50	27.50	0.00	21.40	22.45	1.05
9.10	0.50	5.00	5.65	0.65	2.90	3.40	0.50	5.90	6.65	0.75
0.17	-	0.59	0.59	-	0.10	0.10	-	0.13	0.13	-
2.32	-0.08	1.59	0.82	-0.77	1.11	1.26	0.15	1.74	1.76	0.02
3.80	-0.10	5.20	5.40	0.20	2.50	2.45	-0.05	0.90	0.90	0.00
1.52	-0.04	1.24	1.36	0.12	1.47	1.81	0.34	0.54	0.54	0.00
1.16	0.06	0.70	0.71	0.01	1.56	1.56	0.00	0.99	1.01	0.02
0.13	-	0.13	0.13	-	0.14	0.14	-	0.13	0.13	-
2.13	-0.23	1.59	1.66	0.07	1.41	1.43	0.02	0.58	0.60	0.02
2.23	2.71	100.24	101.77	1.53	100.59	101.05	0.46	100.51	102.77	2.26
3.49	1.32	38.08	38.31	0.23	31.61	32.26	0.65	29.17	30.99	1.82

he recent and earlier results is given.
etrometric Analysis of Coal Ash. Technical Memorandum No. 21 of 1965.)

TABLE

(where a dash is given in the difference columns, the chem

Sample No.	63/559D			63/461D		
Components	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.
SiO_2	63.60	64.10	0.50	43.90	43.90	0.00
Al_2O_3	21.00	21.40	0.40	36.60	34.80	-1.80
Fe_2O_3	5.50	6.20	0.70	5.30	5.60	0.30
P_2O	0.12	0.12	-	0.59	0.59	-
TiO_2	1.79	2.45	0.66	2.81	2.77	-0.04
CaO	3.40	3.60	0.20	4.90	5.05	0.15
MgO	1.55	1.68	0.13	2.64	2.74	0.10
K_2O	0.91	0.93	0.02	0.46	0.50	0.04
Na_2O	0.13	0.13	-	0.23	0.23	-
SO_3	2.13	1.66	-0.47	2.83	3.24	0.41
Total Sum	100.13	102.27	2.14	100.26	99.42	-0.84
Sum of Al_2O_3, Fe_2O_3, P_2O_5 and TiO_2	28.41	30.17	1.76	45.30	43.76	-1.54

5 (Continued)

nically determined value has been used in the fluorescence column)

64/761C			64/750C			64/748B		
Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.	Chem.	X-Ray	Diff.
39.90	40.40	0.50	46.40	47.50	1.10	32.80	33.30	0.50
38.60	36.50	-2.10	30.20	28.45	-1.75	34.60	30.95	-3.65
5.20	4.10	-1.10	5.60	5.60	0.00	9.50	8.55	-0.95
0.52	0.52	-	0.33	0.33	-	1.55	1.55	-
1.70	1.76	0.06	1.28	1.36	0.08	1.35	1.36	0.01
8.40	8.55	0.15	10.30	10.55	0.25	12.40	12.60	0.20
2.71	2.70	-0.01	2.64	2.74	0.10	3.20	3.15	-0.05
0.41	0.41	0.00	0.64	0.61	-0.03	0.39	0.33	-0.06
0.41	0.41	-	0.19	0.19	-	0.46	0.46	-
2.43	2.43	0.00	2.21	2.45	0.24	3.54	3.62	0.08
100.28	97.78	-2.50	99.79	99.78	-0.01	99.79	95.87	-3.92
46.02	42.88	-3.14	37.41	35.74	-1.67	47.00	42.41	-4.59

Table 6...../

COMPARATIVE NORMAL CHEMICAL AND X-RAY

AND IN THE FIRST 7 CASES ALSO

(where a dash is given in the difference columns, t

Sample No.	63/931D					65/15		
Components	Chem.	X-Ray 1	Diff.	X-Ray 2	Diff.	Chem.	X-Ray 1	Diff
SiO_2	41.30	41.50	0.20	42.70	1.40	42.10	43.10	1.0
Al_2O_3	26.80	27.50	0.70	25.65	-1.15	20.00	19.70	-0.3
Fe_2O_3	10.30	9.80	-0.50	9.70	-0.60	13.60	13.70	0.1
P_2O_5	0.09	0.09	-	0.09	-	1.57	1.57	-
TiO_2	1.03	1.02	-0.01	0.96	-0.07	1.28	1.26	-0.0
CaO	12.50	12.30	-0.20	12.10	-0.40	11.10	11.30	0.2
MgO	2.40	2.40	0.00	2.38	-0.02	2.85	2.49	-0.3
K_2O	0.46	0.43	-0.03	0.45	-0.01	0.30	0.26	-0.0
Na_2O	0.60	0.60	-	0.60	-	0.61	0.61	-
SO_3	3.88	3.88	-	3.74	-0.14	6.60	6.60	-
Total Sum	99.36	99.52	0.16	98.37	-0.99	100.01	100.59	0.5
Sum of Al_2O_3, Fe_2O_3, P_2O_5 and TiO_2	38.22	38.41	0.19	36.40	-1.82	36.45	36.23	-0.2

Sample No.	63/461D					65/15		
Components	Chem.	X-Ray 1	Diff.	X-Ray 2	Diff.	Chem.	X-Ray 1	Diff
SiO_2	43.90	43.10	-0.80	43.90	0.00	44.00	39.60	-4.4
Al_2O_3	36.60	37.40	0.80	34.80	-1.80	28.10	29.50	1.4
Fe_2O_3	5.30	5.50	0.20	5.60	0.30	5.20	4.90	-0.3
P_2O_5	0.59	0.59	-	0.59	-	1.07	1.07	-
TiO_2	2.81	2.85	0.04	2.77	-0.04	1.51	1.54	0.0
CaO	4.90	5.20	0.30	5.05	0.15	10.90	13.50	2.6
MgO	2.64	2.85	0.21	2.74	0.10	4.28	3.64	-0.6
K_2O	0.46	0.49	0.03	0.50	0.04	0.75	0.76	0.0
Na_2O	0.23	0.23	-	0.23	-	0.61	0.61	-
SO_3	2.83	2.83	-	3.24	0.41	3.46	3.46	-
Total Sum	100.26	101.04	0.78	99.42	-0.84	99.88	98.58	-1.3
Sum of Al_2O_3, Fe_2O_3, P_2O_5 and TiO_2	45.30	46.34	1.04	43.76	-1.54	35.88	37.01	1.1

With the first 7 samples, a comparison is also made with the resul

X-Ray 1: Results obtained from fused discs.

X-Ray 2: Results obtained from pressed powder specimens.

TABLE 6.

FLUORESCENCE ANALYSES OBTAINED FROM FUSED DISCS (X-RAY 1) FROM PRESSED POWDER SPECIMENS (X-RAY 2) FOR COMPARISON.

he chemically determined value has been used in the fluorescence column)

51A			65/1551B					5639				
.	X-Ray 2	Diff.	Chem.	X-Ray 1	Diff.	X-Ray 2	Diff.	Chem.	X-Ray 1	Diff.	X-Ray 2	Diff.
0	46.30	4.20	50.60	52.90	2.30	53.80	3.20	56.70	57.10	0.40	53.80	-2.90
0	19.10	-0.90	20.10	21.50	1.40	20.93	0.83	28.70	31.00	2.30	29.60	0.90
0	13.40	-0.20	8.60	8.90	0.30	8.80	0.20	6.50	7.10	0.60	7.50	1.00
	1.57	-	0.73	0.73	-	0.73	-	0.13	0.13	-	0.13	-
2	1.16	-0.12	1.44	1.45	0.01	1.38	-0.06	1.61	1.71	0.10	1.67	0.06
0	10.90	-0.20	9.30	9.30	0.00	9.20	-0.10	0.10	1.10	0.00	1.10	0.00
6	2.52	-0.33	2.97	2.52	-0.45	2.34	-0.63	0.93	0.80	-0.13	0.83	-0.10
4	0.28	-0.02	0.50	0.58	0.08	0.52	0.02	2.78	2.76	-0.02	2.75	-0.03
	0.61	-	0.40	0.40	-	0.40	-	0.59	0.59	-	0.59	-
	5.70	-0.90	5.13	5.13	-	4.24	-0.89	0.47	0.47	-	0.73	0.26
8	101.54	1.53	99.77	103.41	3.64	102.34	2.57	99.51	102.76	3.25	98.70	-0.81
2	35.23	-1.22	30.87	32.58	1.71	31.84	0.97	36.94	39.94	3.00	38.90	1.96

585A			62/585B					65/1585B		
.	X-Ray 2	Diff.	Chem.	X-Ray 1	Diff.	X-Ray 2	Diff.	Chem.	X-Ray 1	Diff.
0	40.80	-3.20	56.10	58.20	2.10	58.40	2.30	43.80	43.70	-0.10
0	27.80	-0.30	21.70	20.80	-0.90	22.50	0.80	28.80	28.80	0.00
0	4.90	-0.30	12.50	12.90	0.40	13.35	0.85	5.20	5.20	0.00
	1.07	-	0.12	0.12	-	0.12	-	1.21	1.21	-
3	1.43	-0.08	1.39	1.55	0.16	1.49	0.10	1.48	1.50	0.02
0	13.30	2.40	3.10	3.10	0.00	3.10	0.00	11.60	10.90	-0.70
4	3.69	-0.59	1.42	1.42	0.00	1.34	-0.08	3.74	3.74	0.00
1	0.75	0.00	1.12	1.13	0.01	1.13	0.01	0.45	0.51	0.06
	0.61	-	0.13	0.13	-	0.13	-	0.50	0.50	-
	3.41	-0.05	2.35	2.35	-	1.93	-0.42	2.79	2.79	-
0	97.76	-2.12	99.93	101.70	1.77	103.49	3.56	99.57	98.85	-0.72
3	35.20	-0.68	35.71	35.37	-0.34	37.46	1.75	36.69	36.71	0.02

s obtained from the pressed powder specimens.

TABLE (

'where a dash is given in the difference columns, the chemic

Sample No.	63/814C			62/584B		
Components	Chem.	X-Ray 1	Diff.	Chem.	X-Ray 1	Diff.
SiO_2	46.50	47.30	0.80	52.20	52.10	-0.10
Al_2O_3	28.20	26.40	-1.80	22.80	21.60	-1.20
Fe_2O_3	9.30	9.00	-0.30	14.40	14.60	0.20
P_2O_5	0.49	0.49	-	0.25	0.25	-
TiO_2	1.54	1.50	-0.04	1.64	1.74	-0.10
CaO	7.20	7.00	-0.20	3.50	3.50	0.00
MgO	1.18	1.30	0.12	1.44	1.48	0.04
K_2O	1.78	1.80	0.02	0.99	0.99	0.00
Na_2O	0.90	0.90	-	0.13	0.13	-
SO_3	2.55	2.55	-	2.80	2.80	-
Total Sum	99.64	98.24	-1.40	100.15	99.19	-0.96
Sum of Al_2O_3 Fe_2O_3, P_2O_5 and TiO_2	39.53	37.39	-2.14	39.09	38.19	-0.90

Sample No.	65/1886C			65/1
Components	Chem.	X-Ray 1	Diff.	Chem.	X-Ray
SiO_2	46.00	45.60	-0.40	43.80	43.
Al_2O_3	34.80	36.30	1.50	42.10	43.
Fe_2O_3	3.30	3.30	0.00	1.50	1.
P_2O_5	1.06	1.06	-	1.51	1.
TiO_2	1.80	1.77	-0.03	1.84	1.
CaO	9.50	9.60	0.10	7.30	7.
MgO	1.36	1.38	0.02	0.77	0.
K_2O	0.38	0.38	0.00	0.41	0.
Na_2O	0.20	0.20	-	0.17	0.
SO_3	1.62	1.62	-	1.22	1.
Total Sum	100.02	101.21	1.19	100.62	101.
Sum of Al_2O_3 Fe_2O_3, P_2O_5 and TiO_2	40.96	42.43	1.47	46.95	48.

Sample No.	11/63 7/8" x $\frac{3}{8}$"			11/63 6"
Components	Chem.	X-Ray 1	Diff.	Chem.	X-Ray
SiO_2	41.00	39.40	-1.60	36.60	35.
Al_2O_3	41.00	39.10	-1.90	39.60	34.
Fe_2O_3	3.10	7.30	4.20	6.50	11.
P_2O_5	1.78	1.78	-	1.78	1.
TiO_2	1.65	1.76	0.11	1.41	1.
CaO	7.10	6.90	-0.20	8.90	8.
MgO	1.34	1.32	-0.02	1.63	1.
K_2O	0.55	0.53	-0.02	0.47	0.
Na_2O	0.19	0.19	-	0.19	0.
SO_3	1.82	1.82	-	2.32	2.
Total Sum	99.53	100.10	0.57	99.40	97.
Sum of Al_2O_3 Fe_2O_3, P_2O_5 and TiO_2	47.53	49.94	2.41	49.29	49.

X-Ray 1: Results obtained from fused discs.

