

QUANTITATIVE X-RAY DIFFRACTION  
AND FLUORESCENCE ANALYSIS OF  
PAINT PIGMENT SYSTEMS

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## INTRODUCTION

The X-ray diffractometer has been used for several years in our laboratory to obtain rough estimates of pigment composition in paint. The use of the instrument for precise calculations has been excluded because the relationship of instrumental peak heights to weight per cent of pigments does not correlate directly. In one example, a 50/50 mixture by weight of titanium dioxide ( $\text{TiO}_2$ ) and iron oxide ( $\text{Fe}_2\text{O}_3$ ) resulted in net intensities of 432 counts per second (cps) for  $\text{TiO}_2$  and 281 cps for  $\text{Fe}_2\text{O}_3$ . Expressed as per cent of total counts, the titanium dioxide contributed 61%, while iron oxide contributed 39% of the counts. It is evident from these and other results<sup>1</sup> that not all diffraction patterns are of the same intensity.

The presence of many components in a prepared paint would cause a myriad of small constructive and destructive effects on the fluorescence intensity of any given pigment, resulting in an analytical result of unknown deviation from the actual composition. This project was intended to develop a procedure for preparing and treating samples and data which would result in more accurate analyses of common paint types by X-ray techniques.

## SCOPE

This study attempted to correlate measured X-ray intensities with concentrations of each member of paint pigment systems, thereby establishing calibration curves for the quantitative analyses of such systems.

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<sup>1</sup>R. K. Scott, "The Case for a Universal X-Ray Diffraction Intensity Scale," Fifty Years of Progress in Metallographic Techniques, ASTM STP.430, Am. Soc. Testing Mats., 1968, pp. 201-203.

## METHODOLOGY

Work was first done to determine reproducibility and predictability of results with pigment mixtures and paint films. Then model systems were prepared using pigments common to highway paint formulae. These systems, which included both dry pigment mixtures and prepared paint samples, were analyzed by X-ray diffraction and fluorescence. A study of one group of paints utilizing scanning electron microscopy with energy dispersive analysis (SEM-EDS) was included to give added information on the nature of the variance between true composition and X-ray results.

### I. Reproducibility of Results as Affected by Instrumental Factors and Operator Handling

A copper disc was used to study repeatability of the fluorescence spectrophotometer in testing of a standard remaining in place through a series of counts.

Further, a quartz crystal was used by the service representative to show repeatability of results on the diffractometer where a standard is removed and replaced for each of a series of runs. This removal procedure is not necessary for the fluorescence spectrophotometer, since the sample spins constantly and no particular holder orientation would be a factor in counting.

A final study of count rate repeatability was carried out on pigment peaks (for both diffraction and fluorescence). This study included both orientation and packing effects for the diffractometer.

In the diffraction study, three pigments were subjected to the following series of treatments:

1. Holder was packed with given pigment, placed in X-ray diffractometer, counted ten times at the major peak, each count lasting ten seconds. The peak to background ratio was also determined. This treatment shows variation in machine counting only.
2. Holder was removed, replaced, and recounted ten times. This treatment shows effect of slight changes in the area of excitation (orientation of holder).
3. Same as 2, except that with each removal, the holder was emptied and repacked before recounting. These conditions show effects of slight compositional changes due to packing.

For all runs in Part I, the standard deviation (S) and coefficient of variation (CV) were computed by the following formulae:

$$S = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n - 1}}$$

$$C.V. (\%) = \frac{S}{\bar{x}} \times 100$$

In these equations,  $\bar{x}$  is the mean of the individual readings,  $\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i$



## II. Predictability of Results as Influenced by Various Methods of Specimen Preparation

This portion of the work was concerned with preparing samples to minimize effects of particle size and particle density. The work encompasses grinding methods, mixing methods, and application methods.

### A. Grinding Methods

1. Abbe pebble mill - Mixtures of titanium dioxide and zinc oxide at varying concentrations were ground for 10 minutes and x-rayed, followed by 15 additional minutes of grinding before the second run. These specimens were also run on fluorescence after 25 minutes of grinding.

2. Hand-ground samples prepared by 15 minutes of grinding by mortar and pestle were compared to the results of 1 above for advantages in predictability.

### B. Mixing Methods

1. Pressing of pigment discs at various pressures.

2. Various pigments at different concentrations in Rhoplex 388 binder, mixed in paint shaker.

3. Pigments in 2 above, but premixed by hand with small amounts of binder before shaking.

### C. Application Methods

1. Films were drawn down, at various thicknesses, using mixtures from part 3 of mixing methods.

2. Films were painted on two-way tape and removed for examination of both surfaces by SEM-EDS. Paints in this case were prepared by a paint company, employing ordinary production methods, on a small scale.

III. Model Systems - The paint systems chosen for evaluation are listed below, along with the experimental specimens prepared for each.

### A. Marine enamel, MS-62 requiring

Titanium dioxide	20.0%
Zinc oxide	46.7%
Magnesium silicate	33.3%
	<u>100.0%</u>

1. Seventeen mixtures of titanium dioxide and zinc oxide, Abbe mill ground.
2. Six mixtures of titanium dioxide and zinc oxide, hand ground.
3. Three mixtures of titanium dioxide and magnesium silicate, hand ground.
4. Three mixtures zinc oxide and magnesium silicate, hand ground.

5. Seven mixtures titanium dioxide, zinc oxide, and magnesium silicate, hand ground.
  6. One sample of marine enamel was subjected to centrifugation, and the pigment portion was washed, dried, and hand ground before X-ray analysis was performed.
- B. Basic Lead Silico Chromate, MS-85, requiring
- |                            |       |
|----------------------------|-------|
| Basic Lead Silico Chromate | 93.2% |
| Red Iron Oxide             | 6.3%  |
| Bentone 38 or equal        | 0.5%  |
1. Dry pigment mixtures in varying concentrations.
  2. Paint mixtures with pigment concentrations close to those of the specification, and using all other ingredients as required. Detailed formulation follows.
  3. A drawdown was made from stock paint and run for comparison to curves.

Materials Testing Laboratory Formulation No. 1

Material	Grams
Mix:	
I-4-3 Permax	635.0
RC 1681 Iron Oxide	43.0
Bentone 38	3.43
Raw Linseed Oil	129.6
Aroplaz 1266-M-70	181.3
Mineral Spirits	80.5
(Mixing loss: 4.0 grams)	
Ball mill grind 22 hrs.	
Fineness: 6 N. S.	

Thin:	
Mineral Spirits	85.8
6% Zircatalox	5.81
6% Mn Naphthenate	2.07
6% Co Naphthenate	1.04
Troykyd Antiskin	1.02
Methanol	1.50
Water	0.06
(Grinding and thinning loss: 8.0 grams)	

Consistency: 69 Krebs Units  
 Wt./gal.: 13.60 lbs.

Drying: Sets in 1:35 hrs., sunny, 26.8° C.  
 Color: Lighter and more yellow than standard.

Materials Testing Laboratory Formulation No. 2

	Material	Grams
Mix:	I-4-3 Permox	615.1
	RC 1681 Iron Oxide	64.9
	Bentone 38	3.42
	Raw Linseed Oil	129.6
	Aroplaz 1266-M-70	181.3
	Mineral Spirits	10.0
	(Mixing loss: 4.1 grams)	
	Ball mill grind 24 hrs.	
	Fineness: 5 1/2 N. S.	

Thin:

Mineral Spirits	39.5
6% Zircatalox	5.84
6% Mn Naphthenate	1.97
6% Co Naphthenate	0.98
Troykyd Antiskin	0.97
Methanol	1.49
Water	0.11
(Grinding and thinning loss: 9.3 grams)	

Consistency: 82 Krebs Units

Wt./gal.: 15.45 lbs.

Color: Lighter and more yellow than standard.  
(Slightly darker than #46)

Materials Testing Laboratory Formulation No. 3

	Material	Grams
Mix:	I-4-3 Permox	652.6
	RC 1681 Iron Oxide	23.8
	Bentone 38	3.38
	Raw Linseed Oil	129.6
	Aroplaz 1266-M-70	181.3
	Mineral Spirits	10.0
	(Mixing loss: 4.2 grams)	
	Ball mill grind 22 hrs.	
	Fineness: 5 1/2 N. S.	

Materials Testing Laboratory Formulation No. 3  
(continued)

Thin:	Material	Grams
	Mineral Spirits	65.3
	6% Zircatalox	5.83
	6% Mn Naphthenate	1.97
	6% Co Naphthenate	0.98
	Troykyd Antiskin	0.99
	Methanol	1.48
	Water	0.08
	(Grinding and thinning loss: 6.7 grams)	

Consistency: 76 Krebs Units  
Wt./gal.: 14.86 lbs.

Drying: Set, streak free, slight tack, after 5 hrs.,  
sunny, 28° C. Slight tack after overnight dry.

Color: Much lighter and more yellow than standard and  
also much lighter and more yellow than #46 or #47.

- C. Traffic Stripe, Fast-dry, MS-87, with no pigment compositional requirements. Test paints varying pigment concentrations and pigment mixtures with the following formulations were tested:

Yellow Traffic Stripe

<u>Mixture</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Medium Chrome Yellow Pigment (%)	30	10	30	10
Magnesium Silicate (%)	5	5	25	25
Calcium Carbonate (%)	65	85	45	65
 <u>Mixture</u>	 <u>5</u>	 <u>6</u>	 <u>7</u>	 <u>8</u>
Titanium Dioxide (%)	10	10	30	30
Magnesium Silicate (%)	25	5	25	5
Calcium Carbonate (%)	65	85	45	65

- D. Vinyl Topcoat for Organic Zinc Bridge System, LDH Spec. 358-006, having no pigment compositional requirements. Paints were prepared with the following pigment compositions:

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
Titanium dioxide (%)	36.2	40.9	36.8	40.8	38.7
Talc 57 (%)	53.6	49.6	53.9	48.9	51.7
Celite 499 (%)	8.0	7.4	7.4	8.2	7.7
Aerosil 200 (%)	1.3	1.2	1.2	1.2	1.2
Lampblack (%)	0.3	0.3	0.3	0.3	0.3
Chrome Yellow (%)	0.6	0.6	0.6	0.6	0.6

#### IV. Treatment of Data

Various methods were employed for ascertaining the most useful representation of data.

1. Peak count rates were plotted against wt. %.
2. Ratios of peak counts of various components were plotted against ratios of wt. % of various components.
3. Integrated peak area as counted by the instrument was related to wt. % of a given component.
4. Ratios were made of count rates against a 100% substance and plotted against wt. percent.
5. Peak areas as measured with a planimeter were related to dry % pigment in a paint film.

For both diffraction and fluorescence, the most useful representations for data appeared to be obtained when compositional ratios were plotted against intensity ratios.

For diffraction, the ratio of the weight per cent of one pigment to the sum of the other weight per cents was plotted against the ratio of the intensity of that pigment to the sum of the other intensities. In fluorescence, more predictable patterns emerged by plotting elemental wt. per cent ratios, using one element in each compound, to the intensity ratios.

## DISCUSSION OF RESULTS

### I. Instrumental Factors and Operator Handling

The data from determinations of count rate variation (Tables I & II) show that the instrument is precise in repeating solid standards (quartz and copper).

For our types of samples, the tables also show that a much better coefficient of variation is obtained by fluorescence than diffraction. The varied alignment of crystal planes due to repacking causes the diffractometer results to change slightly. However, the elemental composition of the compounds is more constant; hence the lower coefficient of variation in fluorescence.

In certain compounds, such as talc, the tendency for crystals to align in one plane is very pronounced, causing a plating effect. This plating nature increases the proportion of X-rays diffracted from one plane. The result is that the major peak proportions for a compound will shift from the values in the powder diffraction table. The table gives relative intensities of peaks diffracted by a sample with a random particle arrangement.

A demonstration of this preferred alignment is given below, in a set of diffraction intensities from traffic paint:

	Paint #			
	5	6	7	8
Talc Peak 1 (9.55°)	29893	5284	31403	11655
Talc Peak 2 (28.75°)	25716	4448	21587	8391
Titanium dioxide Peak 1	4395	4732	13785	14208
Calcium carbonate Peak 1	67616	50641	34834	78618
<u>Talc Peak 1</u>	1.16	1.18	1.45	1.39
<u>Talc Peak 2</u>				
<u>Talc Peak 1</u>	0.54	0.54	0.59	0.58
<u>Talc Peak 1 &amp; Talc Peak 2</u>				

In #7 and #8, there is a preference for the plane diffracted at 9.55°. The degree of plating is apparently affected by the concentrations of other components.

The pressing of discs enhances preferred alignment of talc, causing further variations in count rate (Table I8), depending on the degree of random orientation present before pressing.

One effort made to decrease the counting variation for diffraction was to use the average of three determinations for each component in computing intensity ratios. This practice

decreased the coefficient of variation for treatment 3 (Table 1) to 3.8% for titanium dioxide, 4.6% for zinc oxide, and 9.0% for talc. It is to be noted that these percentages refer to count rate, not to per cent pigment.

Taking actual intensity figures for a given mixture and altering the talc count rate by subtracting 9%, we calculated composition from the solving of simultaneous equations using Graphs 7.1-7.3:

	True composition	Computation from Graph Readings	Theoretical at 91% of Talc intensity as 100%
Talc	39.8	41	39
Titanium dioxide	19.9	18	19
Zinc oxide	40.3	40	42

In all probability, the count rate would vary less than 9.0%, since Table 1 was taken on pure compounds, and count rate is more stable at lesser concentrations of talc. Also, it is likely that if talc counts low, the other two components will also count low, resulting in less change in ratios.

We have explained here the maximum expected variance by projecting onto composition near specification the most unstable count rate of a pure compound. The fact that these figures were part of the least accurate of our experiments, the dry pigment portion, is also important. It is evident that we may operate with a low counting error allowance in the laboratory and be certain of a fair test, using wet standards as described elsewhere in this paper.

## II. Specimen Preparation

The Abbe grinder did not show a clear-cut advantage over mortar and pestle (Tables 3 & 4). Also, the 10-minute grinding time yielded samples with a correlation coefficient (R) which compared favorably to that of the samples ground 25 minutes [.999 (Graph 3-1) vs. .991 (Graph 3-5) for  $\text{TiO}_2/\text{ZnO}$ ]. From the appearance of the distribution it looks as if loss sometimes occurred in the second transfer of material, which was not representative of the entire sample (Table 3).

Our attempts at mixing pigment into a binder were aborted because wet standards were obtained from a paint company which contained all other paint ingredients for the given specification. This type of standard removes from consideration the ease of dispersion of a given pigment in a given binder, the use of wetting agents, and the determination of mixing times necessary for a given system. Some data from mixing is included, showing variance of intensities and peak area, with total pigment content and film thickness (Tables 16 & 17). These tables further delineate some mixing problems.

These prepared paint standards would obviously be preferable to dry pigment mixtures, since the time for pigment extraction is saved. The differences between dried pigment samples and prepared paints are illustrated in the Basic Lead Silico Chromate and Traffic Paint Data. The establishment of an entire paint matrix according to the specification being evaluated is recommended for reasons of time and accuracy (Tables 8, 12, 13, Graphs 8.1-13.8).