5 (Continued)
ually determined value has been used in the fluorescence column)

65/1551C			62/584D			65/1880C		
Chem.	X-Ray 1	Diff.	Chem.	X-Ray 1	Diff.	Chem.	X-Ray 1	Diff.
50.70	51.10	0.40	53.00	53.60	0.60	36.60	36.10	-0.50
24.80	25.90	1.10	26.20	25.60	-0.60	37.20	37.20	0.00
6.40	6.80	0.40	8.70	9.10	0.40	4.50	4.60	0.10
0.32	0.32	-	0.35	0.35	-	2.09	2.09	-
1.34	1.33	-0.01	2.11	2.19	0.08	1.64	1.63	-0.01
9.00	9.10	0.10	4.50	4.60	0.10	12.30	12.40	0.10
2.95	2.55	-0.40	1.81	1.88	0.07	2.72	2.54	-0.18
0.40	0.34	-0.06	1.11	1.08	-0.03	0.41	0.38	-0.03
0.61	0.61	-	0.16	0.16	-	0.39	0.39	-
3.79	3.79	-	2.28	2.28	-	2.51	2.51	-
100.31	101.84	1.53	100.22	100.84	0.62	100.36	99.85	-0.51
32.86	34.35	1.49	37.36	37.24	-0.12	45.43	45.52	0.09

399C		62/2127A			62/210B		
r 1	Diff.	Chem.	X-Ray 1	Diff.	Chem.	X-Ray 1	Diff.
50	-0.30	32.60	35.00	2.40	41.10	42.40	1.30
20	1.10	36.60	35.80	-0.80	28.00	26.60	-1.40
70	0.20	4.30	3.90	-0.40	11.50	11.30	-0.20
51	-	0.49	0.49	-	0.35	0.35	-
37	0.03	1.66	1.58	-0.08	1.38	1.41	0.03
40	0.10	15.60	15.20	-0.40	9.60	9.20	-0.40
56	-0.11	2.71	2.90	0.19	2.08	1.90	-0.18
41	0.00	0.64	0.89	0.25	1.14	1.21	0.07
L7	-	0.36	0.36	-	1.69	1.69	-
22	-	5.52	5.52	-	2.98	2.98	-
54	1.02	100.48	101.64	1.16	99.82	99.04	-0.78
28	1.33	43.05	41.77	-1.28	41.23	39.66	-1.57

x 7/8"		62/390B6			62/1168A		
y 1	Diff.	Chem.	X-Ray 1	Diff.	Chem.	X-Ray 1	Diff.
30	-1.30	25.00	26.40	1.40	49.10	48.70	-0.40
LO	-5.50	19.40	19.40	0.00	29.50	30.30	0.80
30	5.30	22.30	17.10	-5.20	11.00	10.60	-0.40
78	-	1.01	1.01	-	0.09	0.09	-
41	0.00	0.76	0.66	-0.10	1.42	1.65	0.23
50	-0.40	20.70	21.90	1.20	2.80	2.70	-0.10
58	0.05	3.27	2.72	-0.55	1.23	1.14	-0.09
46	-0.01	0.23	0.20	-0.03	1.60	1.55	-0.05
19	-	0.10	0.10	-	1.45	1.45	-
32	-	6.50	6.50	-	1.34	1.34	-
54	-1.86	99.27	95.99	-3.28	99.53	99.52	-0.01
09	-0.20	43.47	38.17	-5.30	42.01	42.64	0.63

Sample No.	No. on graph.
62/1194 A	1
62/712 B	2
62/1261 F	3
62/712 D	4
63/723 C	5
63/879 C	6
63/930 D	7
5639	8
63/931 D	9
5647	10
65/1551 A	11
65/1551 B	12
65/1585 A	13
62/585 B	14
62/585 D	15
64/1178 A	16
64/741 A	17
63/559 B	18
63/559 D	19
63/461 D	20
64/761 C	21
64/750 C	22
64/748 B	23

$X10^{3}$

2·5

FIG. 1.

PRESSED POWDER SPECIMENS.

⊙ - GROUND FOR 2 HOURS.

⊡ - EARLIER RESULTS.

△ = MINUS 325 MESH.

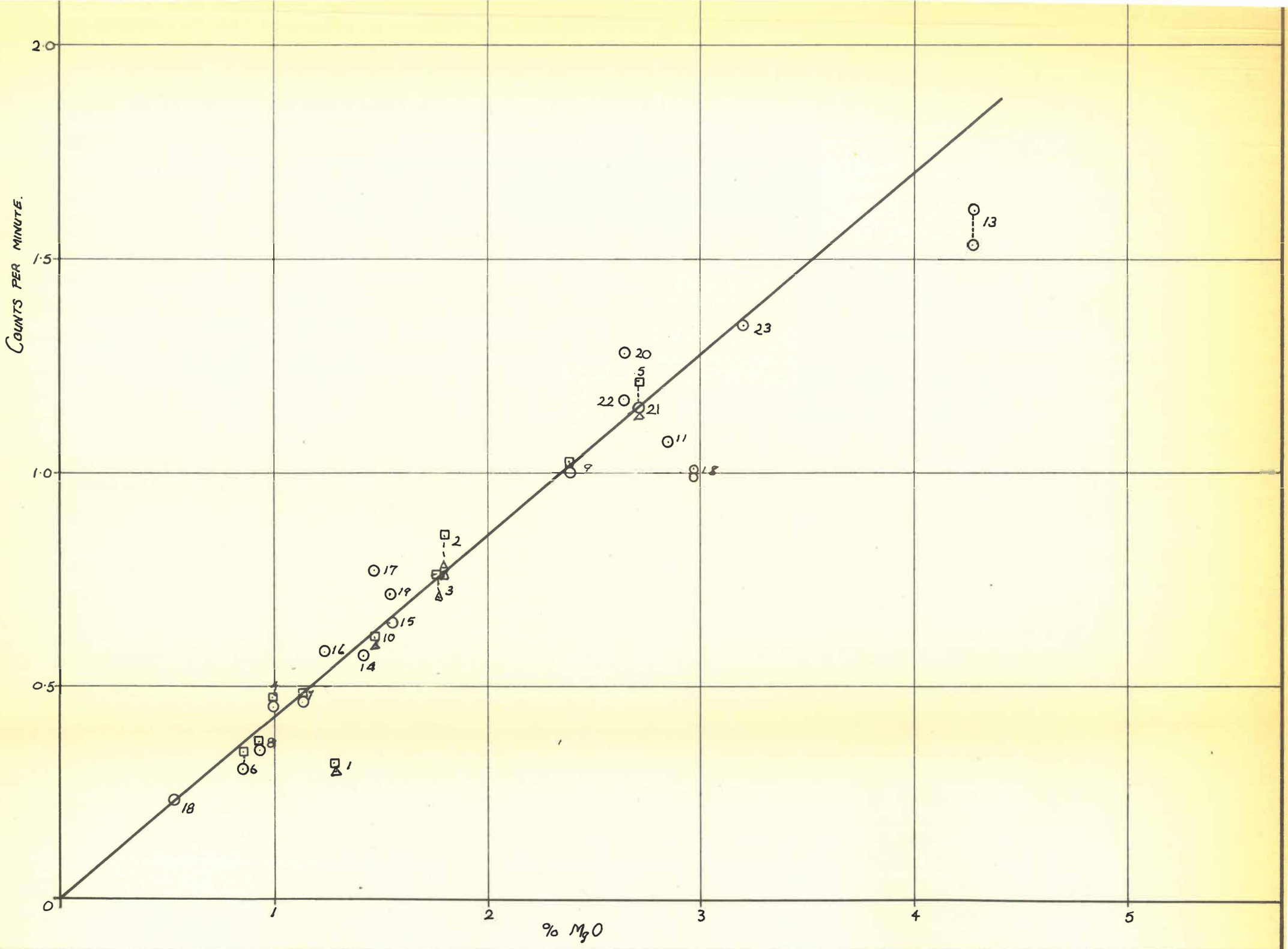
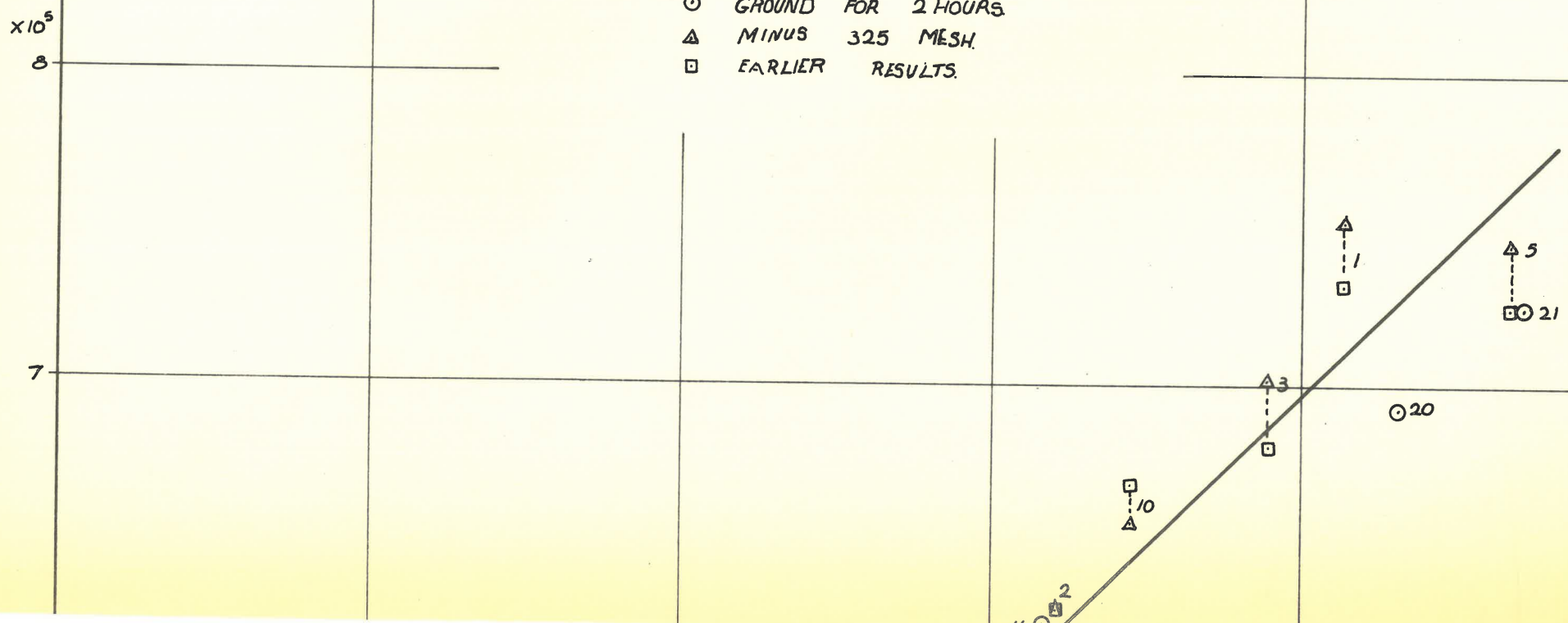
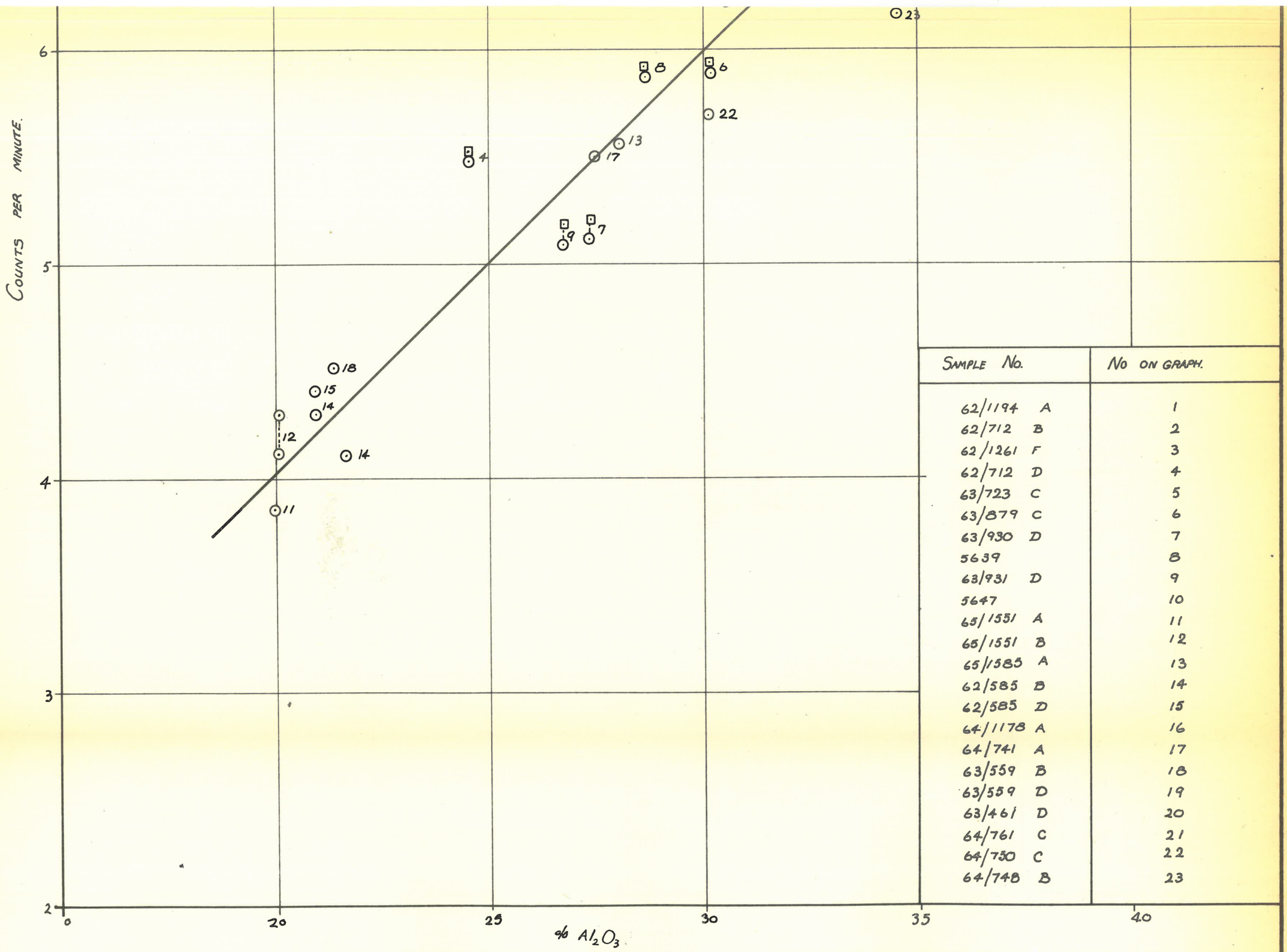

COUNTS PER MINUTE.
2·0
1·5
1·0
0·5
0
1
2
3
4
5
% MgO
1
2
3
4
5
6
7
8
9
10
11
13
14
15
16
17
18
19
20
21
22
23

FIG. 2.

PRESSED POWDER SPECIMENS.





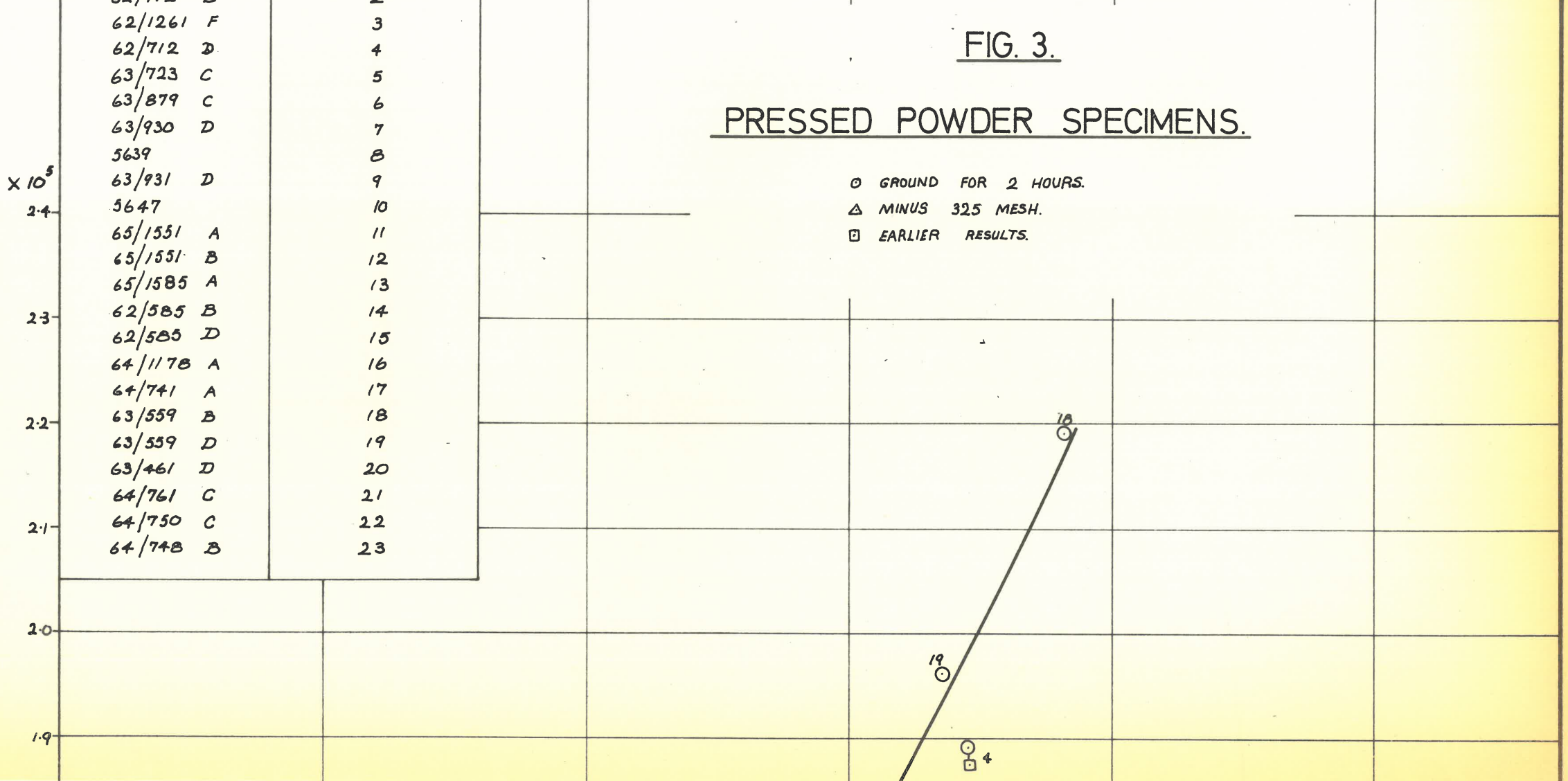
SAMPLE No.	No ON GRAPH.
62/1194 A	1
62/712 B	2
62/1261 F	3
62/712 D	4
63/723 C	5
63/879 C	6
63/930 D	7
5639	8
63/931 D	9
5647	10
65/1551 A	11
65/1551 B	12
65/1585 A	13
62/585 B	14
62/585 D	15
64/1178 A	16
64/741 A	17
63/559 B	18
63/559 D	19
63/461 D	20
64/761 C	21
64/750 C	22
64/748 B	23

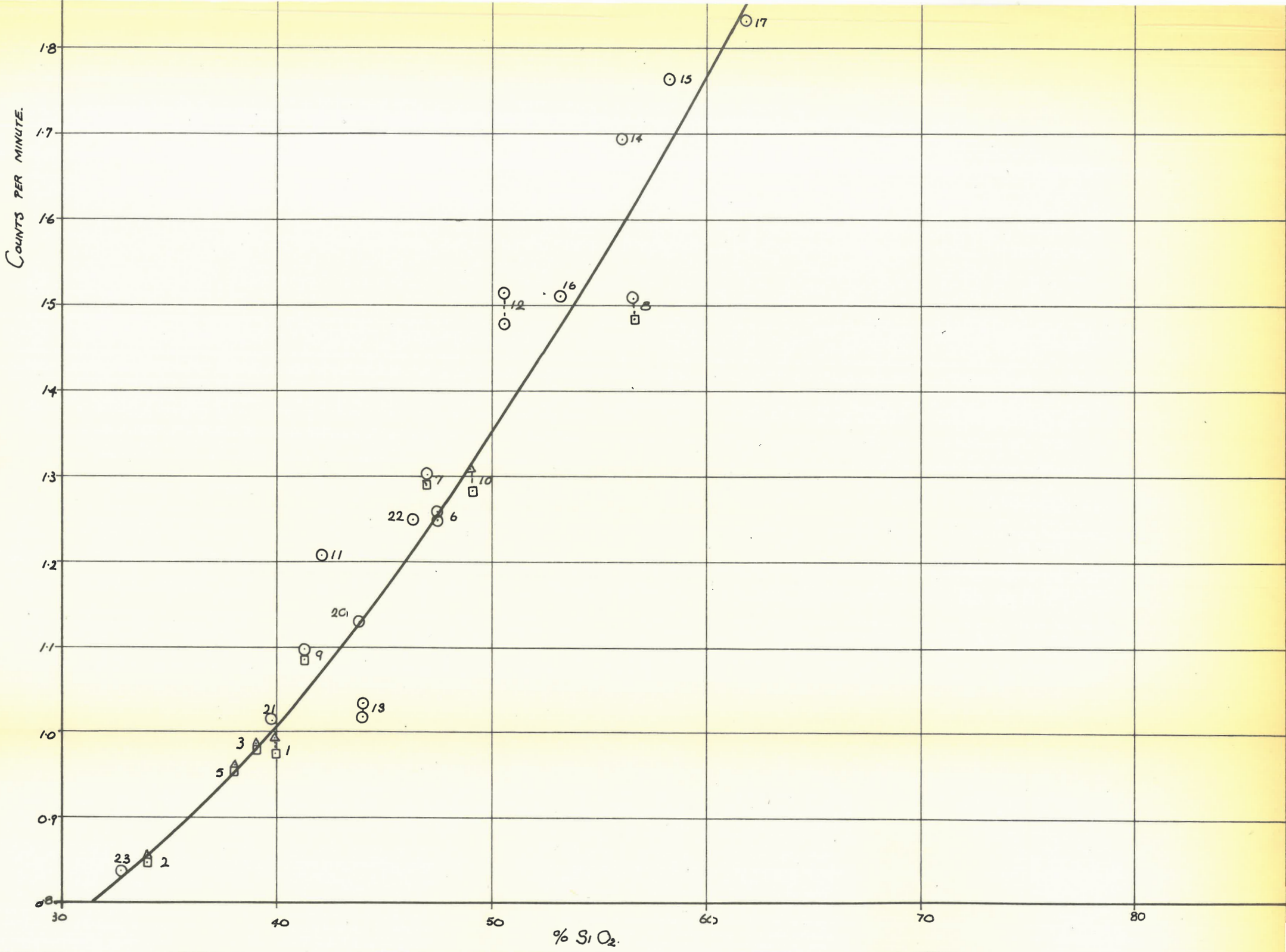
Sample No.	No. on graph.
62/1194 A	1
62/712 B	2
62/1261 F	3
62/712 D	4
63/723 C	5
63/879 C	6
63/930 D	7
5639	8
63/931 D	9
5647	10
65/1551 A	11
65/1551 B	12
65/1585 A	13
62/585 B	14
62/585 D	15
64/1178 A	16
64/741 A	17
63/559 B	18
63/559 D	19
63/461 D	20
64/761 C	21
64/750 C	22
64/748 B	23

FIG. 3.

PRESSED POWDER SPECIMENS.

⊙ GROUND FOR 2 HOURS.
△ MINUS 325 MESH.
⊡ EARLIER RESULTS.



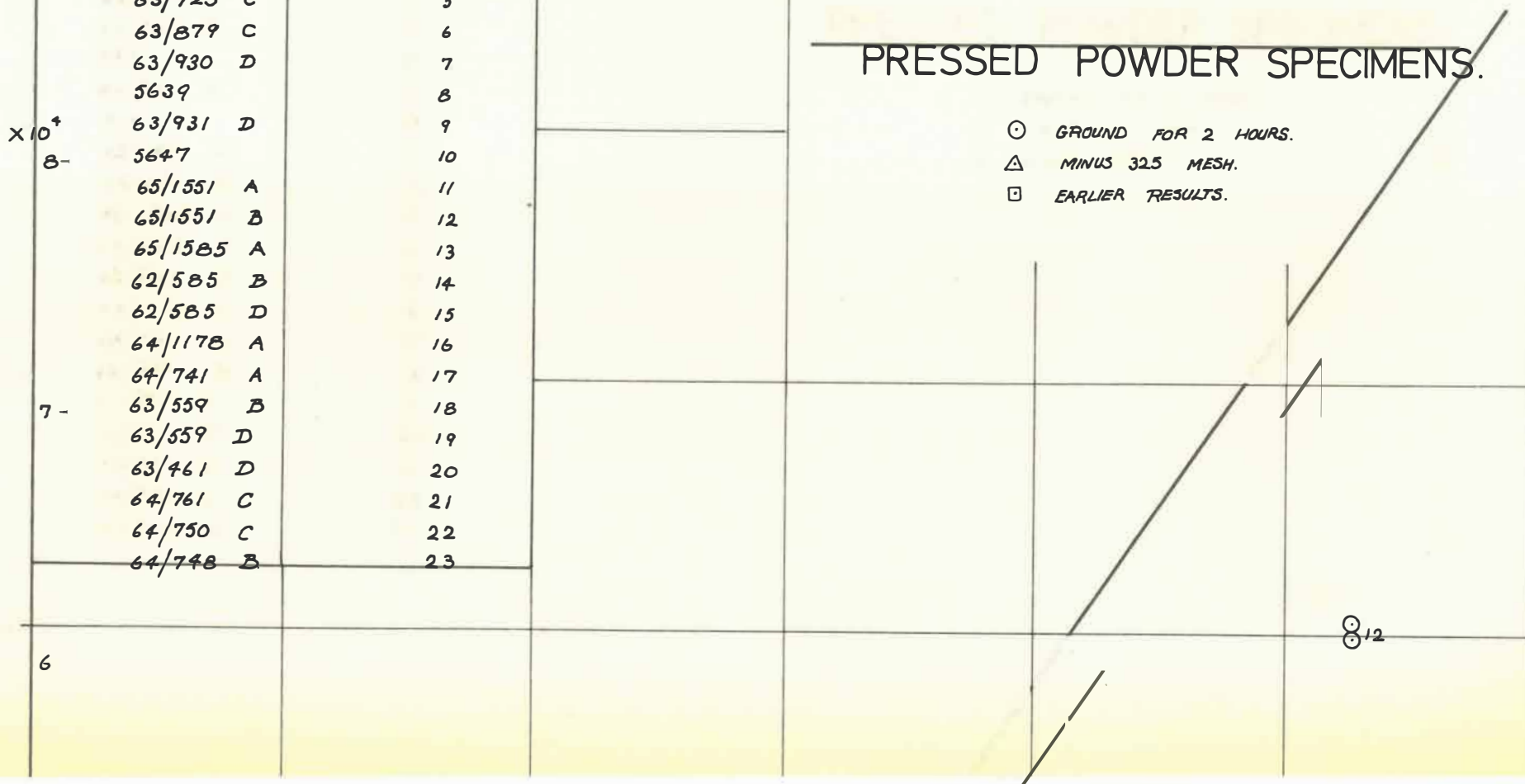

COUNTS PER MINUTE.
1·8
1·7
1·6
1·5
1·4
1·3
1·2
1·1
1·0
0·9
0·8
30
40
50
60
70
80
% Si O2.
17
15
14
12
16
8
7
10
22
6
11
20
9
18
21
3
1
5
23
2

Sample No.	No. on Graph.
62/1194 A	1
62/712F B	2
62/1261	3
62/712 D	4
63/723 C	5
63/879 C	6
63/930 D	7
5639	8
63/931 D	9
5647	10
65/1551 A	11
65/1551 B	12
65/1585 A	13
62/585 B	14
62/585 D	15
64/1178 A	16
64/741 A	17
63/559 B	18
63/559 D	19
63/461 D	20
64/761 C	21
64/750 C	22
64/748 B	23

FIG. 4.

PRESSED POWDER SPECIMENS.

⊙ GROUND FOR 2 HOURS.
△ MINUS 325 MESH.
⊡ EARLIER RESULTS.