### III. Model Systems

#### A. Marine Enamel

Difficulty was encountered in the analysis of dry mixtures. While the binary mixtures of zinc oxide and titanium dioxide showed good correlation coefficients (Tables 3 & 4), other mixtures showed variable correlation tendencies (Tables 5-7). It also became clear that a paint from stock could not be tested against the curves for dry mixtures (Table 7). Further, it appears that fluorescence is less accurate than diffraction for this system, regardless of the plating effect of talc in diffraction (Graphs 7-1 through 7-6). Quality control testing for this system would have to be attempted from the standpoint of prepared paint standards, as proved beneficial with later systems.

#### B. Basic Lead Silico Chromate

In this system, prepared paint standards were first used. Pigment mixtures were compared to the paint samples, and slightly higher intensity ratios were obtained for pigment in fluorescence (Table II). Fluorescence appeared to correlate slightly better for this system (Graphs 8-1 through 8-8), and the paint from stock would be analyzed as 5.3% iron oxide and 94.7% lead chromate, results which were very close to specification.

Also from this system, it appears from Table 9 that integrated peak area is also very accurate for measuring large concentrations of an element.

Finally, the SEM-EDS study (Tables 10 & 11) gives evidence that the smaller area studied, the larger variance from concentrations will be obtained. Small fluctuations in pigment composition do occur, but these fluctuations are important only where components present in small concentrations are critical to paint performance. In the Basic Lead Silico Chromate System, iron oxide is not the active ingredient; therefore, the lead silico chromate pigment may be measured and the iron oxide calculated by difference, if necessary.

The SEM-EDS study also shows the difference between the surface of paint exposed to the air while drying and that touching the substrate (Table II). Each mixture was applied to double-stick tape and allowed to dry. The films were removed from the tape and both surfaces were analyzed. The substrate surface (tape side) of the films did show slightly more accurate results. The photographs of both surfaces (Figures 1-4) of one paint show the tape residue and appearance of a small area of the film.

Photographs of the other two paints were similar to these.

#### C. Traffic Paint

All curves for prepared paint samples showed correlation coefficients (R) of .95 or better (Graphs 12:odd). The pigment mixtures, on the other hand, had some curves which ran lower, (Graphs 12:even). Compositional requirements for quick-dry traffic paint pigment will be established during the coming year so that control testing can become a matter of monitoring stability of standard curves and checking new samples by the curve equations.



#### D. Vinyl Topcoat

R values showed that fluorescence testing of these samples is not useful (Table 15, Graphs 15.1 and 15.2). Diffraction has a degree of usefulness (Table 14, Graphs 14.1 and 14.2); however, any pigment requirements established for this system would probably require slightly wider ranges for acceptance than traffic paint.

## CONCLUSIONS

The data indicate that the X-ray fluorescence spectrophotometer can be used for routine analysis of pigment composition in paint. It is most useful in testing systems where maxima or minima for all pigments comprise the requirements, or where acceptable ranges for each pigment are listed.

Further points to be noted in considering these conclusions are as follows:

- a. Paint standards varying only pigment compositions were used in establishing optimum standard curves.
- b. All data for this study were obtained by the same technician, whose work is deemed of routinely acceptable quality. The work is, therefore, representative of the results our laboratory will obtain using our facilities. It is likely that others will find that their results will vary from those in this paper.
- c. It is realistic to assume that a specific portion of a drawdown may vary slightly from the percentages of pigments present in a larger paint sample. This is one point in favor of pigment ranges in specifications, and for counting allowances for maxima or minima criteria.
- d. Whether a particular specification is to be tested by diffractometry or fluorescence spectrophotometry depends on the accuracy of each instrument for that analysis. Questions of peak overlap in diffraction and of absorption or enhancement in fluorescence will influence this decision.

## IMPLEMENTATION

Specifications under which large volumes of paint are purchased are to be arranged for X-ray analysis, beginning with quick-dry traffic paint this year. This will include establishment of pigment composition requirements for some specifications, and obtaining wet standards for all specifications.

## TABLES

I. Reproducibility of Results as Affected by Instrumental Factors and Operator Handling

TABLE I: Diffraction\*

SPECIMEN TREATMENT	QUARTZ --	TiO <sub>2</sub>			ZnO			MgSiO <sub>3</sub>		
		1	2	3	1	2	3	1	2	3
P/B	-----	94.2	87.6	97.5	116.3	113.0	117.4	45.5	50.0	49.2
n	9	10	10	8	10	10	10	10	10	7
$\bar{x}$	30311	32237	29078	33741	98368	91946	95823	86236	94332	97294
x	30770	32320	29490	33570	98960	93950	96830	86630	92530	82870
	30320	32100	28740	29720	98730	91820	92140	86720	93110	100430
	30540	32110	29100	33170	98490	95290	93720	86600	95370	83880
	29860	32110	28070	34710	98480	95230	101210	86540	93040	75540
	29460	32220	28610	31320	98580	92830	105200	86330	95770	119760
	30990	32240	29930	35580	98100	92760	87270	86280	97130	106350
	30570	32240	29960	37290	98480	92570	108920	85970	93520	112230
	29440	32410	28850	34570	97560	88050	105940	85960	90930	-----
	30850	32160	28830	-----	98860	89900	80330	85710	96270	-----
	-----	32460	29200	-----	97440	87060	86670	85620	95650	-----
S	588	122	592	2390	516	2810	10983	398	1975	13422
C.V. (%)	1.9	0.4	2.0	7.1	0.5	3.1	11.4	0.5	2.1	13.8

\* Counts per 10 seconds

TABLE 2: Fluorescence \*

SPECIMEN	COPPER	TiO <sub>2</sub>	ZnO	MgSiO <sub>3</sub>	
	300 (Cu)	147 (Ti)	259 (Zn)	75 (Si)	1.4 (Mg)
P/B	10	10	3	3	3
$\bar{x}$	253390	21771	726713	377393	21437
x	25357	21783	724240	377393	21437
	25329	21786	727940	375060	21370
	25421	21709	727960	377850	21390
	25370	21814		379270	21550
	25377	21803			
	25326	21757			
	25327	21776			
	25318	21728			
	25322	21809			
	25329	21741			
S	49	36	2142	2142	99
C.V. (%)	0.19	0.17	0.29	0.57	0.46

\* Counts per 10 seconds

## II. Predictability of Results as Influenced by Various Methods of Specimen Preparation

### Grinding Methods

TABLE 3: Mill ground,  $\text{TiO}_2 + \text{ZnO}$  mixtures

MIX NO.	Grinding Time (minimum)	% $\text{TiO}_2$	%ZnO	Diffraction		Fluorescence	
				$\frac{I_{\text{TiO}_2}}{I_{\text{ZnO}}}$	$\frac{I_{\text{ZnO}}}{I_{\text{TiO}_2}}$	$\frac{\text{Ti}}{\text{Zn}}$	$\frac{\text{Zn}}{\text{Ti}}$
1	10	10.3	89.7	.04	25.78		
	25	10.3	89.7	.14	7.33	.17	5.73
2	10	15.1	84.9	.06	17.67		
	25	15.1	84.9	.15	6.49	.26	3.78
3	10	20.2	79.8	.21	4.66		
	25	20.2	79.8	.19	5.33	.36	2.78
4	10	25.0	75.0	.29	3.39		
	25	25.0	75.0	.27	3.15	.48	2.10
5	10	30.2	69.8	.39	2.56		
	25	30.2	69.8	.40	2.49	.67	1.48
6	10	35.2	64.8	.44	2.27		
	25	35.2	64.8	.36	2.82	.81	1.22
7	10	40.0	60.0	.56	1.79		
	25	40.0	60.0	.51	1.97	1.01	.99
8	10	45.0	55.0	.68	1.47		
	25	45.0	55.0	.69	1.45	1.24	.80
9	10	50.1	49.9	.83	1.21		
	25	50.1	49.9	.82	1.22	1.52	.66
10	10	55.1	44.9	.94	1.06		
	25	55.1	44.9	.92	1.09	1.82	.55
11	10	60.1	40.0	1.24	.80		
	25	60.1	40.0	1.31	.76	2.27	.44
12	10	70.0	30.0	1.78	.56		
	25	70.0	30.0	1.92	.52	3.31	.30
13	10	74.9	25.1	2.38	.42		
	25	74.9	25.1	2.46	.41	4.26	.23
14	10	80.0	20.0	3.34	.30		
	25	80.0	20.0	3.93	.25	5.65	.18
15	10	90.0	10.0		----		
	25	90.0	10.0	6.67	.15	14.36	.07

TABLE 4: HAND MIXED  $TiO_2 + ZnO$ 

Mix Number	% $TiO_2$	% ZnO	Diffraction		Fluorescence	
			$\frac{I_{TiO_2}}{I_{ZnO}}$	$\frac{I_{ZnO}}{I_{TiO_2}}$	$\frac{I_{Ti}}{I_{Zn}}$	$\frac{I_{Zn}}{I_{Ti}}$
I	14.4	85.6	.138	7.27	.28	3.57
II	29.2	70.8	.327	3.06	.70	1.43
III	42.7	57.3	.509	1.96	1.02	.78
IV	57.2	42.8	.978	1.02	1.53	.65
V	71.3	28.7	2.100	.48	2.52	.40
VI	85.8	14.2	4.159	.24	5.50	.18

TABLE 5: HAND MIXED MAGNESIUM SILICATE + ZINC OXIDE

Mix Number	% Talc	% ZnO	Diffraction		Fluorescence	
			$\frac{I_{Talc}}{I_{ZnO}}$	$\frac{I_{ZnO}}{I_{Talc}}$	$\frac{I_{Mg}}{I_{Zn}}$	$\frac{I_{Zn}}{I_{Mg}}$
VII	32.7	67.3	1.05	0.95	0.69	1.45
VIII	30.8	69.2	.66	1.51	0.58	1.72
IX	40.0	60.0	.87	1.15	0.92	1.09
X	20.0	80.0	.42	.90	0.31	3.21
XI	50.0	50.0	2.29	.43	1.16	0.86

TABLE 6: HAND MIXED MAGNESIUM SILICATE + TITANIUM DIOXIDE

Mix Number	% Talc	% $TiO_2$	$\frac{I_{Talc}}{I_{TiO_2}}$	$\frac{I_{TiO_2}}{I_{Talc}}$	$\frac{I_{Mg}}{I_{Ti}}$	$\frac{I_{Ti}}{I_{Mg}}$	$\frac{I_{Si}}{Ti}$	$\frac{I_{Ti}}{Si}$
X	20.5	79.5	0.97	1.03	.01	111.74	.60	1.66
XI	30.7	69.3	1.90	0.53	.02	55.66	1.09	.92
XII	39.7	60.3	2.23	0.45	.03	33.22	1.85	.54

TABLE 7: HAND MIXED TITANIUM DIOXIDE (A) + ZINC OXIDE (B) + MAGNESIUM SILICATE (C)

Mix Number	%A	%B	%C	Diffraction			Fluorescence		
				$\frac{I_A}{I_B + I_C}$	$\frac{I_B}{I_A + I_C}$	$\frac{I_C}{I_A + I_B}$	$\frac{I_{Ti}}{I_{Zn} + I_{Mg}}$	$\frac{I_{Zn}}{I_{Ti} + I_{Mg}}$	$\frac{I_{Mg}}{I_{Ti} + I_{Zn}}$
XIII	29.6	49.9	20.5	.24	.78	.59	1.15	.85	.004
XIV	19.9	40.3	39.8	.16	.56	1.07	.51	1.84	.02
XV	40.1	39.9	20.0	.36	.57	.62	1.18	.83	.005
XVI	28.3	32.0	39.7	.25	.38	1.10	.69	1.35	.02
XVII	32.9	35.3	31.8	.32	.50	.74	---	---	---
Paint from Stock	---	---	---	.27	1.09	.36	---	---	---
Calculated	27.8	58.4	13.9						



TABLE 8  
Basic Lead Silico Chromate

Formula #	Specimen	% Fe <sub>2</sub> O <sub>3</sub>	% Pb Chromate	Diffraction		Fluorescence	
				$\frac{I_{\text{Pb Chromate}}}{I_{\text{Fe}_2\text{O}_3}}$	$\frac{I_{\text{Fe}_2\text{O}_3}}{I_{\text{Pb Chromate}}}$	$\frac{I_{\text{Pb}}}{I_{\text{Fe}}}$	$\frac{I_{\text{Fe}}}{I_{\text{Pb}}}$
46	test paint	6.3	93.2	1.4	.71	4.8	.21
47	test paint	9.5	90.0	1.2	.83	3.6	.28
48	test paint	3.5	96.0	3.1	.32	18.0	.06
46	pigment mixture	6.3	93.2	4.69	0.213	4.2	.24
47	pigment mixture	9.5	90.0	1.25	0.797	2.8	.36
48	pigment mixture	3.5	96.0	1.85	0.540	10.7	.09
Paint from stock		---	---			10.9	.09

TABLE 9  
Integrated Fluorescence Peak Area as a Function of  
Pigment Composition in Paint

Formula #	% Fe <sub>2</sub> O <sub>3</sub>	% Pb Chromate	$\frac{I_{\text{Pb Chromate}_{46}}}{I_{\text{Pure Pb Chromate}}}$	$\frac{I_{\text{Fe}_3\text{O}_{446}}}{I_{\text{Fe}_3\text{O}_4 \text{ pure}}}$
46	6.3	93.2	92.8	3.6
47	9.5	90.6	90.7	5.2
48	3.5	96.0	97.8	1.9

TABLE 10  
Energy Dispersive X-Ray Data  
from Duplicate Fields of  
Basic Lead Silico Chromate Paint Specimens

Field #	Magnification	Specimen	<u>Intensity Ratios</u>		
			Si/Pb	Si/Fe	Pb/Fe
1	x5000	48	.493	6.69	13.5
2	x5000	48	.749	6.38	8.5
3	x2000	48	.729	----	---
4	x2000	48	.575	----	---
1'	x200	46	.346	3.16	9.13
2'	x200	46	.296	2.69	9.09

TABLE II ENERGY DISPERSIVE X-RAY ANALYSIS  
OF BASIC LEAD SILICO CHROMATE SAMPLES

SUBJECT	MAG.	PIGMENT COMPOSITION	INTENSITY (PULSES)						
			Si	Pb	Cr	Fe	Total	%Pbt CrtSi	% Fe
# 48 Tape Side	x 100	96.0% BLSC 3.5% Fe <sub>2</sub> O <sub>3</sub>	5812	10795	1267	508	18382	97%	3%
# 47 Tape Side	x 100	90.0% BLSC 9.5 Fe2O3	5961	10066	1159	1631	18817	91%	9%
# 48 Air Side	x 100	96.0% BLSC 3.5 Fe2O3	7137	10440	1102	447	19126	98%	2%
# 47 Air Side	x 100	90.0 BLSC 9.5 Fe2O3	7102	9775	990	1234	19101	94%	6%
# 48 Tape Side	x 2000	96.0% BLSC 3.5% Fe2O3	7582	10883	1084	490	20039	98%	2%
# 47 Tape Side	x 2000	90.0% BLSC 9.5% Fe2O3	7647	10464	1145	1031	20287	95%	5%
# 48 Air Side	x 2000	96.0% BLSC 3.5% Fe2O3	6952	10760	1076	309	19097	98%	2%
# 47 Air Side	x 2000	90.0% BLSC 9.5% Fe2O3	6957	9685	1056	927	18625	95%	5%
# 46 Tape Side	x 100	93.2% BLSC 5.3% Fe <sub>2</sub> O <sub>3</sub>	6911	10542	1271	1075	19028	94%	6%