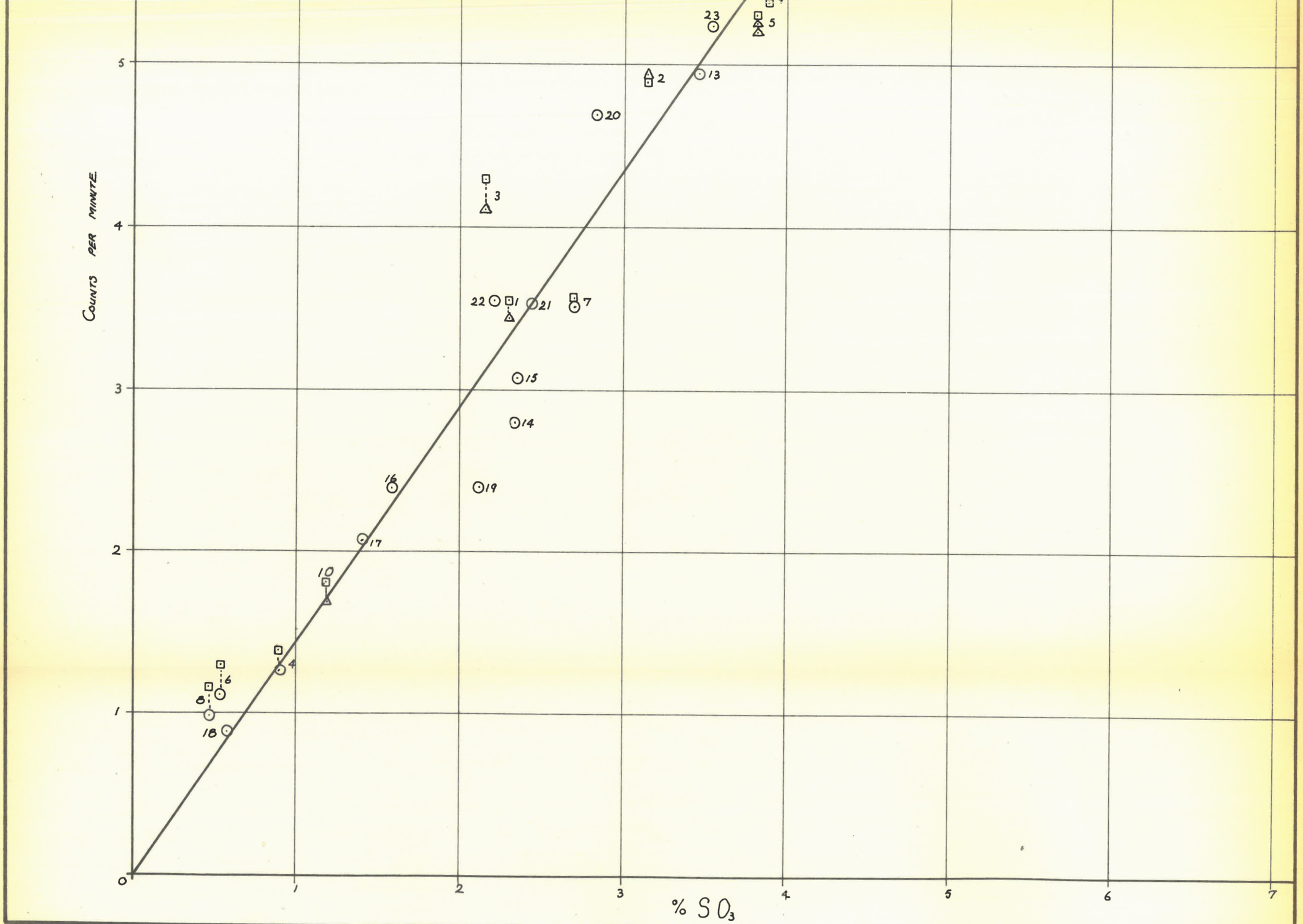
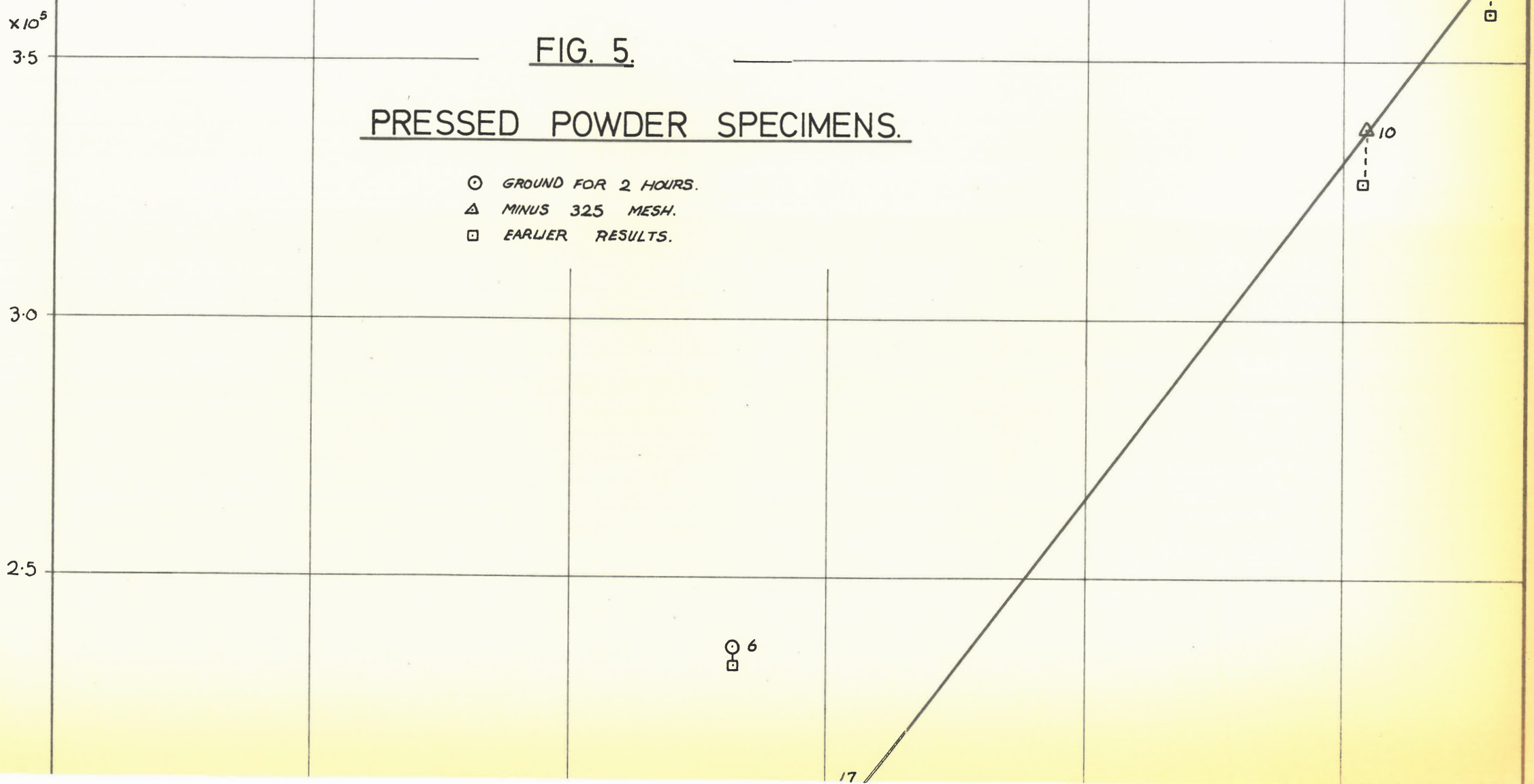
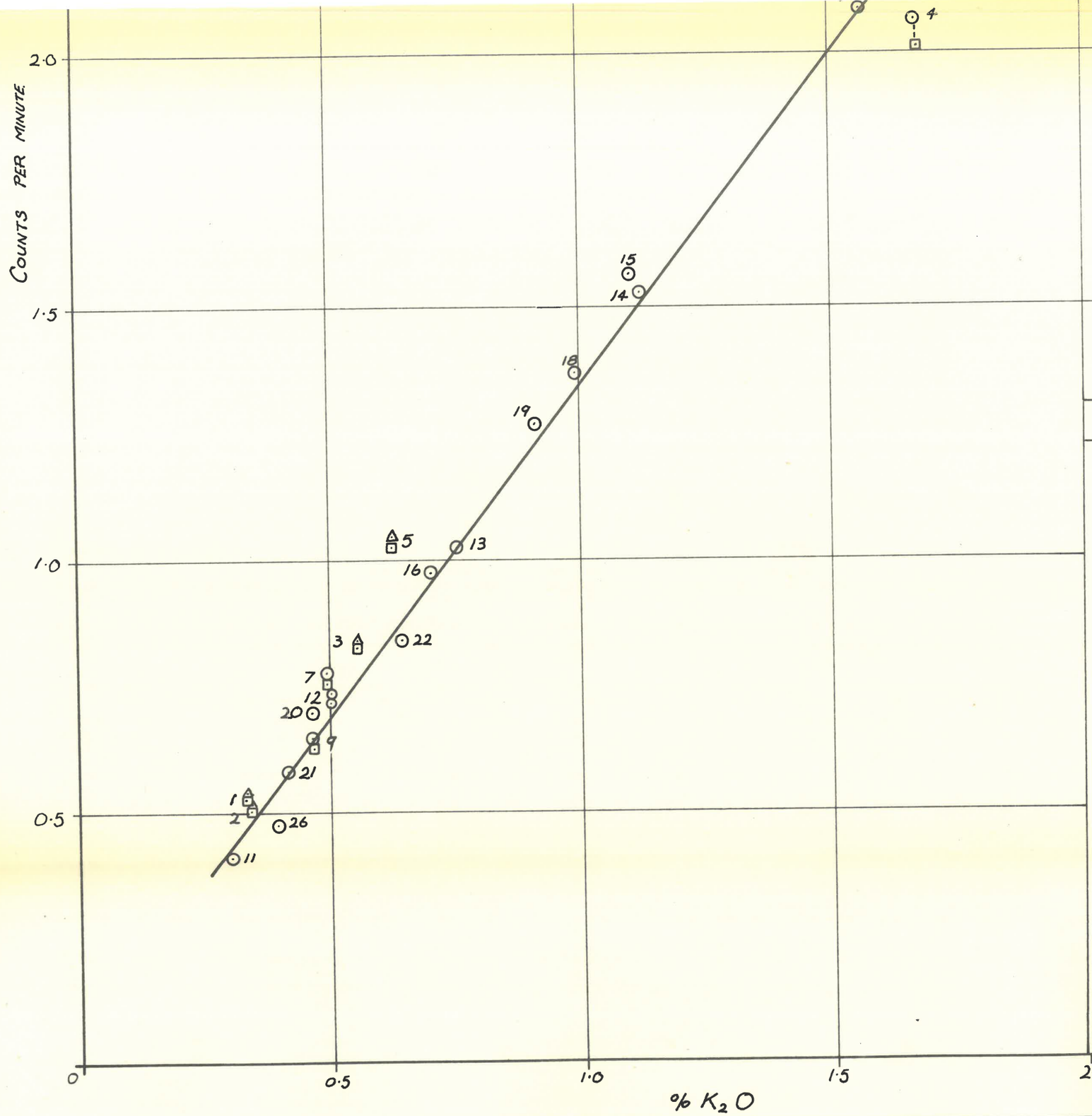

COUNTS PER MINUTE
% SO_3
0
1
2
3
4
5
6
7
23
5
2
13
20
3
22
1
21
7
15
14
16
19
17
10
4
6
8
18

FIG. 5.

PRESSED POWDER SPECIMENS.

⊙ GROUND FOR 2 HOURS.
△ MINUS 325 MESH.
⊡ EARLIER RESULTS.





SAMPLE NO.	NO. ON GRAPH.
62/1194 A	1
62/712 B	2
62/1261 F	3
62/712 D	4
63/723 C	5
63/879 C	6
63/930 D	7
5639	8
63/931 D	9
5647	10
65/1551 A	11
65/1551 B	12
65/1585 A	13
62/585 B	14
62/585 D	15
64/1178 A	16
64/741 A	17
63/559 B	18
63/559 D	19
63/461 D	20
64/761 C	21
64/750 C	22
64/748 B	23

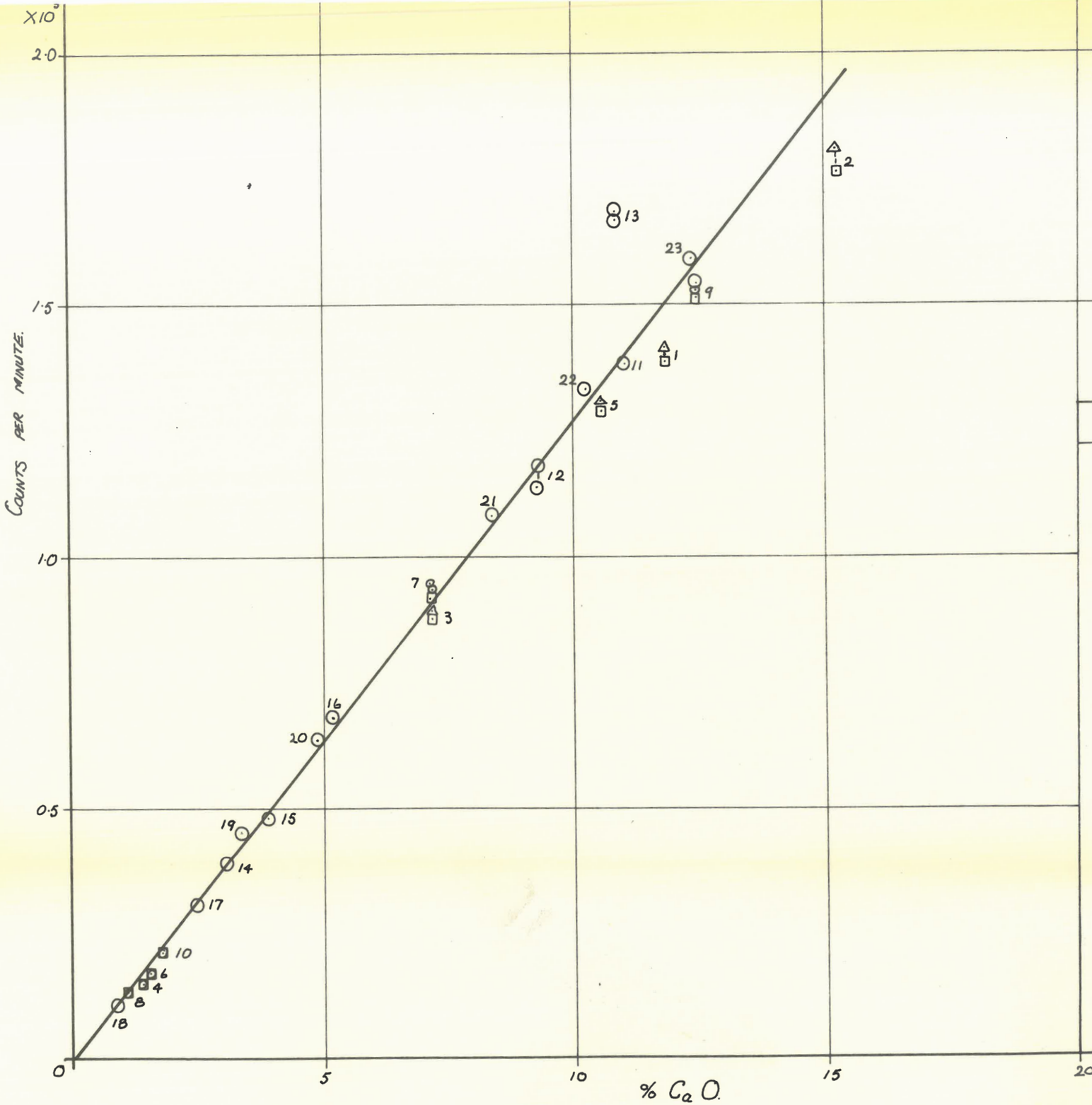
FIG. 6.

PRESSED POWDER SPECIMENS.

⊙ GROUND FOR 2 HOURS.

△ MINUS 325 MESH.

⊡ EARLIER RESULTS.



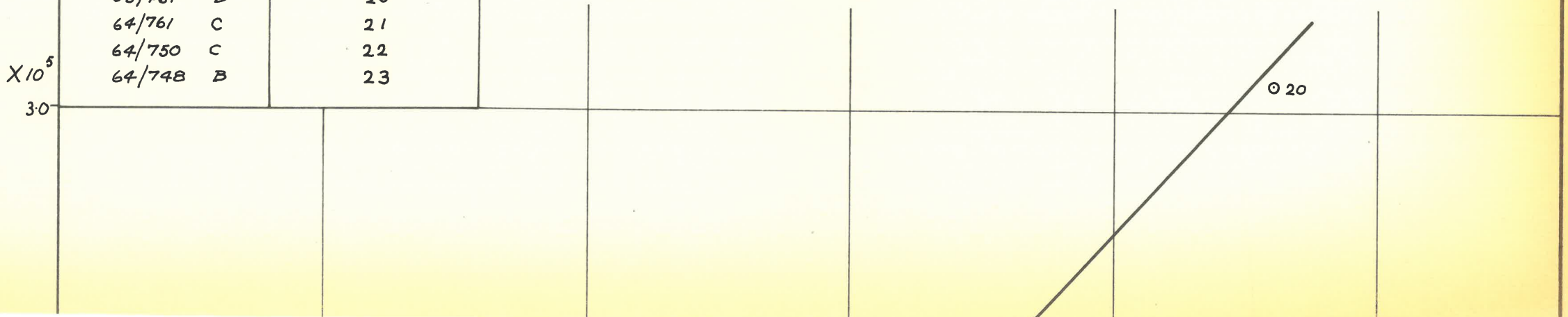
SAMPLE No.	No. ON GRAPH.
62/1194 A	1
62/712 B	2
62/1261 F	3
62/712 D	4
63/723 C	5
63/879 C	6
63/930 D	7
5639	8
63/931 D	9
5647	10
65/1551 A	11
65/1551 B	12
65/1585 A	13
62/585 B	14
62/585 D	15
64/1178 A	16
64/741 A	17
63/559 B	18
63/559 D	19
63/461 D	20
64/761 C	21
64/750 C	22
64/748 B	23

SAMPLE No.		NO. ON GRAPH.
62/1194	A	1
62/712	B	2
62/1261	F	3
62/712	D	4
63/723	C	5
63/879	C	6
63/930	D	7
5639		8
63/931	D	9
5647		10
65/1551	A	11
65/1551	B	12
65/1585	A	13
62/585	B	14
62/585	D	15
64/1178	A	16
64/741	A	17
63/559	B	18
63/559	D	19
63/461	D	20
64/761	C	21
64/750	C	22
64/748	B	23

FIG. 7.

PRESSED POWDER SPECIMENS.

⊙ GROUND FOR 2 HOURS
△ MINUS 325 MESH.
⊡ EARLIER RESULTS.



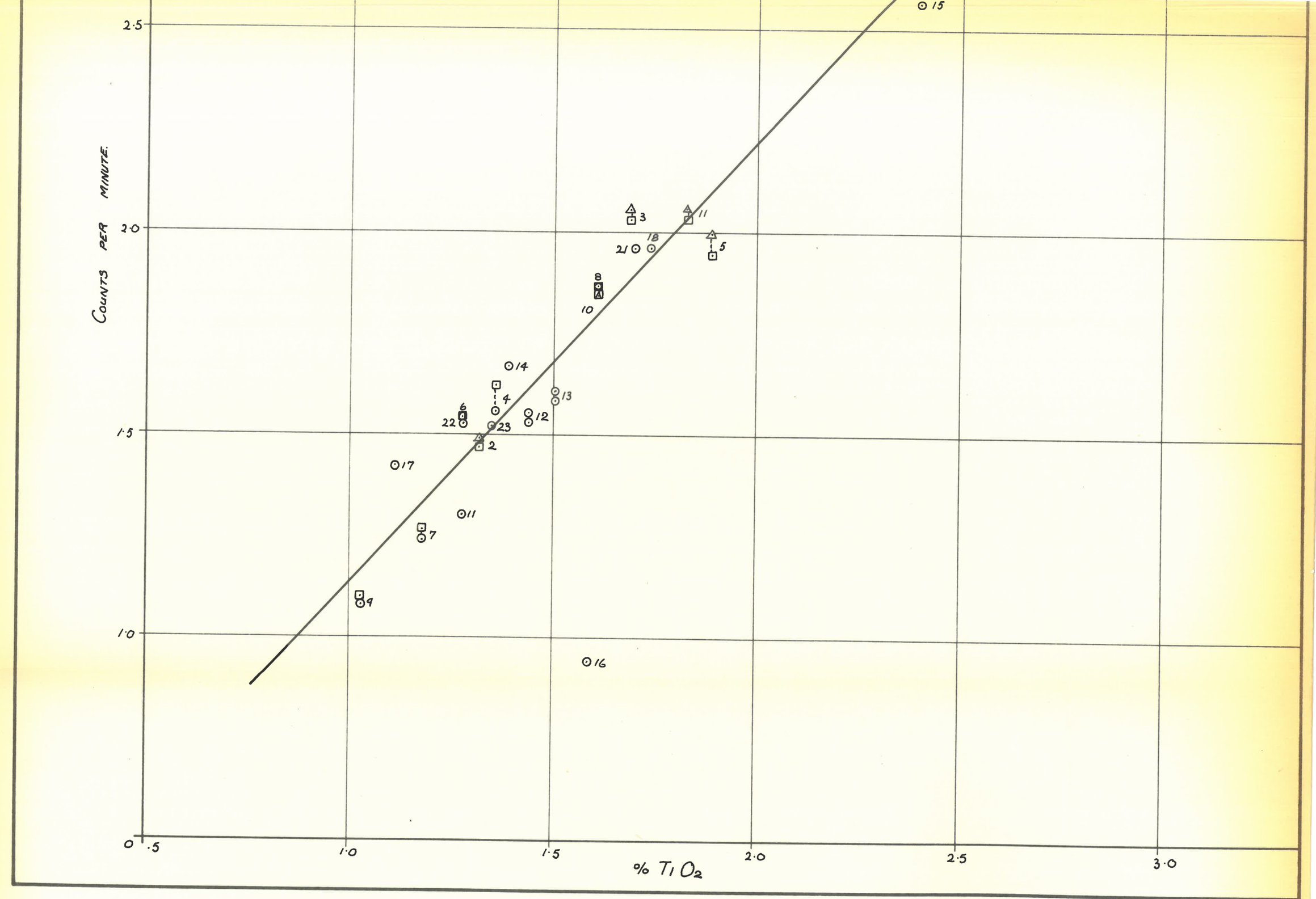
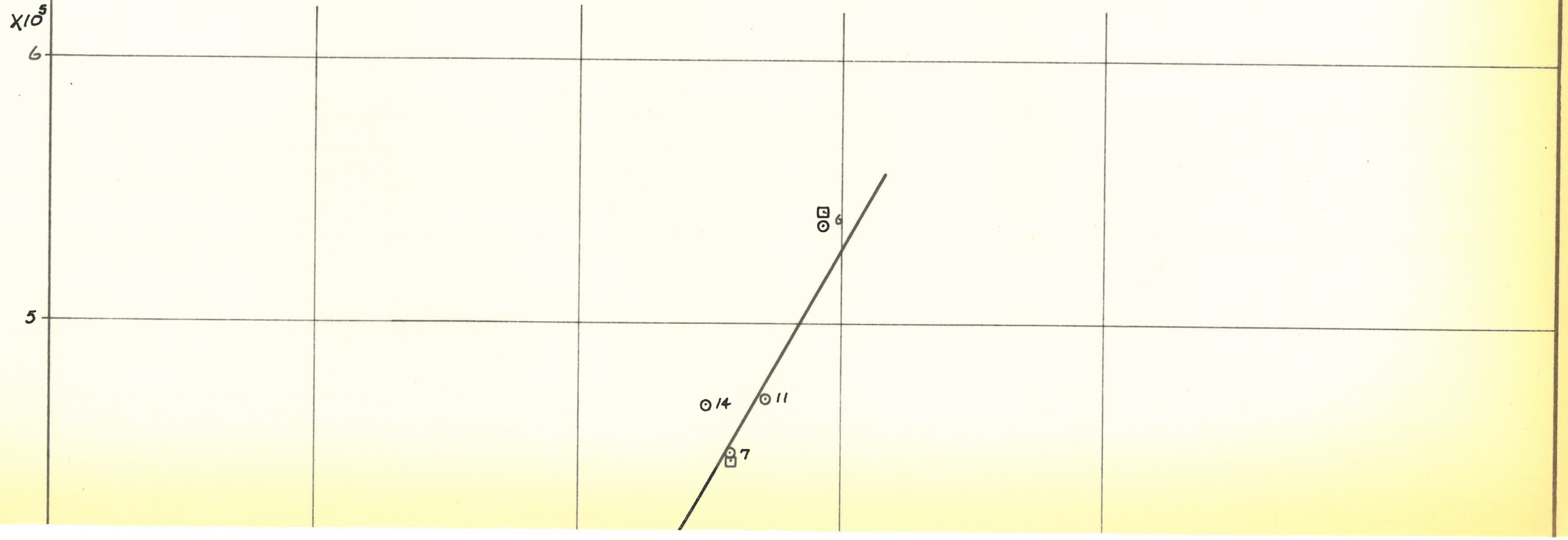
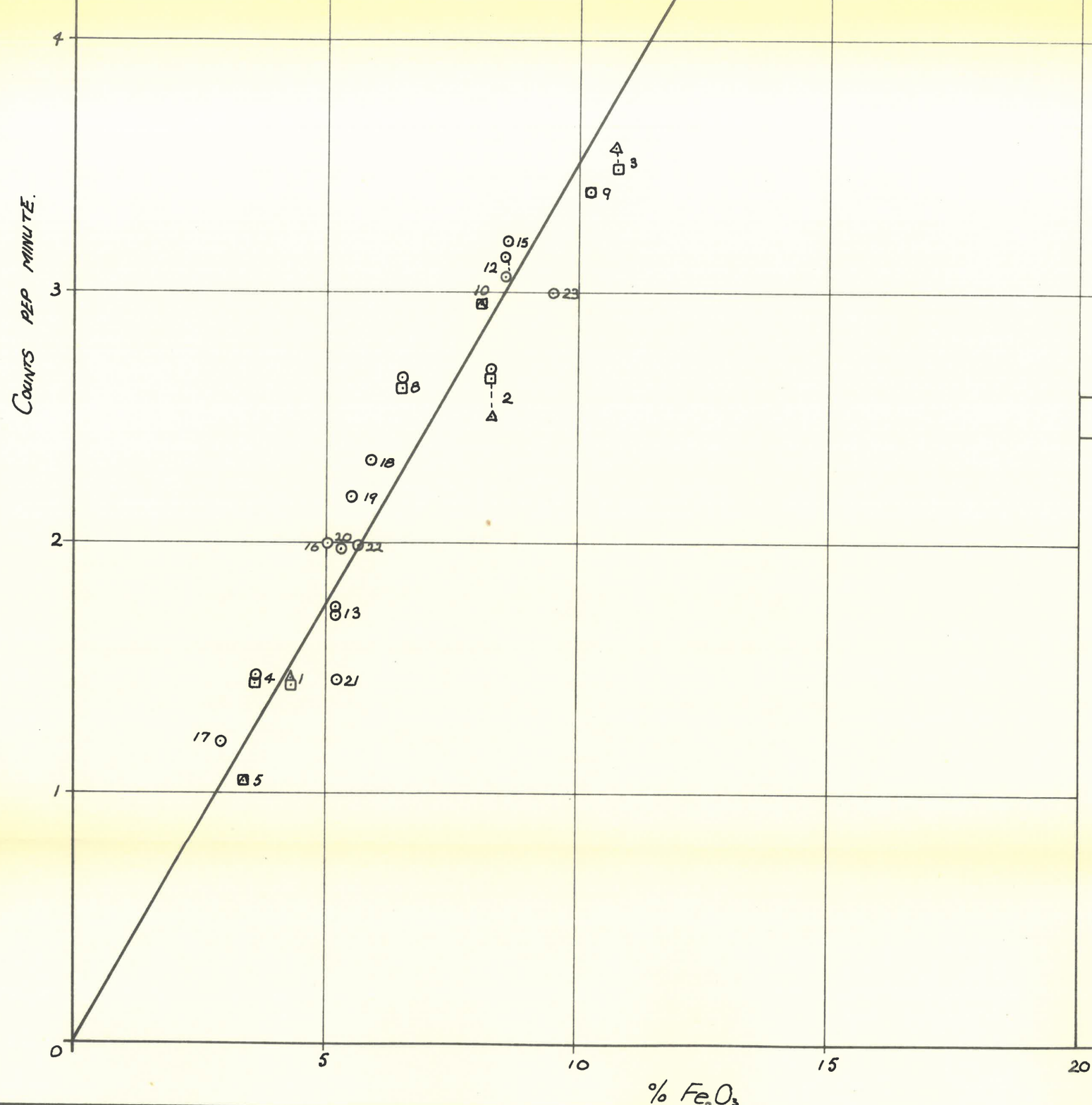

COUNTS PER MINUTE.
2·5
2·0
1·5
1·0
0
·5
1·0
1·5
2·0
2·5
3·0
% TiO2
15
3
11
18
21
5
8
10
14
4
13
6
22
12
23
2
17
11
7
9
16

FIG. 8.

PRESSED POWDER SPECIMENS.

⊙ GROUND FOR 2 HOURS.
△ MINUS 325 MESH.
⊡ EARLIER RESULTS.