TABLE 12 TRAFFIC PAINT - FLUORESCENCE STUDY

Yellow Traffic Paint

Mix	% Chrome Yellow	% Talc	% CaCO <sub>3</sub>	$\frac{I_{Pb}}{I_{Si} + I_{Ca}}$	$\frac{I_{Si}}{I_{Pb} + I_{Ca}}$	$\frac{I_{Ca}}{I_{Pb} + I_{Si}}$
1	30	5	65	1.26	.02	.72
2	10	5	85	.30	.02	2.89
3	30	25	45	1.46	.08	.50
4	10	25	65	.36	.09	1.83

Yellow Traffic Pigment Mixtures

1	30	5	65	.43	.07	1.72
2	10	5	85	.12	.08	4.62
3	30	25	45	.37	.44	0.74
4	10	25	65	.13	.37	1.62

White Traffic Paint

	% TiO <sub>2</sub>	% Talc	% CaCO <sub>3</sub>	$\frac{I_{Ti}}{I_{Si} + I_{Ca}}$	$\frac{I_{Si}}{I_{Ti} + I_{Ca}}$	$\frac{I_{Ca}}{I_{Ti} + I_{Si}}$
5	10	25	65	.39	.09	1.73
6	10	5	85	.31	.02	2.84
7	30	25	45	1.28	.07	.61
8	30	5	65	.82	.02	1.13

Pigment Mixtures

5	10	25	65	.11	.38	1.63
6	10	5	85	.08	.08	6.00
7	30	25	45	.27	.41	1.00
8	30	5	65	.19	.08	3.34

TABLE 13

## Traffic Paint Diffraction Study

## Yellow Traffic Paint

Mix	% Chrome Yellow	% Talc	% CaCO <sub>3</sub>	Chrome Yellow Intensity
1	30	5	65	21525
2	10	5	85	9747
3	30	25	45	25888
4	10	25	65	11285

## Yellow Traffic Pigment Mixtures

1	30	5	65	19549
2	10	5	85	8077
3	30	25	45	21101
4	10	25	65	4637

## White Traffic Paint

Mix	% TiO <sub>2</sub>	% Talc	% CaCO <sub>3</sub>	$\frac{I_{TiO_2}}{Talc+CaCO_3}$	$\frac{I_{Talc}}{TiO_2+CaCO_3}$	$\frac{I_{CaCO_3}}{Talc+TiO_2}$
5	10	25	65	.17	.16	2.53
6	10	5	85	.15	.02	5.61
7	30	25	45	.60	.17	0.91
8	30	5	65	.56	.03	1.56

## White Traffic Pigment

5	10	25	65	.04	.77	1.13
6	10	5	85	.08	.18	3.50
7	30	25	45	.16	1.09	.52
8	30	5	65	.14	.22	2.30

TABLE 14

## Vinyl Topcoat Diffraction Study

Mixture #	% TiO <sub>2</sub>	% Talc	% Celite	$\frac{\text{TiO}_2}{\text{Talc}}$	$\frac{\text{Talc}}{\text{TiO}_2}$
1	36.2	53.6	8.0	.36	2.75
2	40.9	49.6	7.4	.54	1.85
3	36.8	53.9	7.4	.46	2.18
4	40.8	48.9	8.2	.57	1.76
5	38.7	51.7	7.7	.44	2.26

TABLE 15

## Vinyl Topcoat Fluorescence Study

	% TiO <sub>2</sub>	% Talc	% Celite	$\frac{I_{\text{Si}}}{I_{\text{Si}+ \text{Mg}}}$	$\frac{I_{\text{Ti}}}{I_{\text{Mg}+}}$	$\frac{\text{Mg}}{I_{\text{Ti}}}$
1	36.2	53.6	8.0	.88	15.6	.06
2	40.9	49.6	7.4	.88	22.0	.05
3	36.8	53.9	7.4	.87	13.5	.07
4	40.8	48.9	8.2	.88	14.5	.07
5	38.7	51.7	7.7	.87	9.6	.10

TABLE 16  
Peak Intensities of Titanium Dioxide in  
Rhoplex Binder

wt % TiO <sub>2</sub> (wet)	% TiO <sub>2</sub> (Dry film)	8 mil film	Net Intensities, cps	
			10 mil film	12 mil film
10	18	686	669	916
20	33	1003	990	961
30	46	753	1021	1019
40	63	cracked	916	750

TABLE 17  
Peak Area and Peak Intensity  
of Iron Oxide in Rhoplex Binder  
(10 mil films)

Mix	% Fe <sub>2</sub> O <sub>3</sub> (Wet)	% Fe <sub>2</sub> O <sub>3</sub> (Dry film)	Peak Area (sq. ")	Peak Intensity, cps
Ground Fe <sub>2</sub> O <sub>3</sub>	100	100	0.54	
Pigment	Binder	Water		
10g	- 40g	- 10g	0.42	375
32g	- 32g	- 10g	0.38	305
24g	- 48g	- 10g	0.48	353
30g	- 90g	- 0g	0.45	393

TABLE 18

Results of Compression on Pigment Intensity

Pellet #	kg	Time	Mean Intensity	n	S	C.V.
1	9,000	3 min.	179393	3	32837	18.1 %
2	20,000	3 min.	174580	3	5970	3.4 %
3	20,000	3 min.	210658	6	4870	2.3 %
4	20,000	3 min.	185333	6	855	0.5 %

The C. V. of the averages of the three discs prepared at same conditions (2-4) was 8.7%. Removal and recounting of each resulted in a combined C. V. of 15.1 %, similar to that of hand packed pigment in the powder holder.



## GRAPHS

Graph 3-1  
Marine Enamel System - Diffraction  
 $\%TiO_2/\%ZnO$  vs.  $I_{TiO_2}/I_{ZnO}$   
CC = .9990  
 $Y = .0312 + .7726x$   
Abbe grinder - 10 min.

$I_{TiO_2}/I_{ZnO}$

2

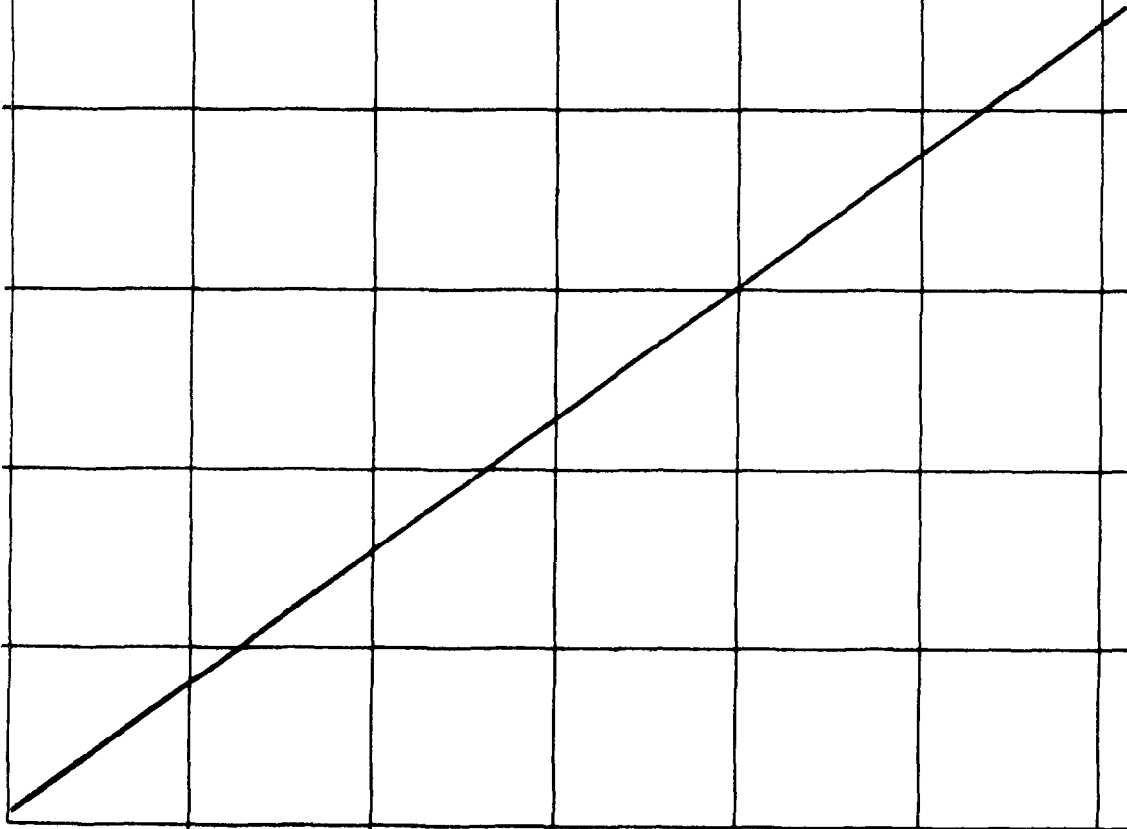
1

1.0

2.0

3.0

$\%TiO_2/\%ZnO$



Graph 3-2  
Marine Enamel System - Diffraction  
 $\%ZnO/\%TiO_2$  vs.  $I_{ZnO}/I_{TiO_2}$   
CC = .9793  
 $Y = .3410 + .9230x$

Abbe grinder - 10 min.

$I_{ZnO}/I_{TiO_2}$

2.0

1.0

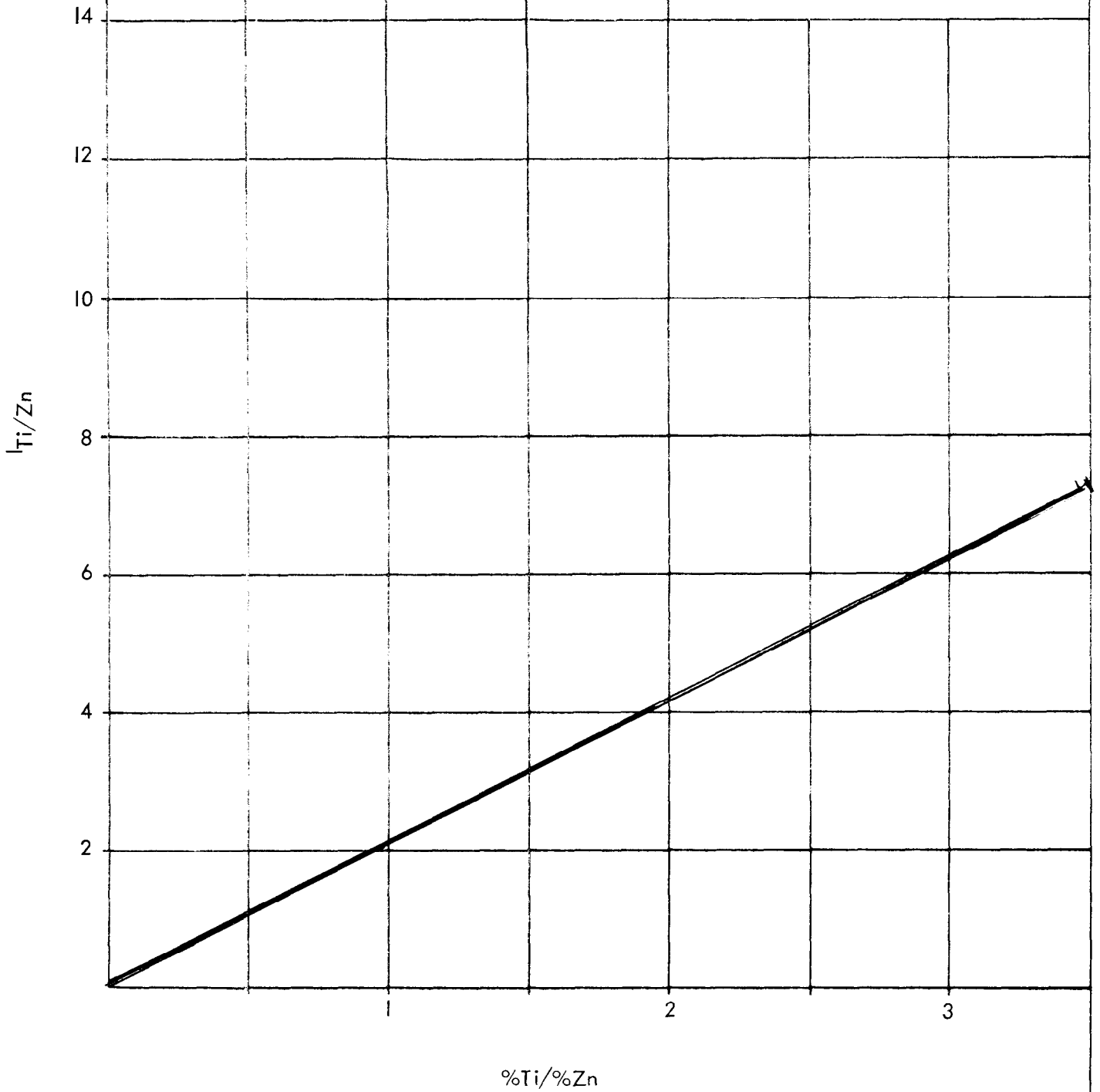
1.0

2.0

$\%ZnO/\%TiO_2$

Graph 3-3  
Marine Enamel System - Fluorescence  
%Ti/%Zn vs. I<sub>Ti</sub>/I<sub>Zn</sub>  
CC = .9983  
Y = .1076 + 2.1001x

Abbe grinder - 10 min.



Graph 3-4  
Marine Enamel System - Fluorescence  
%Zn/%Ti vs.  $I_{Zn}/I_{Ti}$   
CC = .9994  
 $Y = .0177 + .4960x$   
Abbe grinder - 10 min.

$I_{Zn}/I_{Ti}$

4

3

2

1

1

2

3

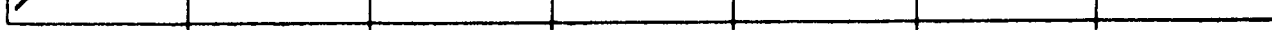
4

5

6

7

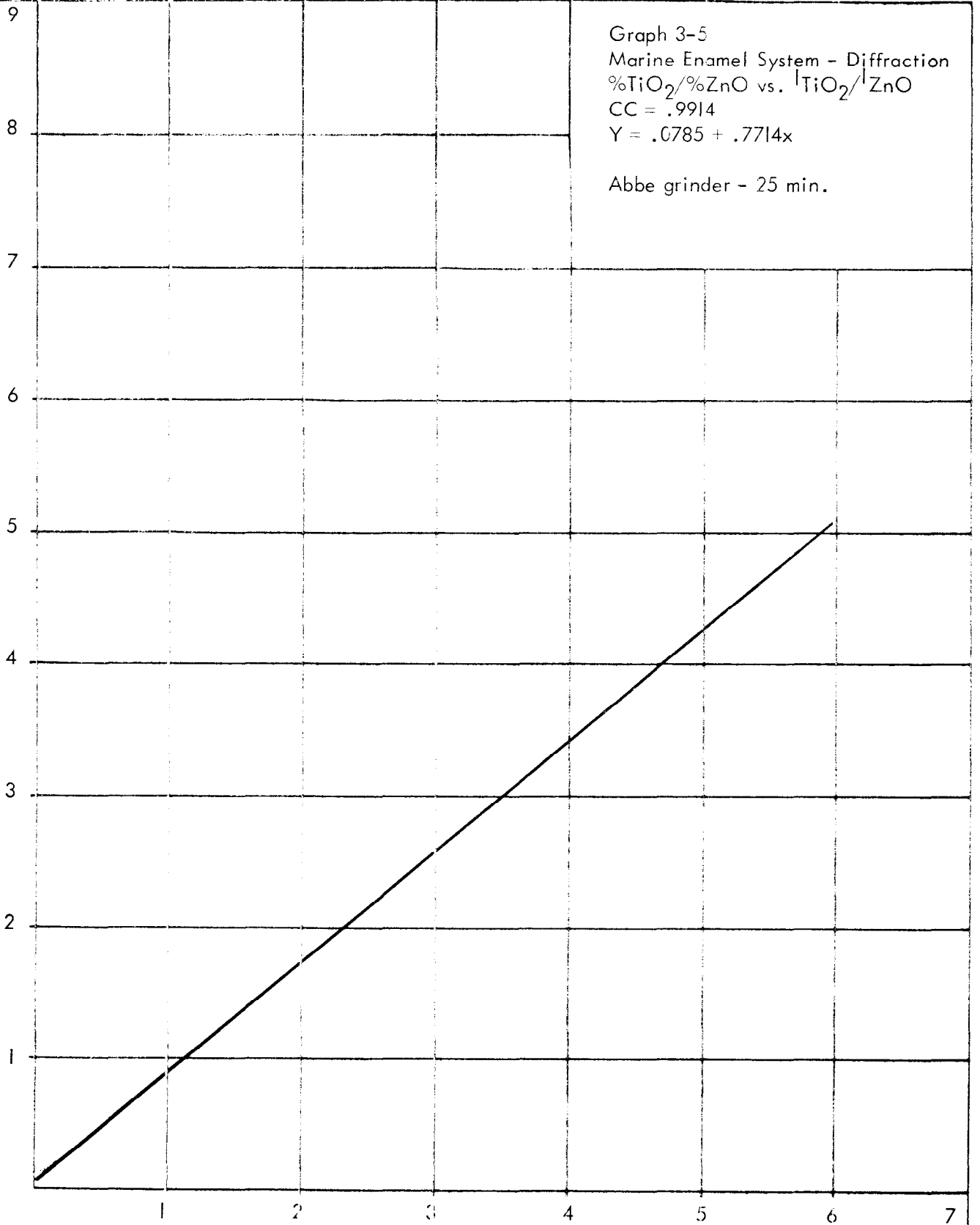
%Zn/%Ti



Graph 3-5  
Marine Enamel System - Diffraction  
 $\%TiO_2/\%ZnO$  vs.  $I_{TiO_2}/I_{ZnO}$   
CC = .9914  
 $Y = .0785 + .7714x$

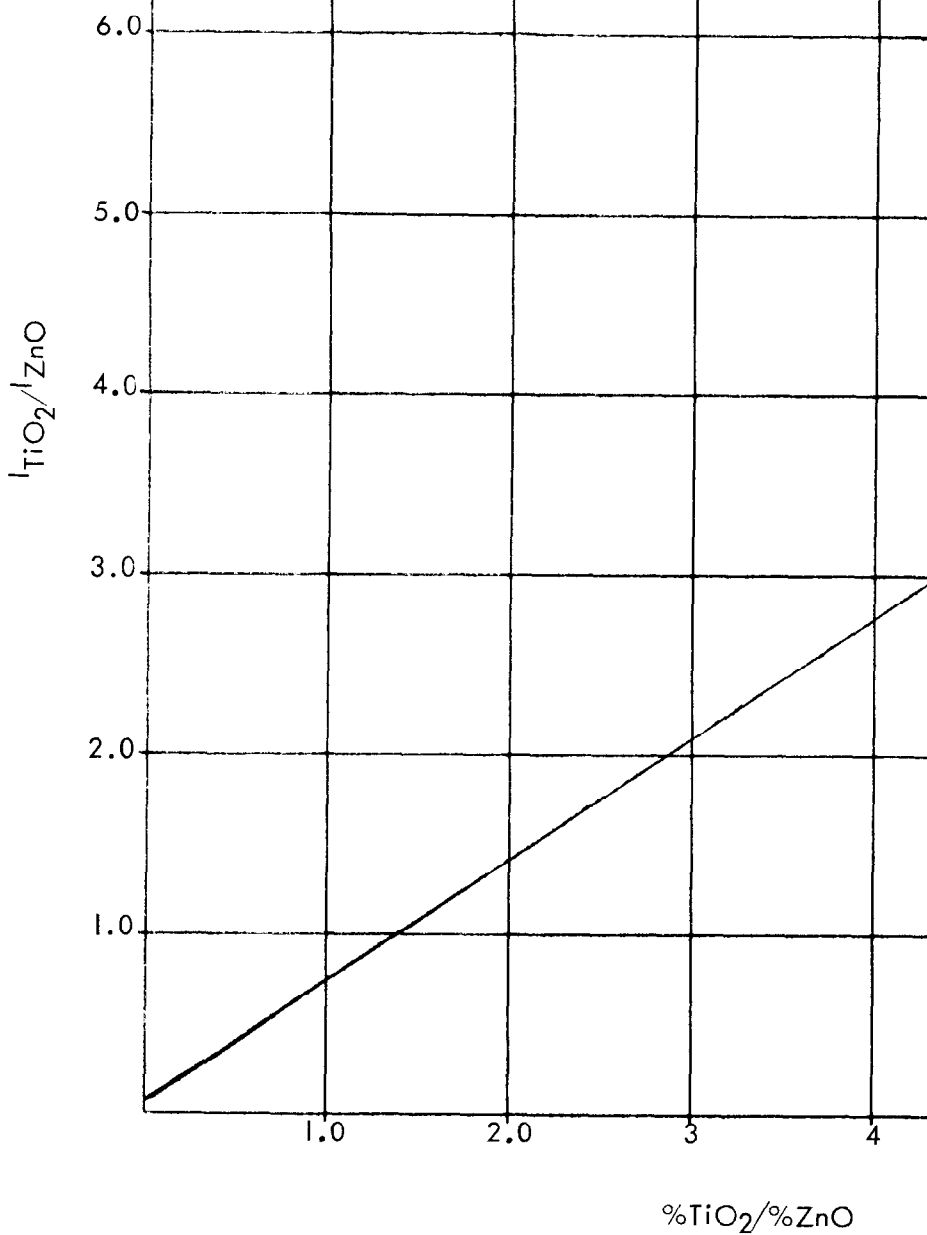
Abbe grinder - 25 min.

$I_{TiO_2}/I_{ZnO}$



$\%TiO_2/\%ZnO$

Graph 4-1  
Marine Enamel System - Diffraction  
%TiO<sub>2</sub>/<sub>%ZnO vs. I<sup>TiO<sub>2</sub></sup>/I<sup>ZnO</sup>  
CC= .9950  
Y = .0752 + .6935x</sub>



Graph 4-2  
Marine Enamel System - Diffraction  
 $\%ZnO/\%TiO_2$  vs.  $I_{ZnO}/I_{TiO_2}$   
CC = .9989  
 $Y = .1104 + 1.2131x$

Mortar and pestle

$I_{ZnO}/I_{TiO_2}$

8  
7  
6  
5  
4  
3  
2  
1

$\%ZnO/\%TiO_2$

1 2 3 4 5 6



Graph 4-3

Marine Enamel System - Fluorescence

%Ti/%Zn vs. I<sub>Ti</sub>/I<sub>Zn</sub>

CC = .9987

Y = .3108 + 1.1605x

Mortar and pestle

I<sub>Ti</sub>/I<sub>Zn</sub>

2

1

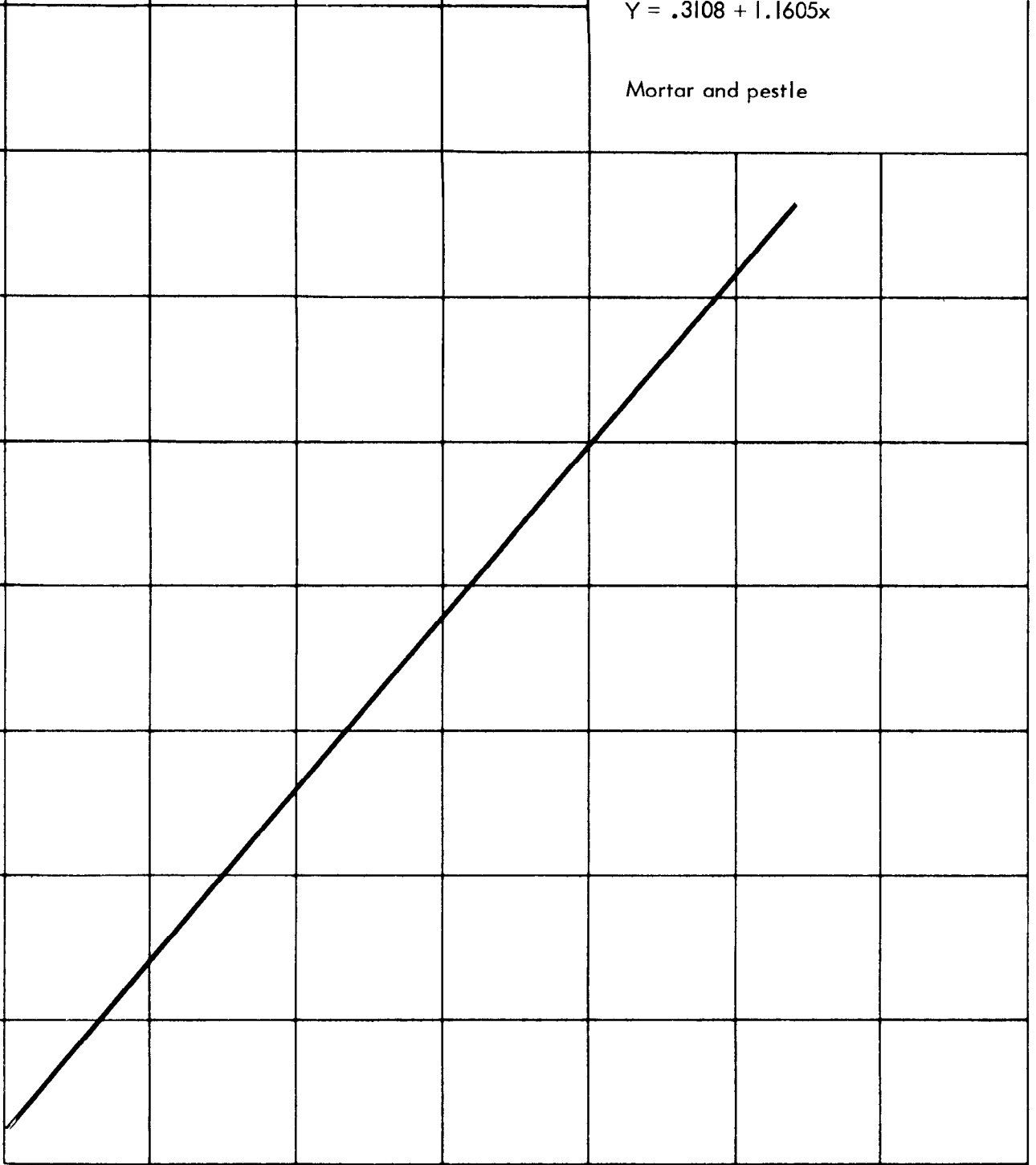
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2

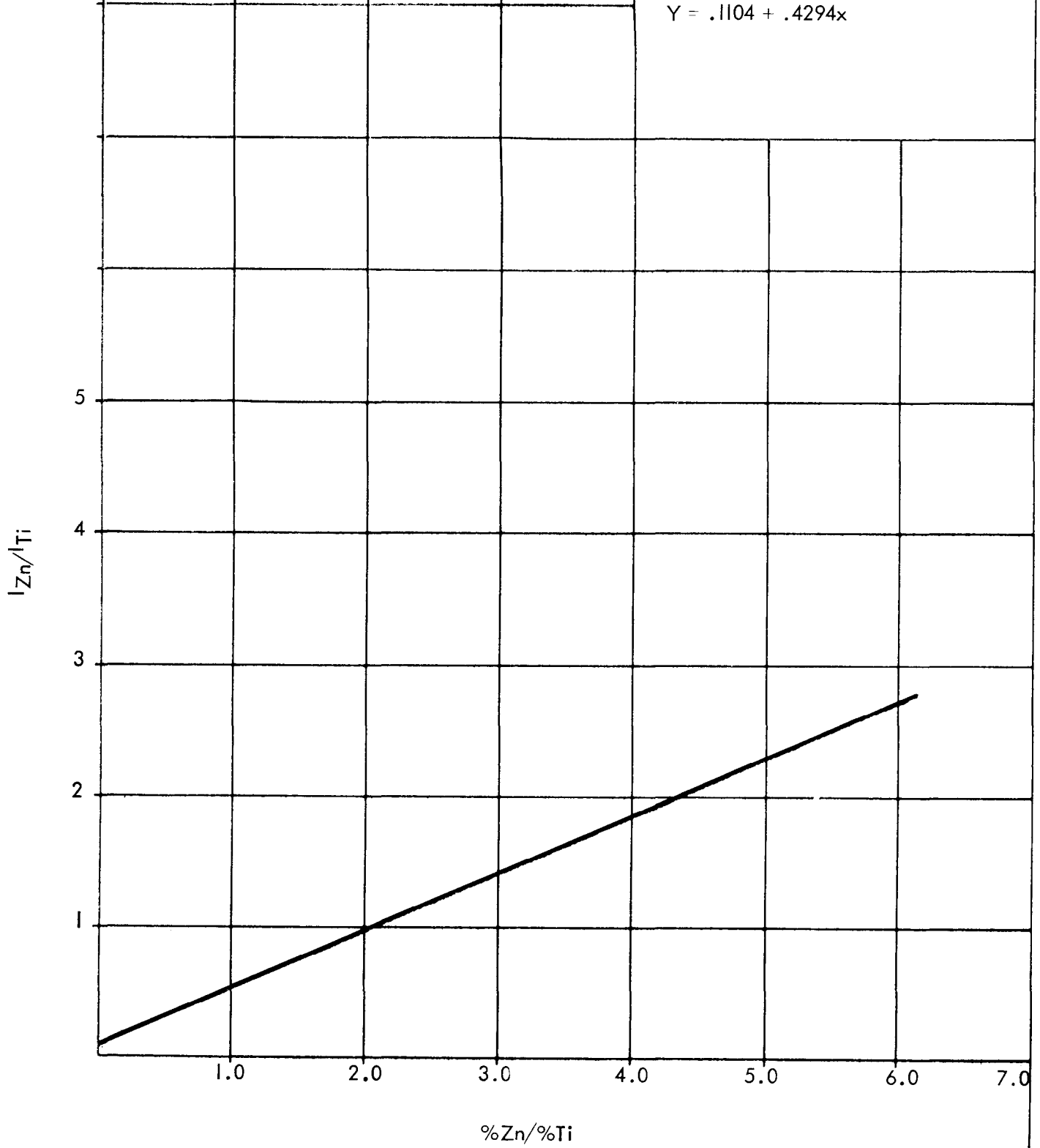
3

4

%Ti/%Zn



Graph 4-4  
Marine Enamel System - Fluorescence  
%Zn/%Ti vs.  $I_{Zn}/I_{Ti}$   
CC = .9978  
 $Y = .1104 + .4294x$