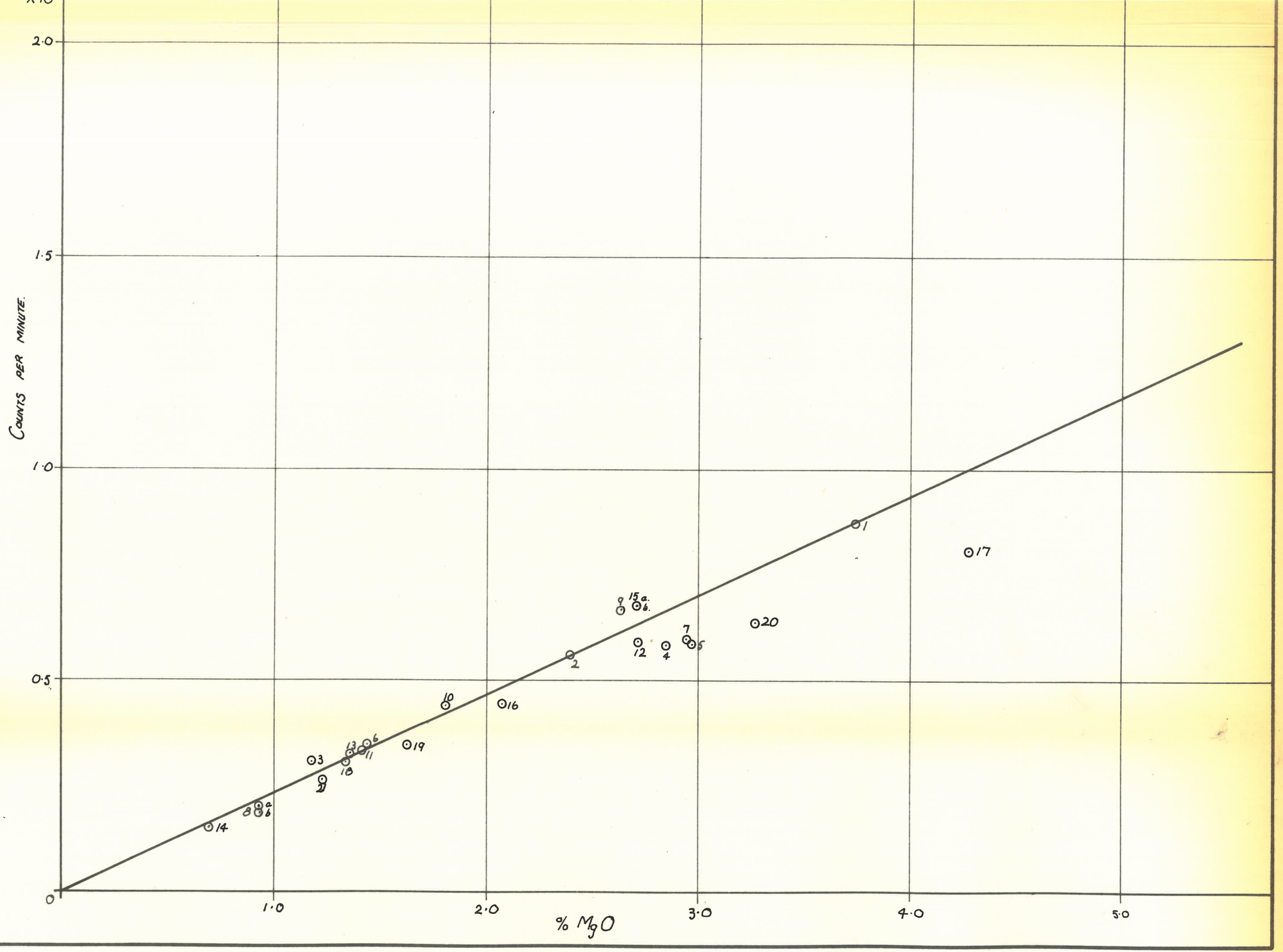


SAMPLE NO.	NO. ON GRAPH.
62/1194 A	1
62/712 B	2
62/1261 F	3
62/712 D	4
63/723 C	5
63/879 C	6
63/930 D	7
5639	8
63/931 D	9
5647	10
65/1551 A	11
65/1551 B	12
65/1585 A	13
62/585 B	14
62/585 D	15
64/1178 A	16
64/741 A	17
63/559 B	18
63/559 D	19
63/461 D	20
64/761 C	21
64/750 C	22
64/748 B	23

Sample No.	No. on graph.
65/1585 B	1
63/931 D	2
63/814 C	3
65/1551 A	4
65/1551 B	5
62/584 B	6
65/1551 C	7
5639	8(a & b)
63/461 D	9
62/584 D	10
63/585 B	11
65/1880 C	12
66/1886 C	13
65/1399 C	14
62/2127 A	15 (a & b)
67/210 B	16
65/1585 A	17
11/63 $\frac{1}{8}$" x $\frac{3}{8}$"	18
11/63 6" x $\frac{7}{8}$"	19
64/390 B_6	20
62/1168 A	21

FIG. 9.

FUSED DISCS.


2·0
1·5
1·0
0·5
0
COUNTS PER MINUTE.
1·0
2·0
3·0
4·0
5·0
% MgO
1
17
15a.
6.
8
20
7
5
12
4
2
10
16
19
13
6
11
3
18
14
8
a
6

SAMPLE No.	No. ON GRAPH.
65/1585 B	1
63/931 D	2
63/814 C	3
65/1551 A	4
65/1551 B	5
62/584 B	6
65/1551 C	7
5639	8(a & b)
63/461 D	9
62/584 D	10
63/585 B	11
65/1880 C	12
65/1886 C	13
65/1399 C	14
62/2127 A	15(a & b)
67/210 B	16
65/1585 A	17
11/63 $\frac{1}{8}$" x $\frac{3}{8}$"	18
11/63 6" x $\frac{7}{8}$"	19
64/396 B_6	20
62/1168 A	21

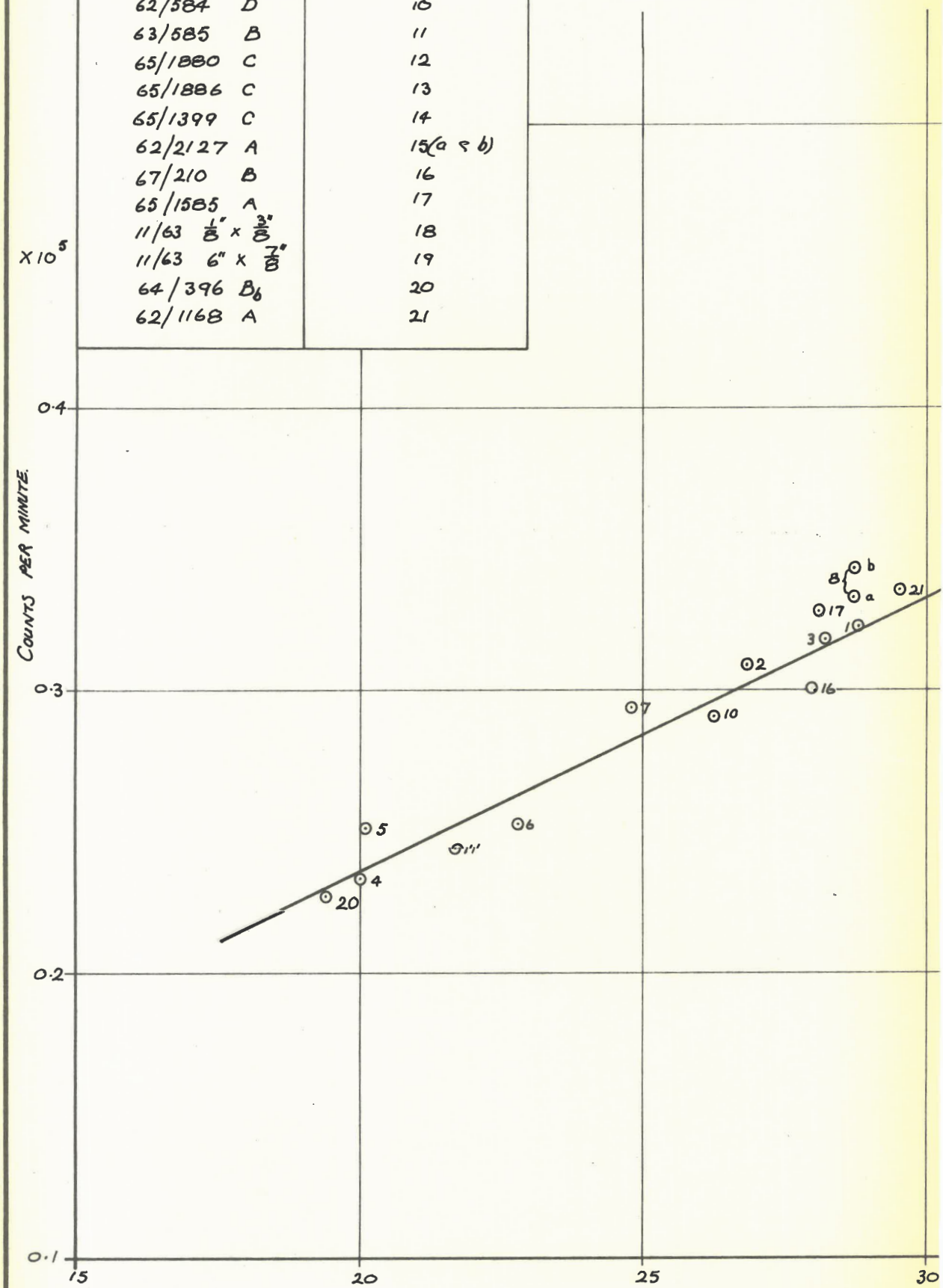


FIG. 10.

FUSED DISCS.

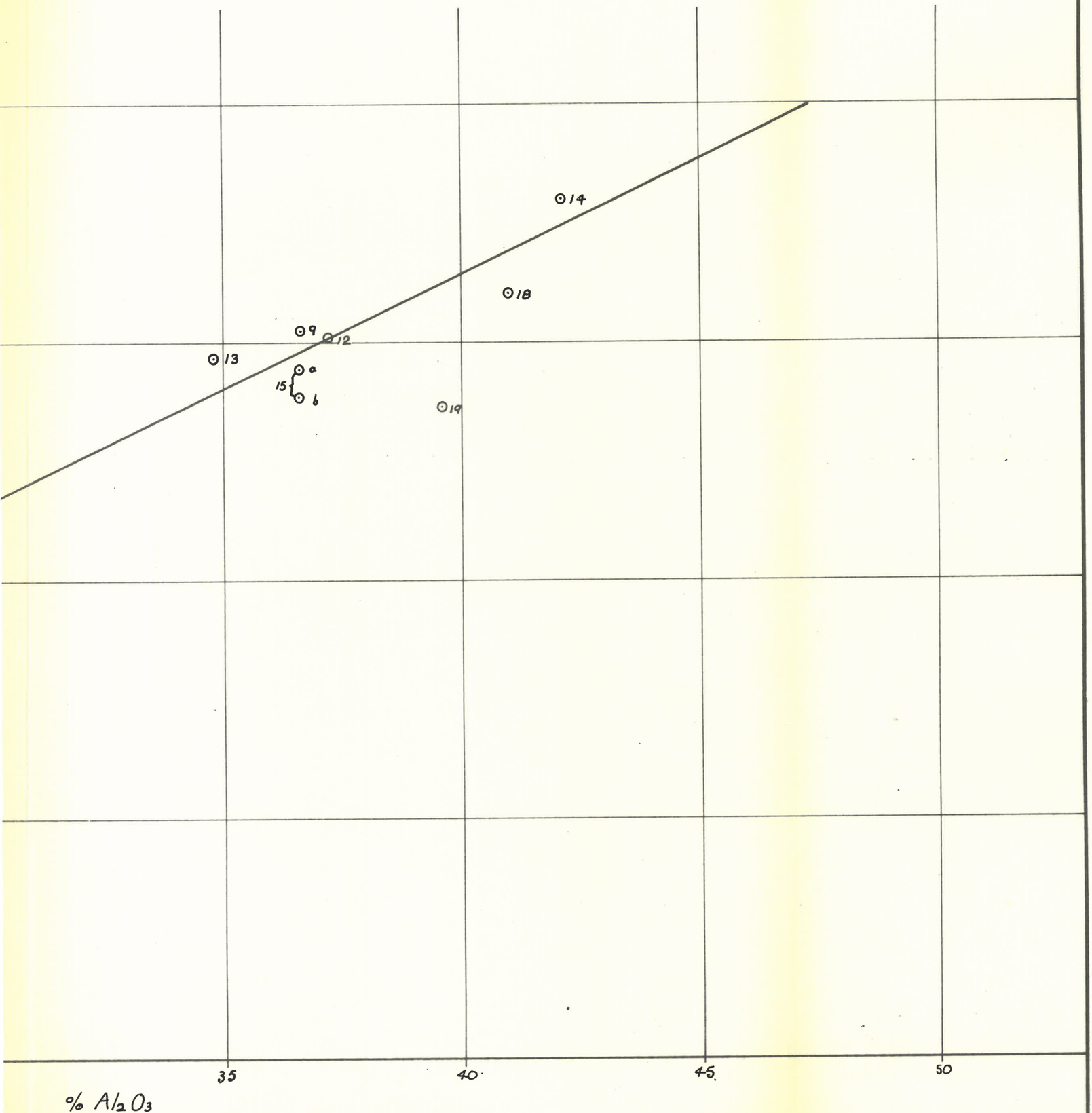


FIG. 11.

FUSED DISCS.