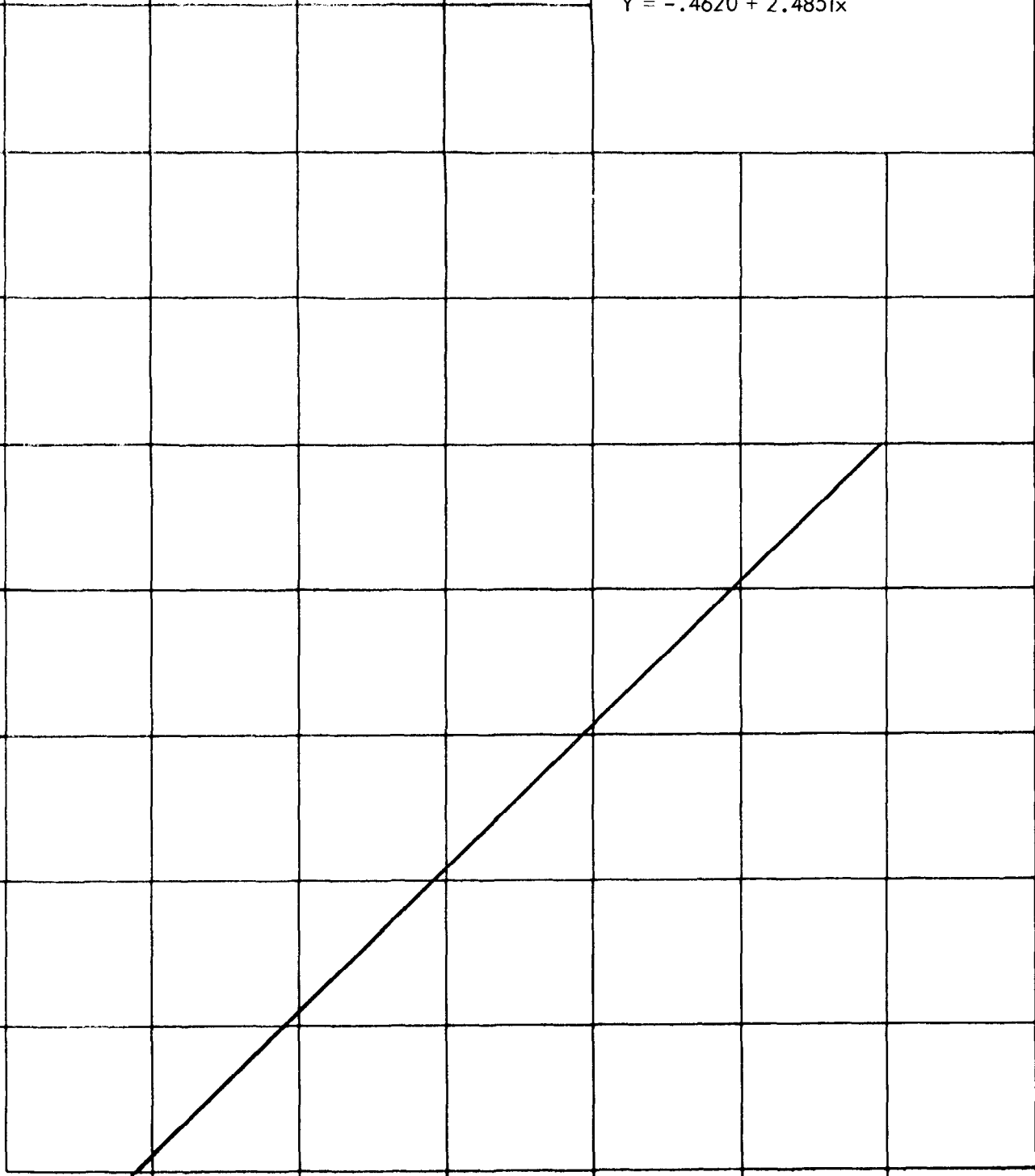
Graph 5-1  
Marine Enamel System - Diffraction  
%Talc/%ZnO vs.  $I_{\text{Talc}}/I_{\text{ZnO}}$   
CC = .8853  
 $Y = -.4620 + 2.4851x$

$I_{\text{Talc}}/I_{\text{ZnO}}$

2.0  
1.5  
1.0  
.5

.2 .4 .6 .8 1.0

%Talc/%ZnO



Graph 5-2  
Marine Enamel - Diffraction  
%ZnO/%Talc vs.  $I_{ZnO}/I_{Talc}$   
CC = .2626

$I_{ZnO}/I_{Talc}$

2.0

1.0

1

2

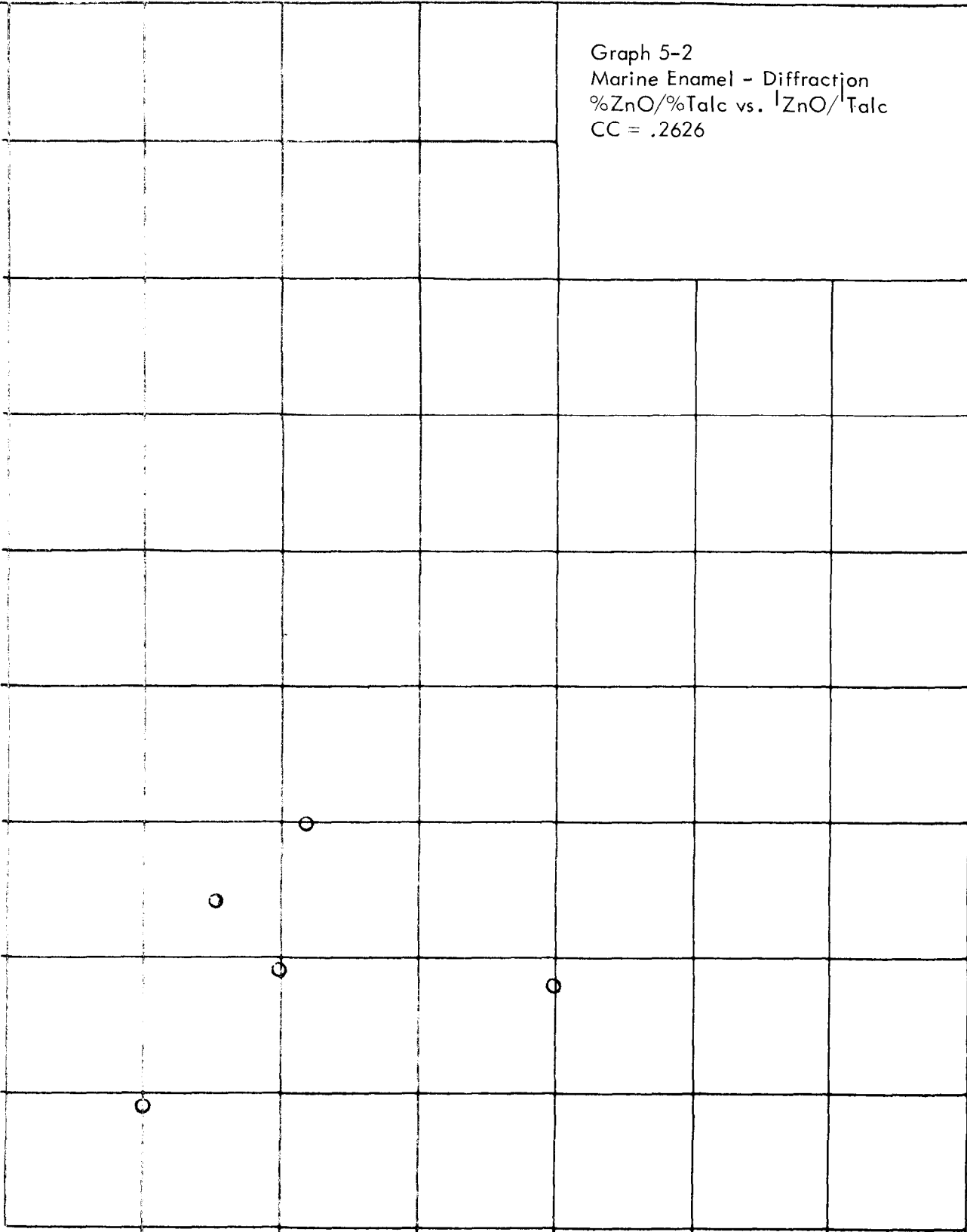
3

4

5

6

%ZnO/%Talc



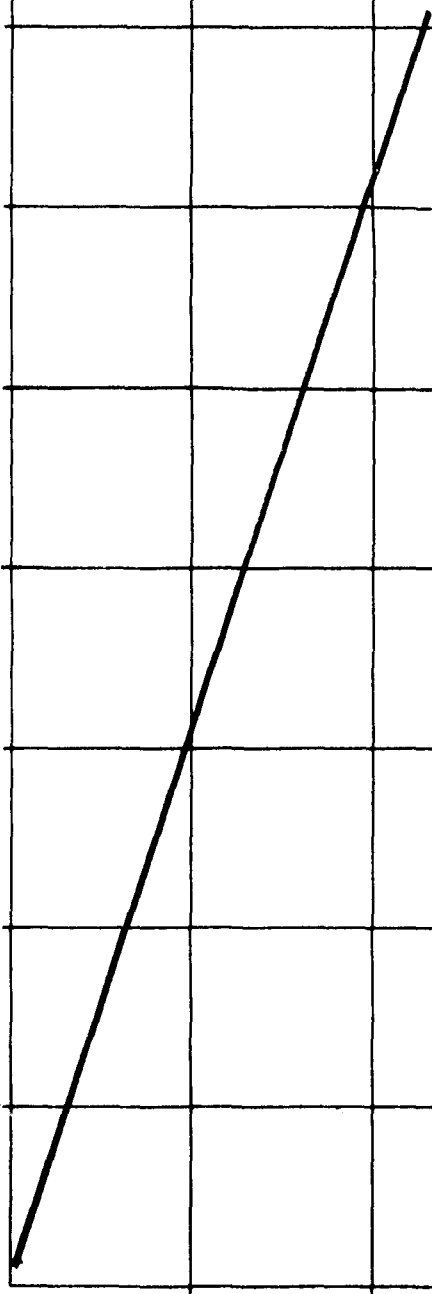
Graph 5-3  
Marine Enamel System - Fluorescence  
%Mg/%Zn vs.  $I_{Mg}/I_{Zn}$   
CC = .9829  
 $Y = .0831 + 3.1504x$

$I_{Mg}/I_{Zn}$

3  
2  
1.5  
1  
.5

.5 1.0 1.5 2.0

%Mg/%Zn



Graph 5-4  
Marine Enamel System - Fluorescence  
%Zn/%Si vs.  $I_{Zn}/I_{Si}$   
CC = .9940  
 $Y = -.0761 + .2923x$

$I_{Zn}/I_{Si}$

3

2

1

2

4

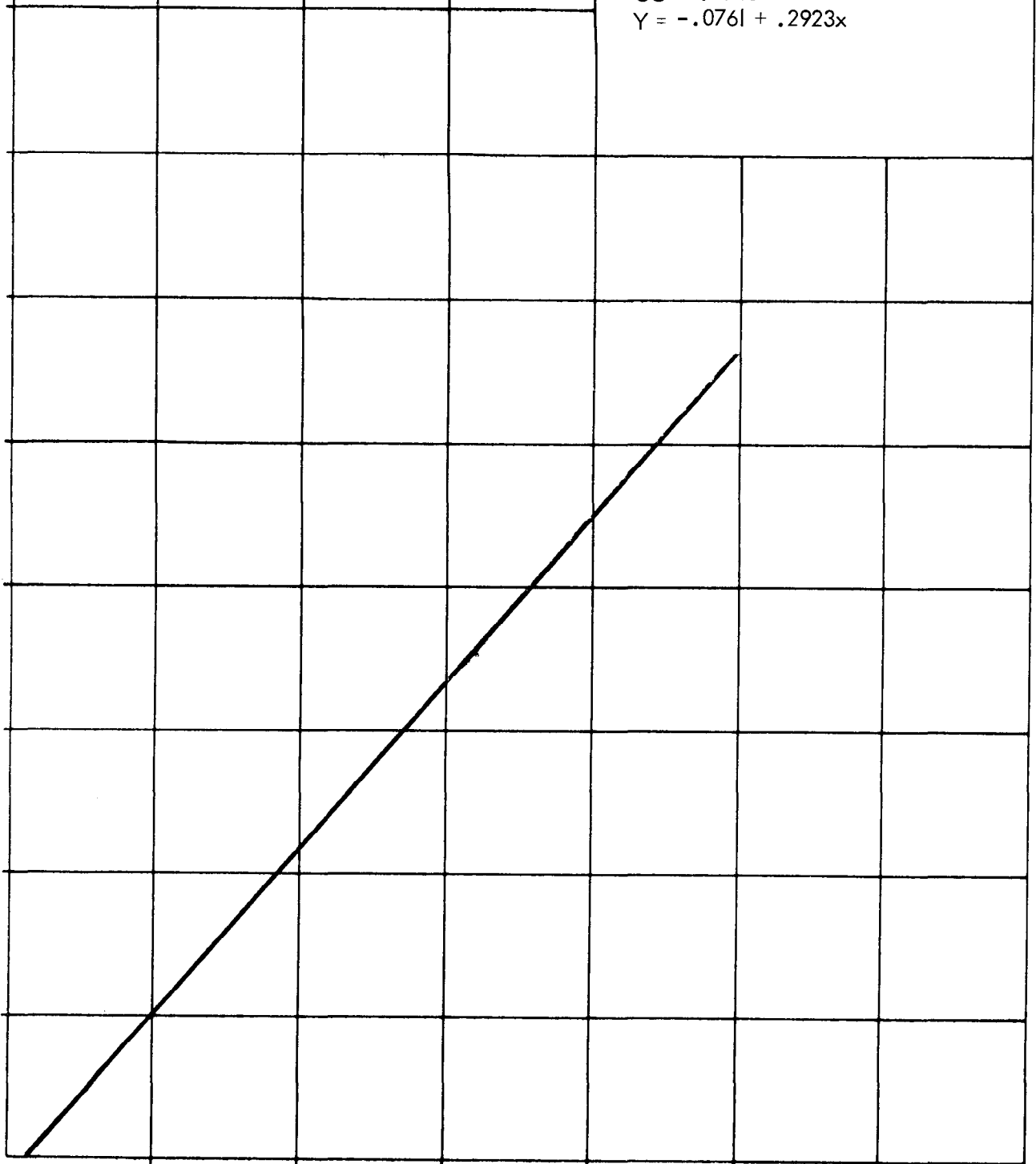
6

8

10

12

%Zn/%Si



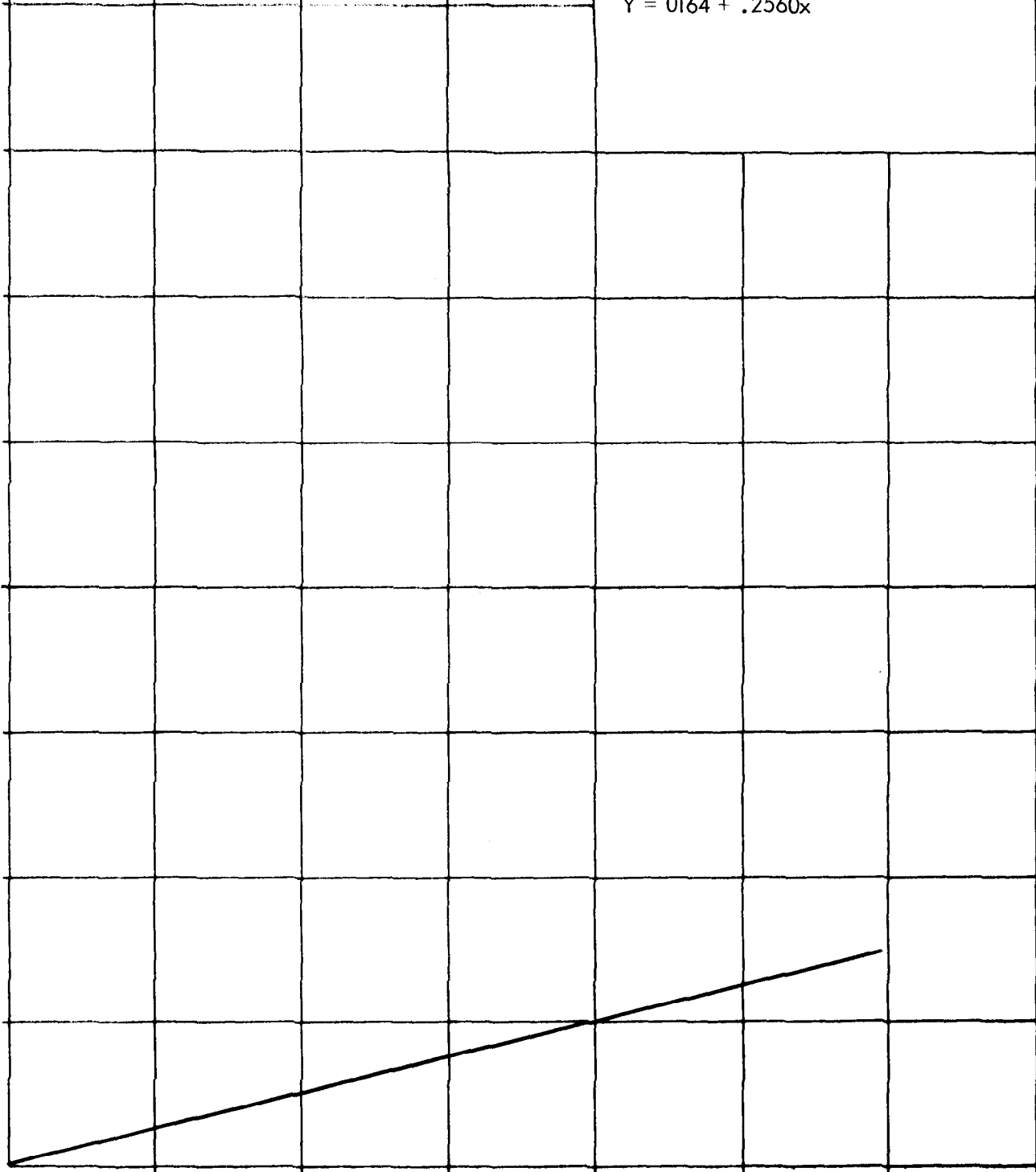
Graph 6-1  
Marine Enamel System - Diffraction  
%TiO<sub>2</sub>/%Talc vs. I<sub>TiO<sub>2</sub></sub>/I<sub>Talc</sub>  
CC = .9831  
Y = 0.164 + .2560x

I<sub>TiO<sub>2</sub></sub>/I<sub>Talc</sub>

3  
2  
1

1.0 2.0 3.0 4.0

%TiO<sub>2</sub>/%Talc



Graph 6-2  
Marine Enamel System - Diffraction  
%Talc/%TiO<sub>2</sub> vs. I<sub>Talc</sub>/I<sub>TiO<sub>2</sub></sub>  
CC = .9469  
Y = .3007 + 3.0870x

I<sub>Talc</sub>/I<sub>TiO<sub>2</sub></sub>

2

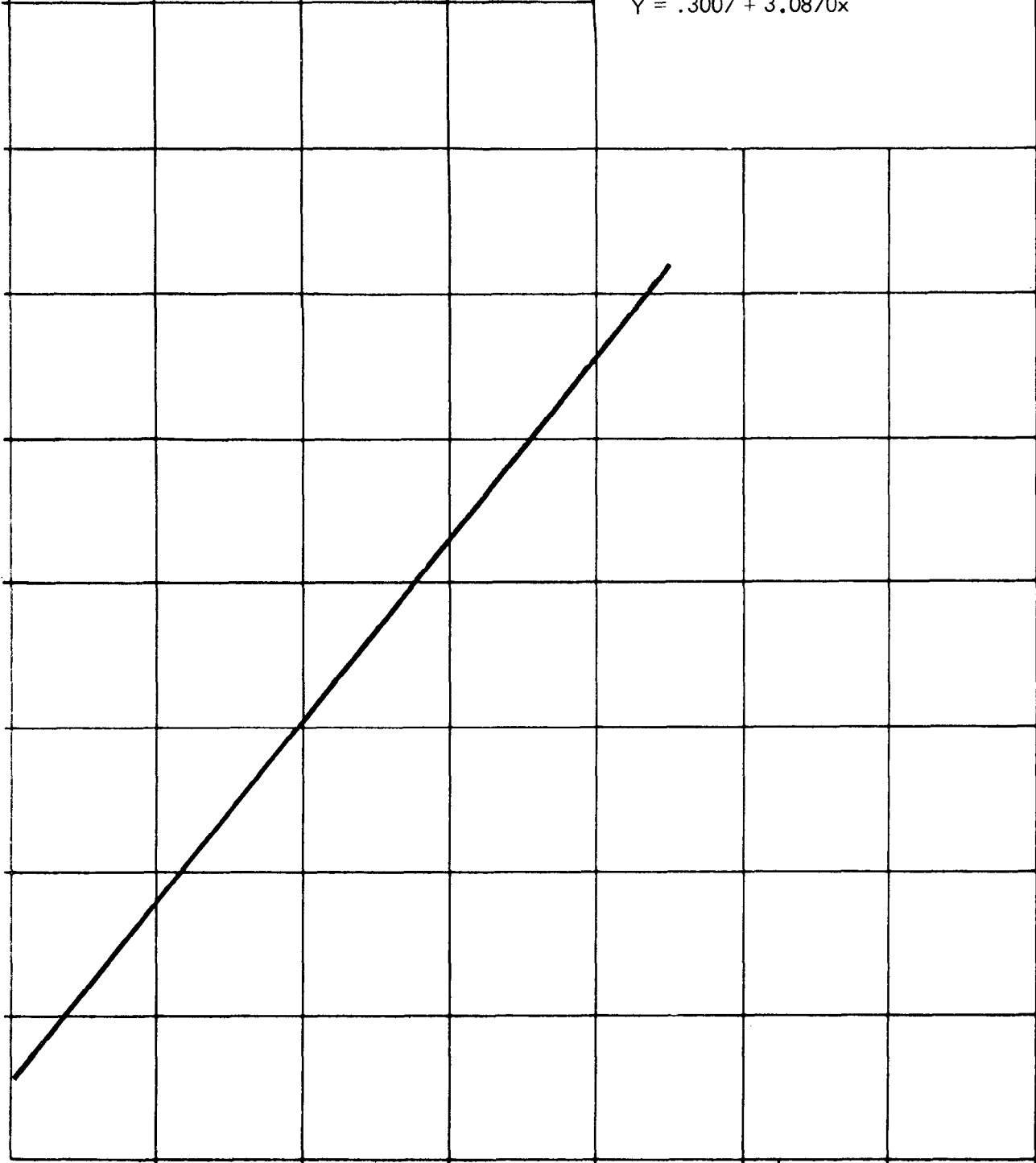
1

.2

.4

.6

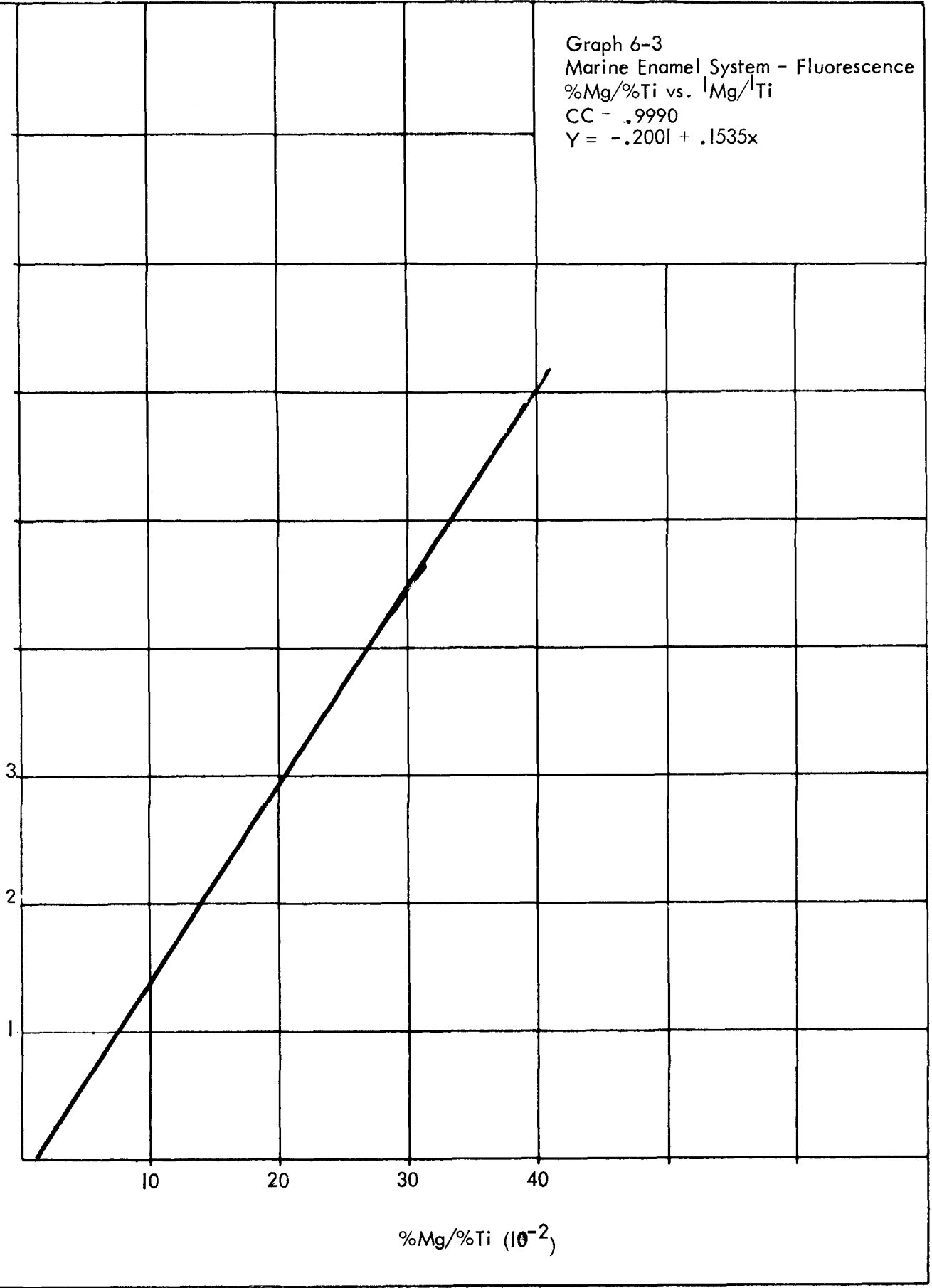
%Talc/%TiO<sub>2</sub>



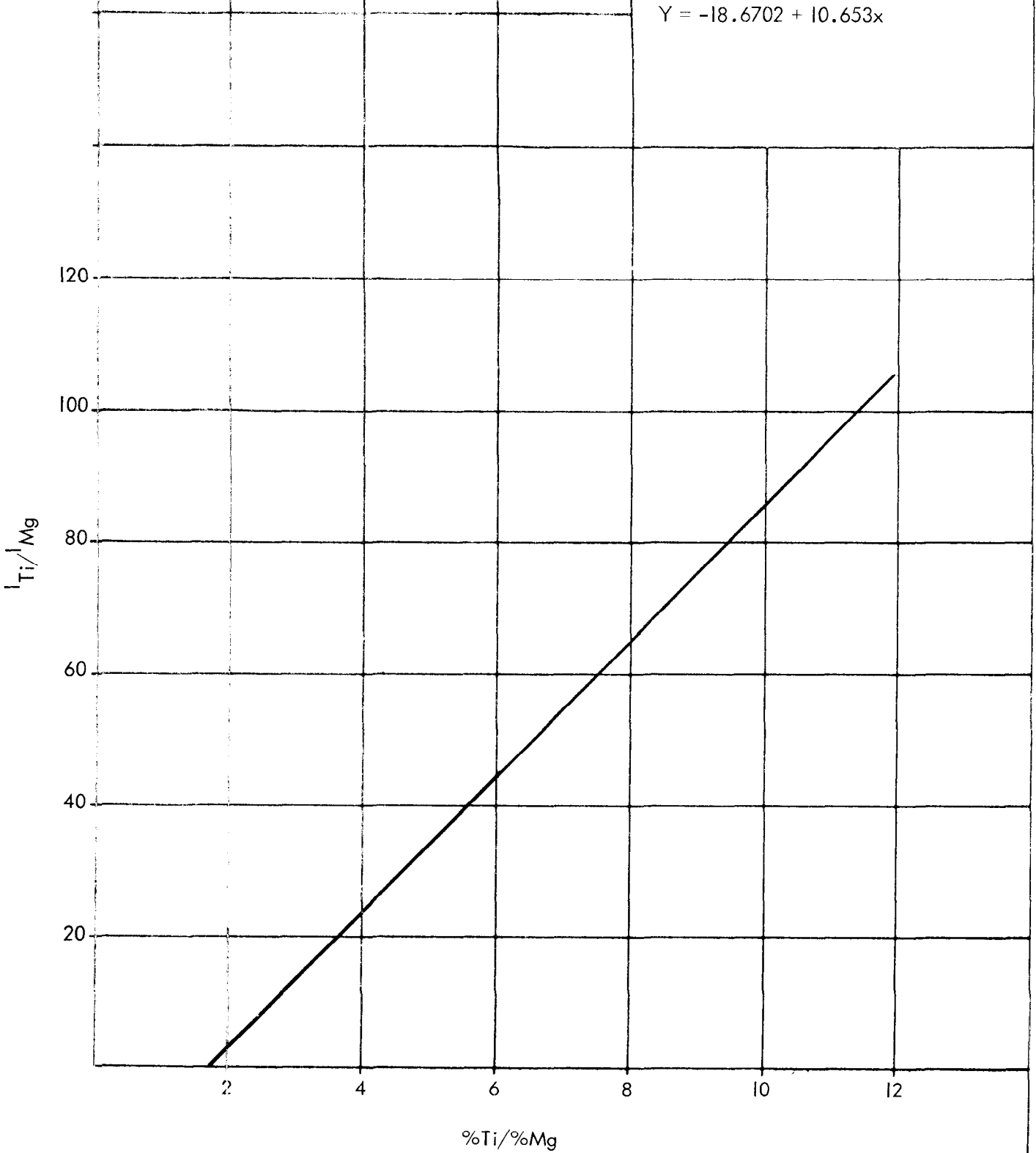


Graph 6-3  
Marine Enamel System - Fluorescence  
%Mg/%Ti vs.  $I_{Mg}/I_{Ti}$   
CC = .9990  
 $Y = -.2001 + .1535x$