Sample No.	No on graph.
65/1585 B	1
63/931 D	2
63/814 C	3
65/1551 A	4
65/1551 B	5
62/584 B	6
65/1551 C	7
5639	8 (a & b)
63/461 D	9
62/584 D	10
63/584 B	11
65/1880 C	12
65/1886 C	13
65/1399 C	14
62/2127 A	15 (a & b)
67/210 B	16
65/1585 A	17
11/63 $\frac{1}{8}$" x $\frac{3}{8}$"	18
11/63 6" x $\frac{1}{8}$"	19
64/930 B6	20
62/1168 A	21

Counts per minute x 10^5

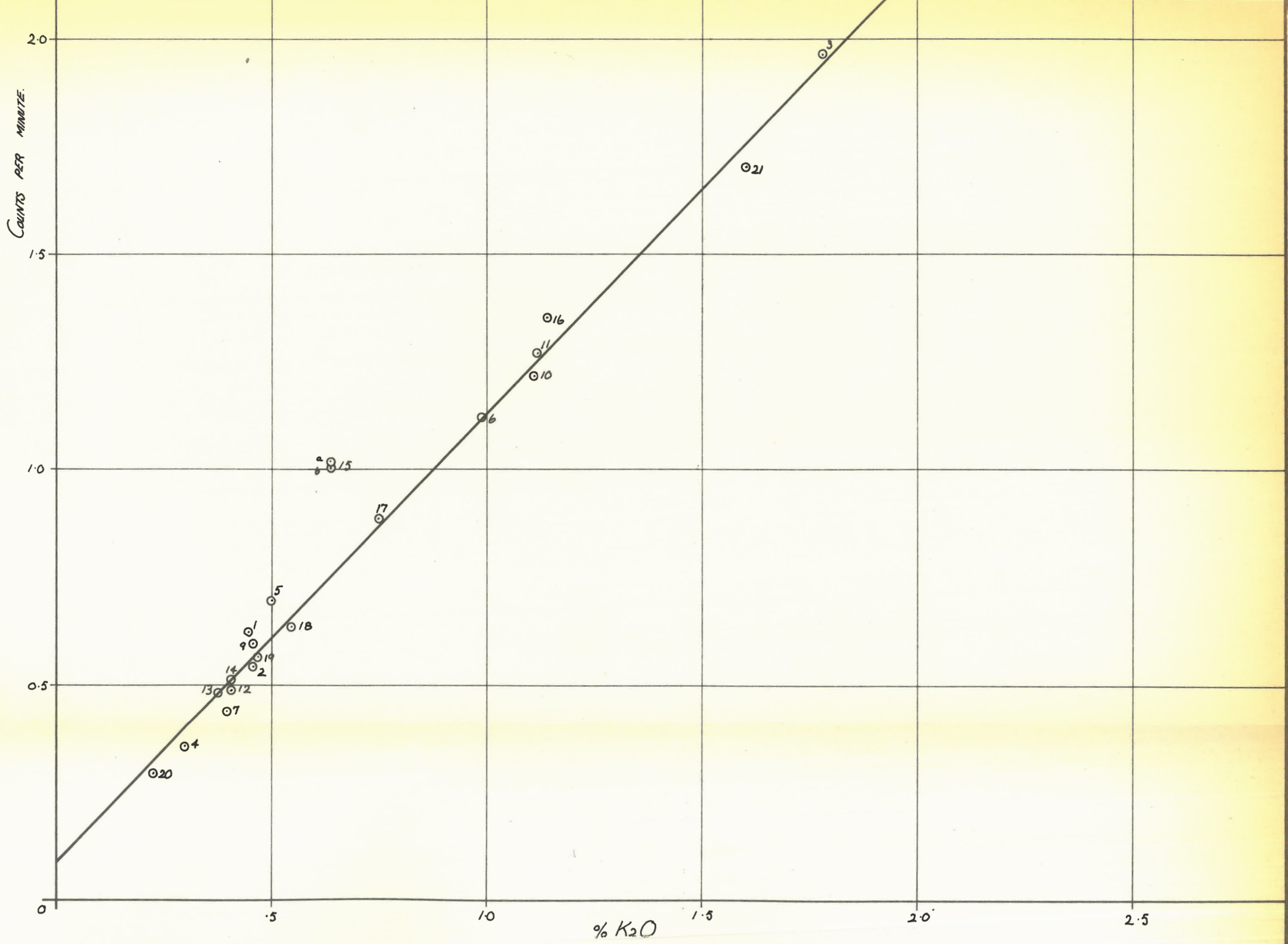
1·3
1·2
1·1
1·0
0·9
0·8
0·7
0·6
0·5
0·4
0·3

20
25

Sample No.	No. on graph.
65/1585 B	1
63/931 D	2
63/814 C	3
65/1551 A	4
65/1551 B	5
62/584 B	6
65/1551 C	7
5639	8 (a & b)
63/461 D	9
62/584 D	10
63/585 B	11
65/1880 C	12
65/1886 C	13
65/1399 C	14
62/2127 A	15 (a & b)
67/210 B	16
65/1585 A	17
11/63 $\frac{1}{8}$" × $\frac{3}{8}$"	18
11/63 6" × $\frac{7}{8}$"	19
64/390 B_b	20
62/1168 A	21

FIG. 12.

FUSED DISCS.


COUNTS PER MINUTE.
2·0
1·5
1·0
0·5
0
·5
1·0
1·5
2·0
2·5
% K2O
3
21
16
11
10
6
8
9
15
17
5
1
18
9
19
2
14
13
12
7
4
20

SAMPLE No.	No ON GRAPH.
65/1585 B	1
63/931 D	2
63/814 C	3
65/1551 A	4
65/1551 B	5
62/584 B	6
65/1551 C	7
5639	8 (a & b)
63/461 D	9
62/584 D	10
63/585 B	11
65/1880 C	12
65/1886 C	13
65/1399 C	14
62/2127 A	15 (a & b)
67/210 B	16
65/1585 A	17
11/63 $\frac{1}{8}$" x $\frac{3}{8}$"	18
11/63 6" x $\frac{7}{8}$"	19
64/390 B_6	20
62/1168 A	21

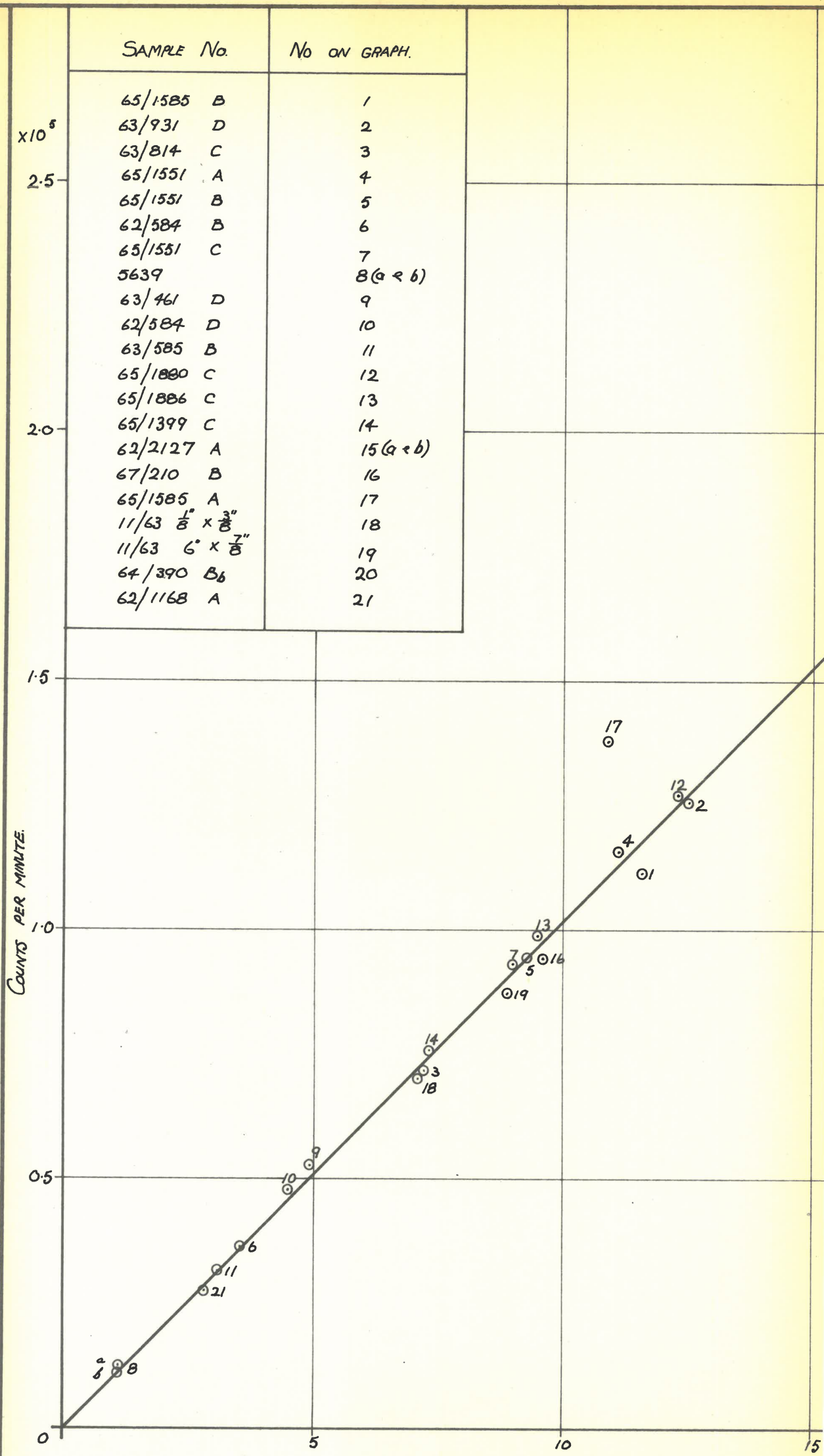
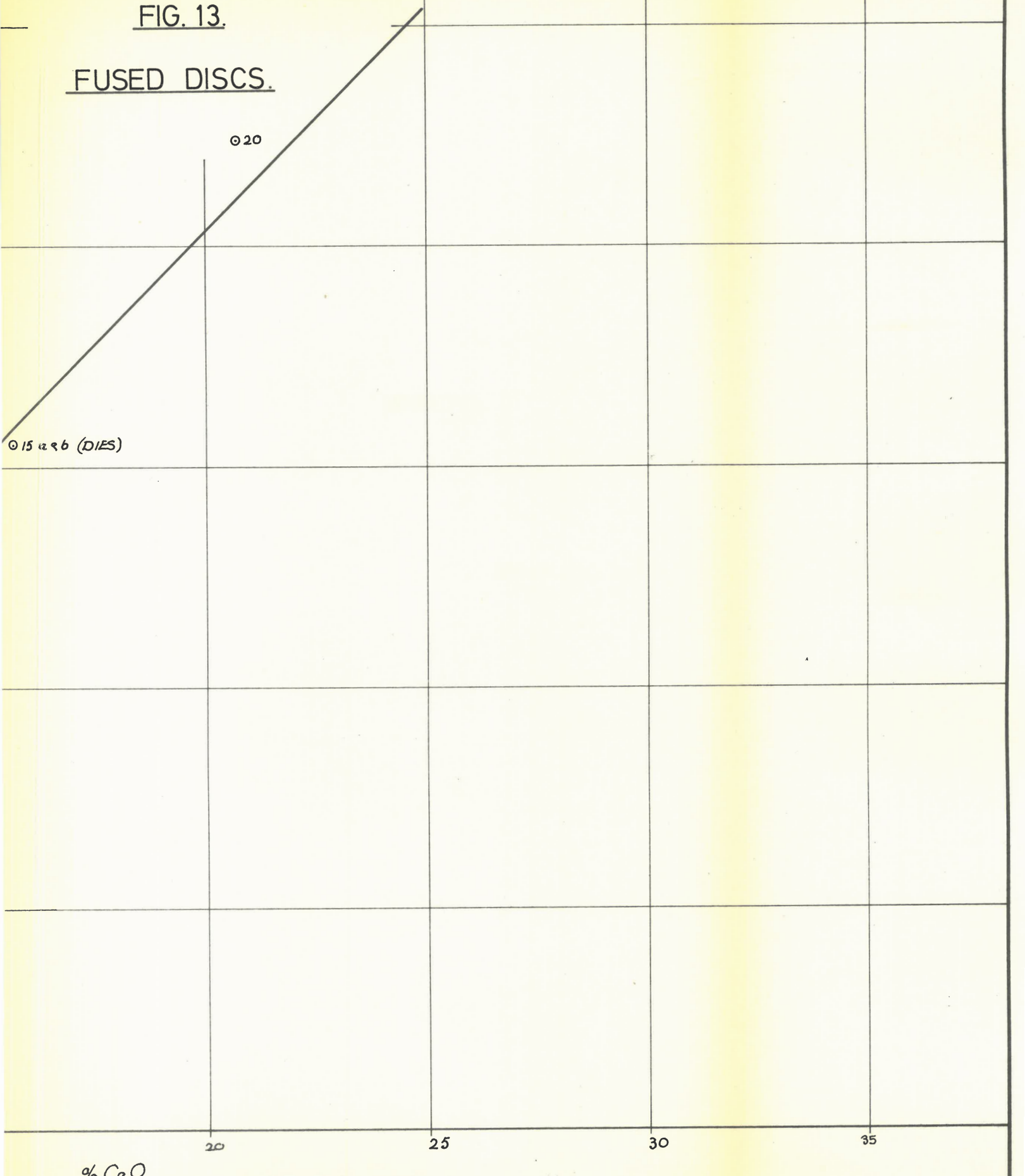


FIG. 13.

FUSED DISCS.