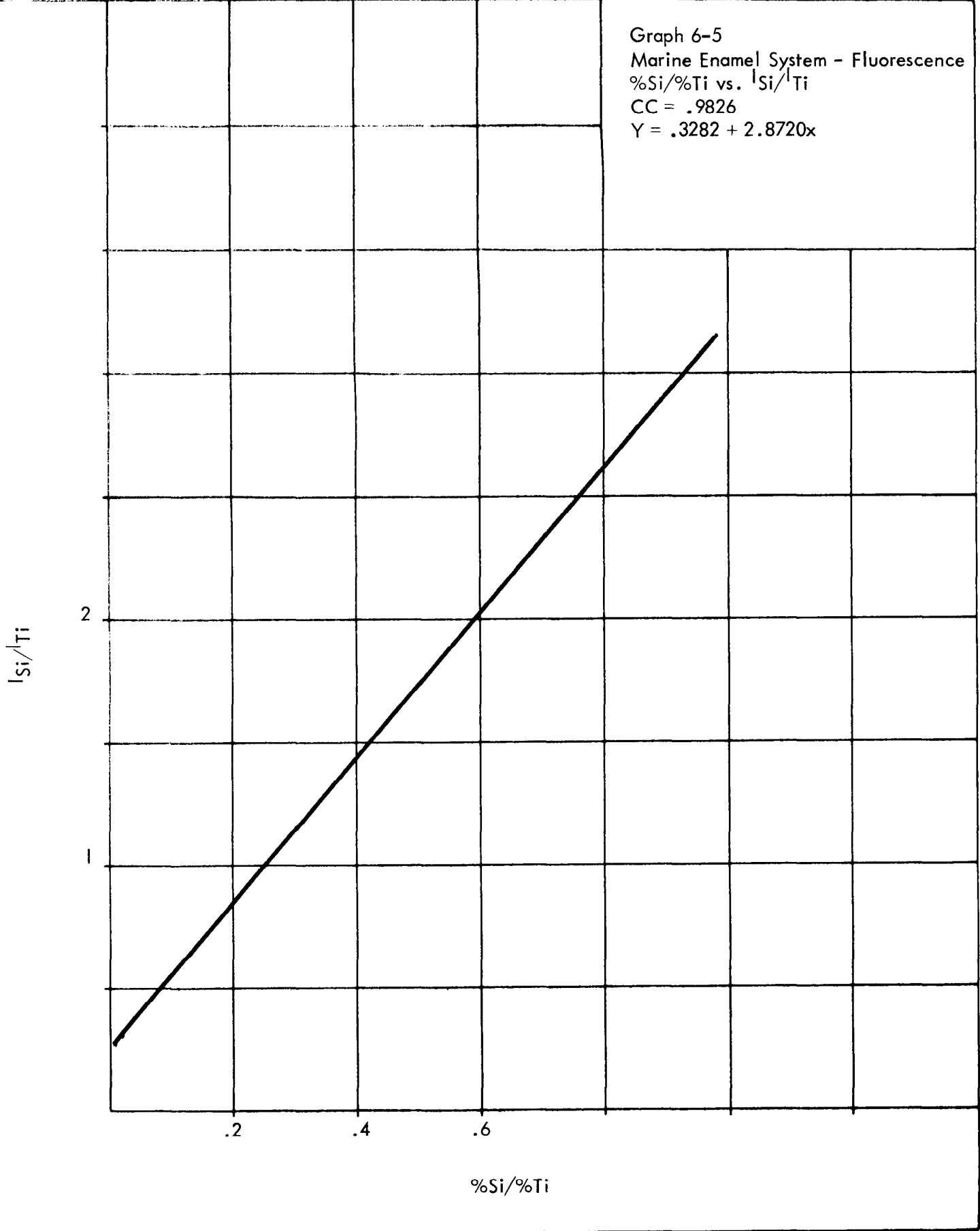
$I_{Mg}/I_{Ti} (10^{-2})$



Graph 6-4  
Marine Enamel System - Fluorescence  
%Ti/%Mg vs.  $I_{Ti}/I_{Mg}$   
CC = .9995  
 $Y = -18.6702 + 10.653x$



Graph 6-5  
Marine Enamel System - Fluorescence  
%Si/%Ti vs. I<sub>Si</sub>/I<sub>Ti</sub>  
CC = .9826  
Y = .3282 + 2.8720x



Graph 6-6  
Marine Enamel System - Fluorescence  
%Ti/%Si vs.  $^{47}\text{Ti}/^{47}\text{Si}$   
CC = .9995  
 $Y = -0.1595 + 0.2352x$

$^{47}\text{Ti}/^{47}\text{Si}$

2

1

1

2

3

4

5

6

7

% Ti/%Si

Graph 7-1  
Marine Enamel System - Diffraction  
%A/%B + %C vs.  $I_A/I_B + I_C$   
CC = .9394  
 $Y = .0507 + .4851x$