SAMPLE No.	No. ON GRAPH.
65/1585 B	1
63/931 D	2
63/814 C	3
65/1551 A	4
65/1551 B	5
62/584 B	6
65/1551 C	7
5639	8 (a & b)
63/461 D	9
62/584 D	10
63/585 B	11
65/1880 C	12
65/1886 C	13
65/1399 C	14
62/2127 A	15 (a & b)
67/210 B	16
65/1585 A	17
11/63 $\frac{1}{8}$" x $\frac{3}{8}$"	18
11/63 6" x $\frac{7}{8}$"	19
64/390 B_b	20
62/1168 A	21

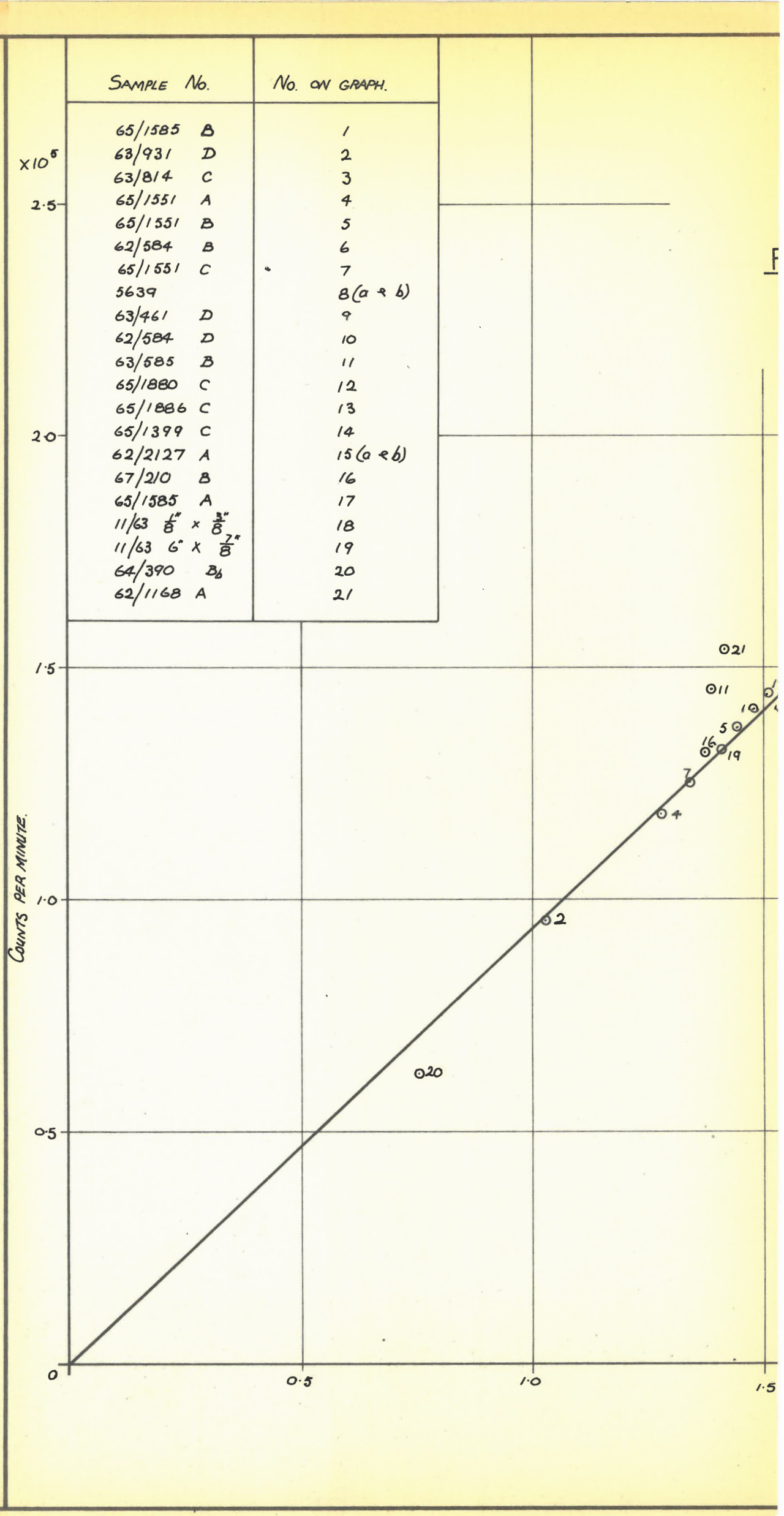
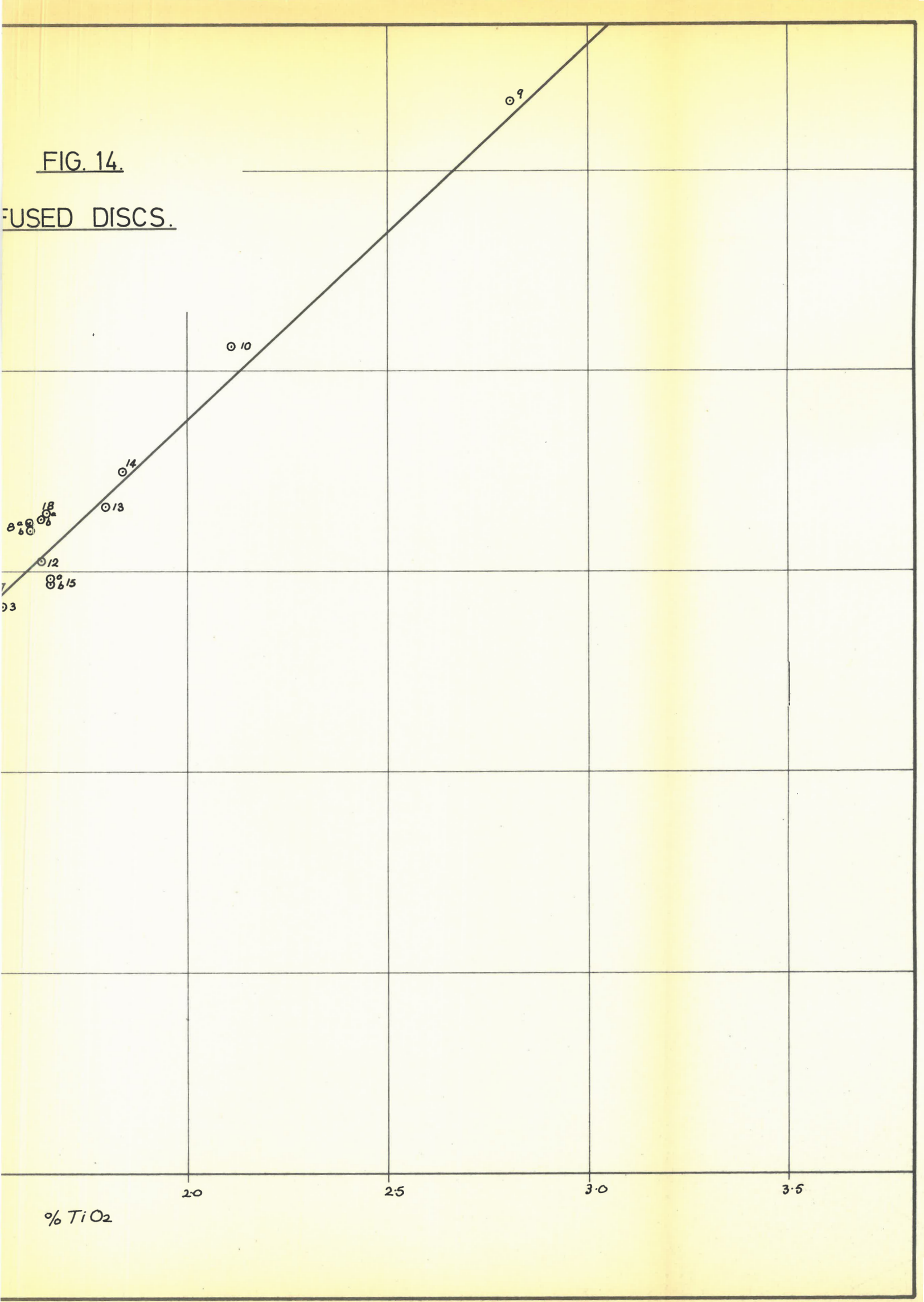


FIG. 14.

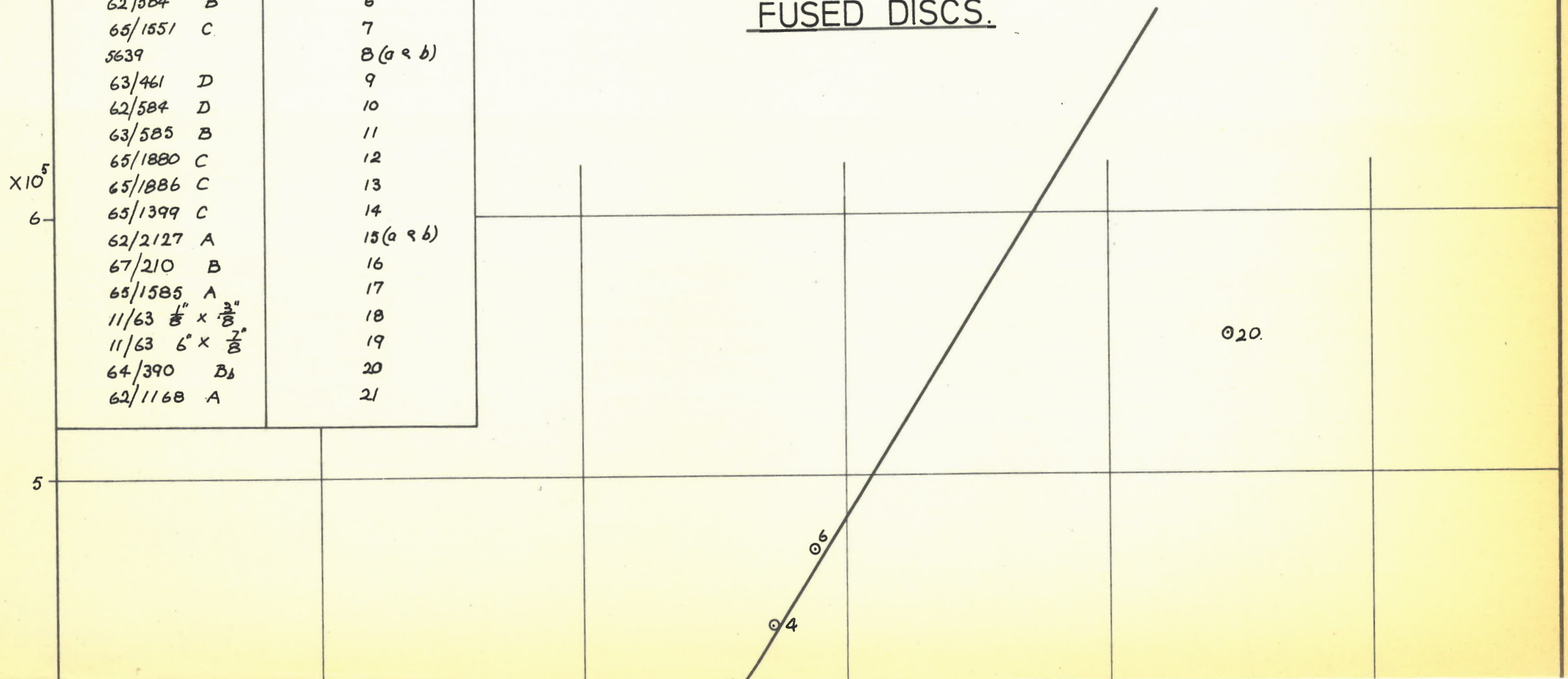
FUSED DISCS.

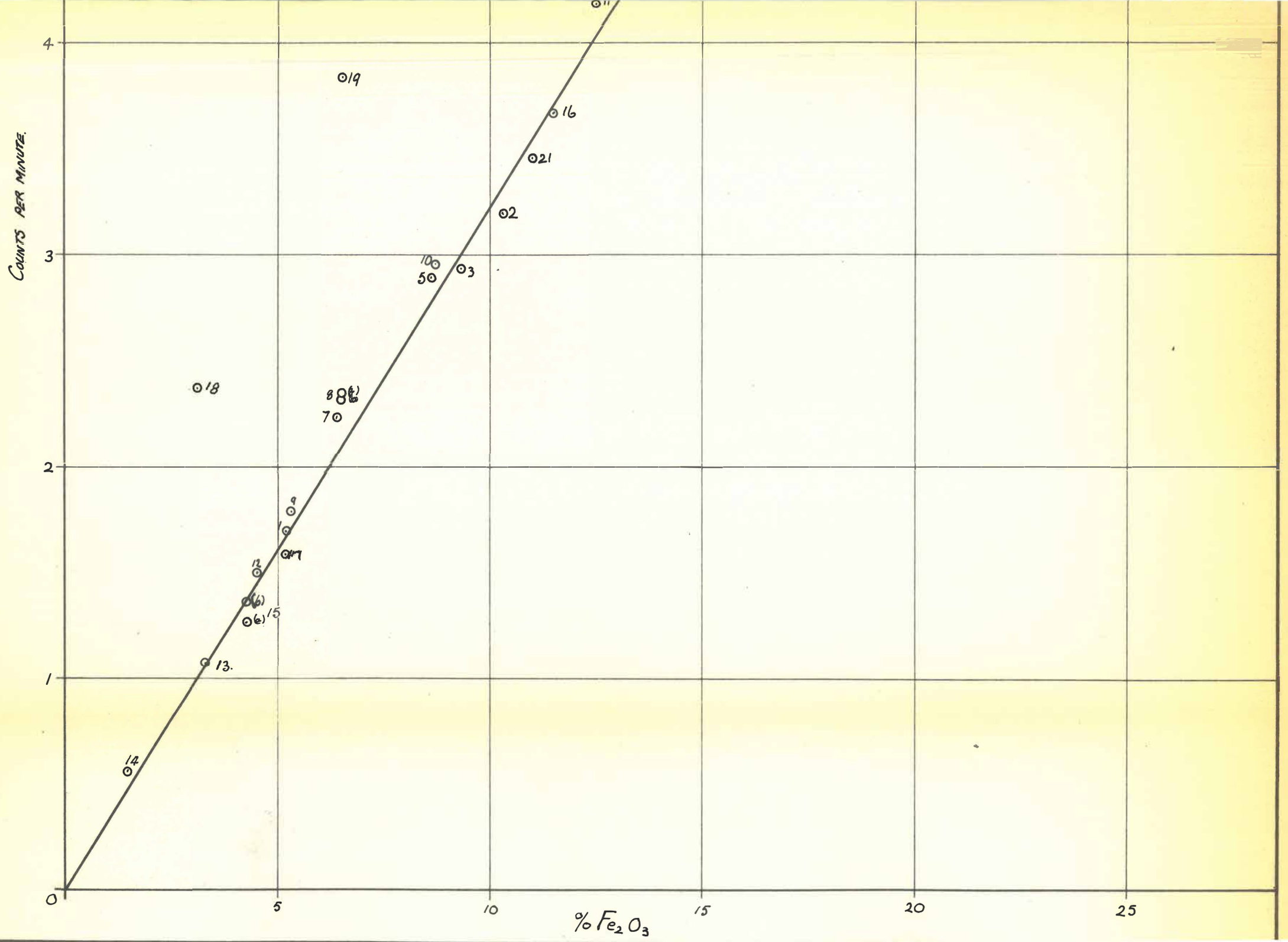


SAMPLE No.	No. ON GRAPH.
65/1585 B	1
63/931 D	2
63/814 C	3
65/1551 A	4
65/1551 B	5
62/584 B	6
65/1551 C	7
5639	8 (a & b)
63/461 D	9
62/584 D	10
63/585 B	11
65/1880 C	12
65/1886 C	13
65/1399 C	14
62/2127 A	15 (a & b)
67/210 B	16
65/1585 A	17
11/63 $\frac{1}{8}$" x $\frac{3}{8}$"	18
11/63 6" x $\frac{7}{8}$"	19
64/390 B$_b$	20
62/1168 A	21

FIG. 15.

FUSED DISCS.




COUNTS PER MINUTE
% Fe_2O_3
0
1
2
3
4
5
10
15
20
25
19
16
21
2
100
50
3
18
88
70
9
1
7
12
6
6
15
13.
14