$I_A/I_B + I_C$

.4

.3

.2

.1

.1

.2

.3

.4

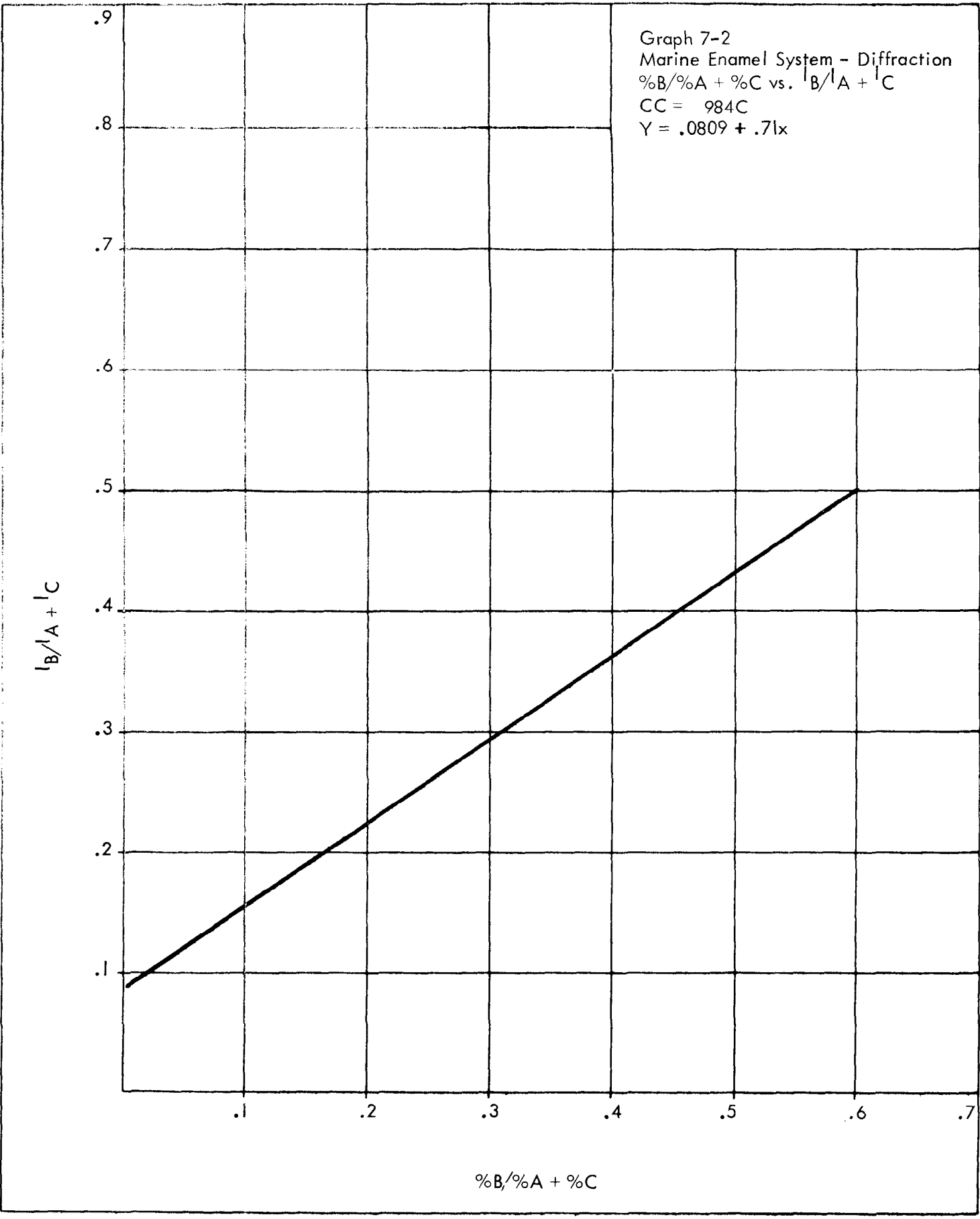
.5

.6

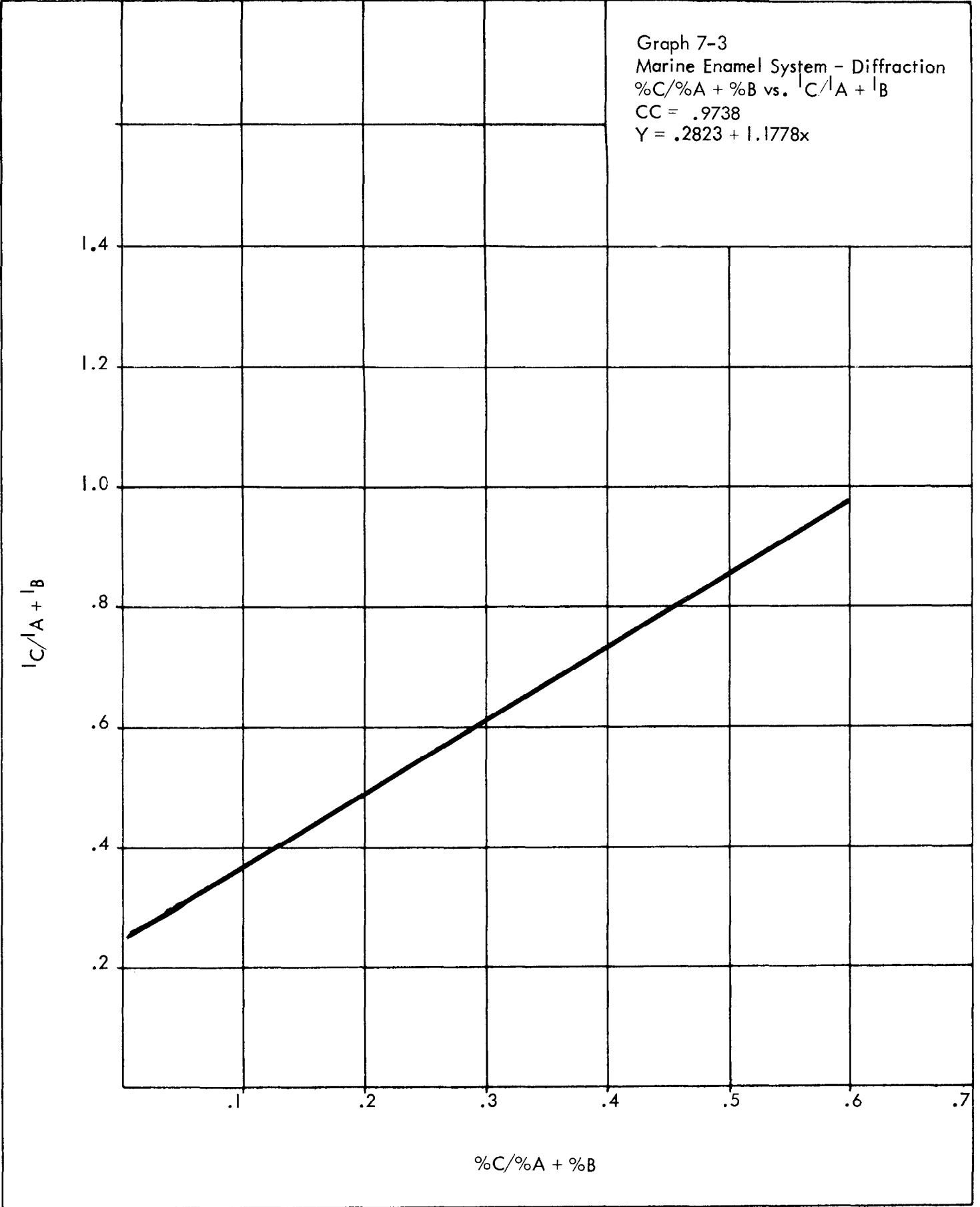
.7

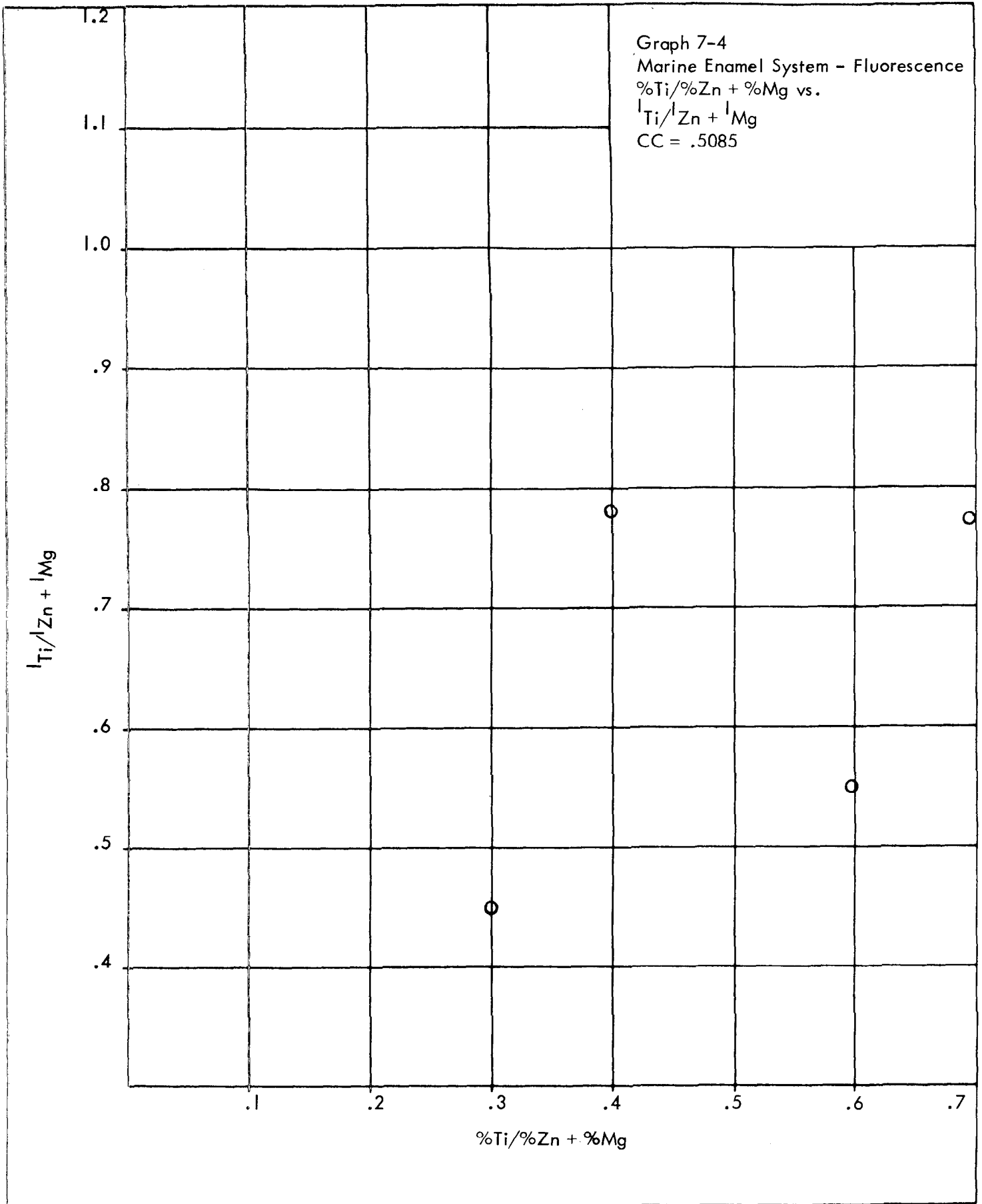
%A/%B + %C

Graph 7-2  
Marine Enamel System - Diffraction  
%B/%A + %C vs.  $I_B/I_A + I_C$   
CC = 984C  
 $Y = .0809 + .71x$



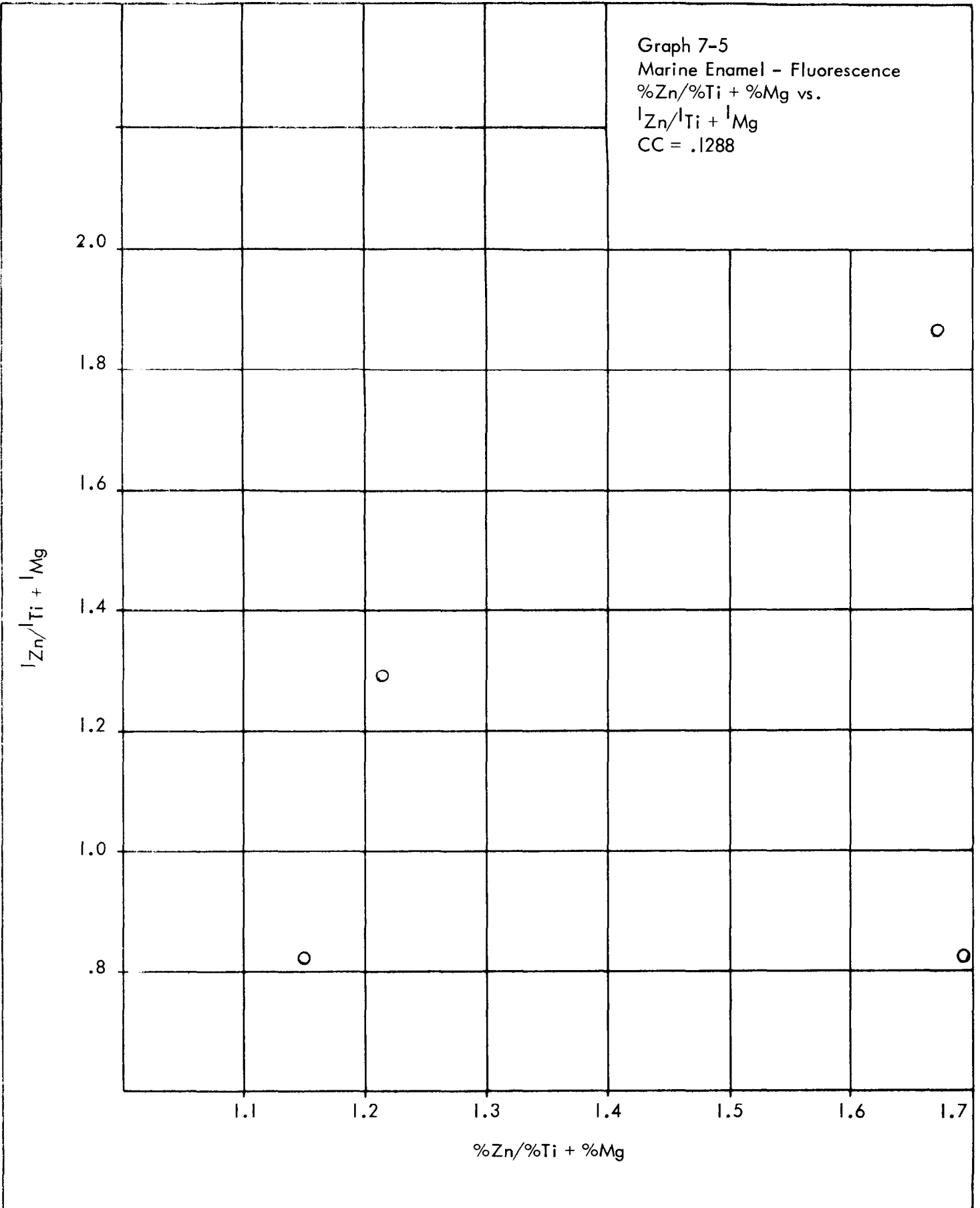
Graph 7-3  
Marine Enamel System - Diffraction  
%C/%A + %B vs.  $I_C/I_A + I_B$   
CC = .9738  
Y = .2823 + 1.1778x





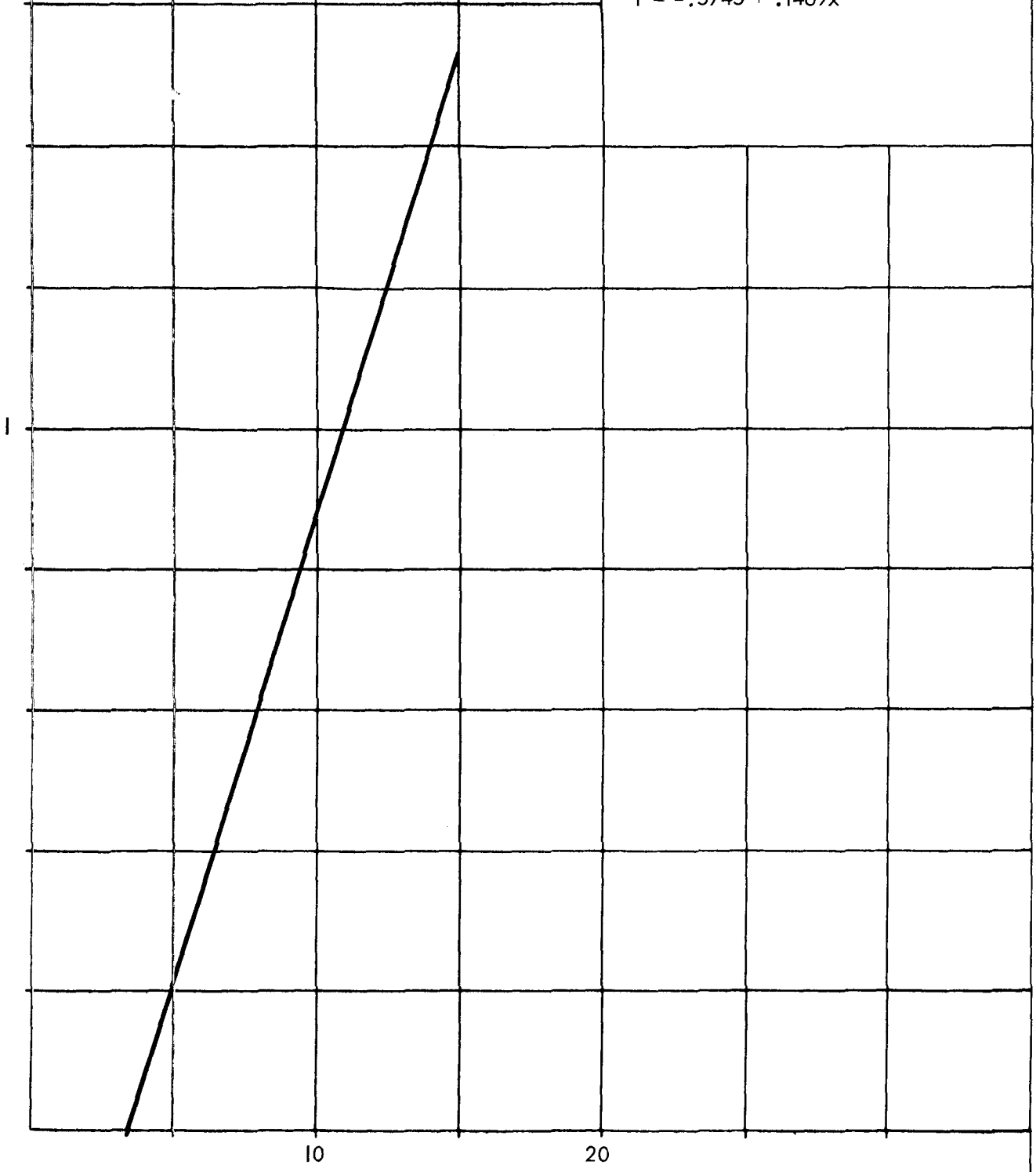


Graph 7-5  
Marine Enamel - Fluorescence  
 $\%Zn/\%Ti + \%Mg$  vs.  
 $I_{Zn}/I_{Ti} + I_{Mg}$   
CC = .1288



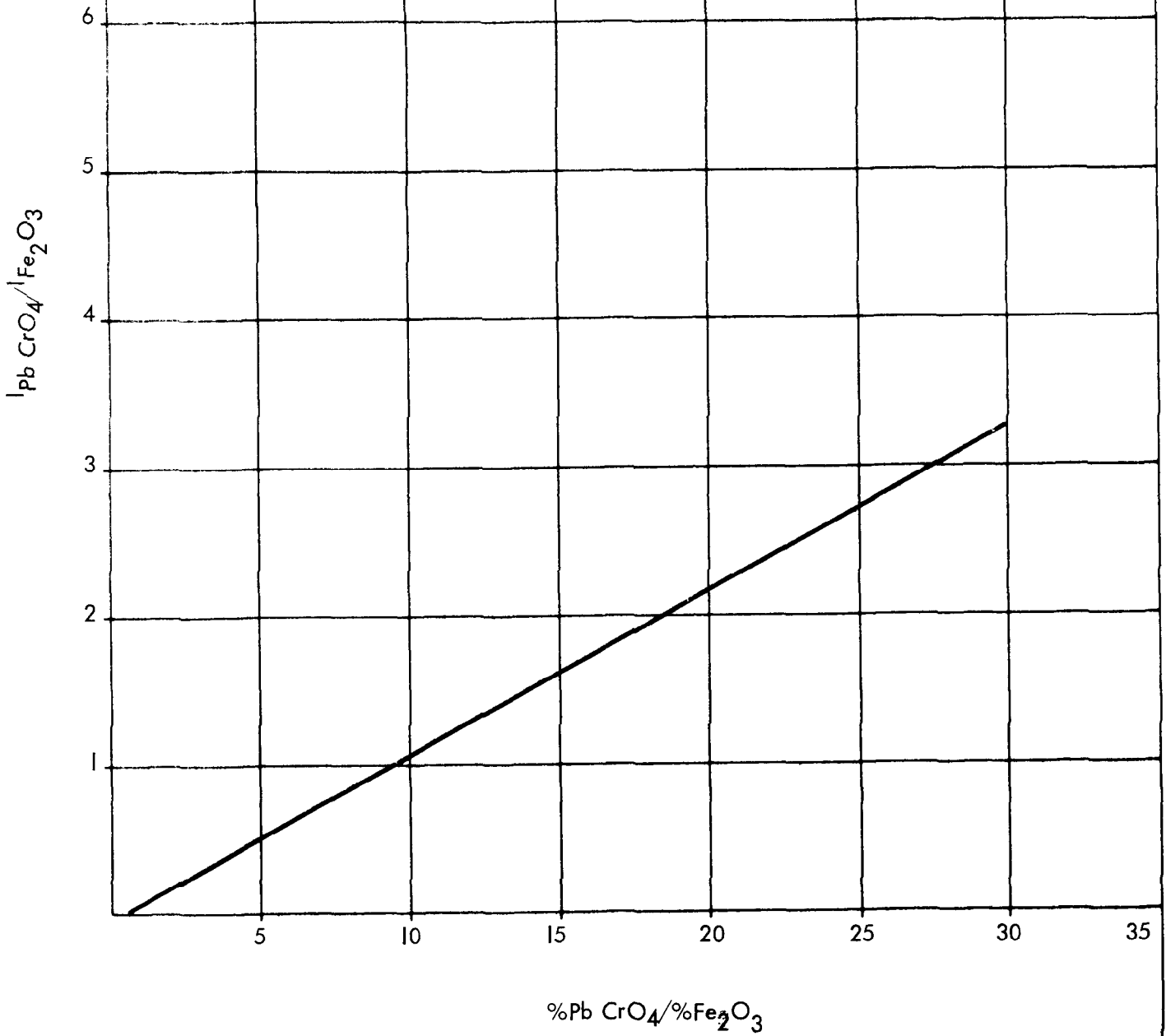
Graph 7-6  
Marine Enamel System - Fluorescence  
%Mg/%Ti + %Zn vs.  $I_{Mg}/I_{Ti} + I_{Zn}$   
CC = .9967  
 $Y = -.5745 + .1469x$

$I_{Mg}/I_{Ti} + I_{Zn} (10^{-2})$



%Mg/%Ti + %Zn ( $10^{-2}$ )

Graph 8-1  
Basic Lead Silico Chromate - Paint  
Diffraction  
%Pb CrO<sub>4</sub>/ %Fe<sub>2</sub>O<sub>3</sub> vs.  
I<sub>Pb CrO<sub>4</sub></sub> / I<sub>Fe<sub>2</sub>O<sub>3</sub></sub>  
CC = .9807  
Y = -.0127 + .1110x



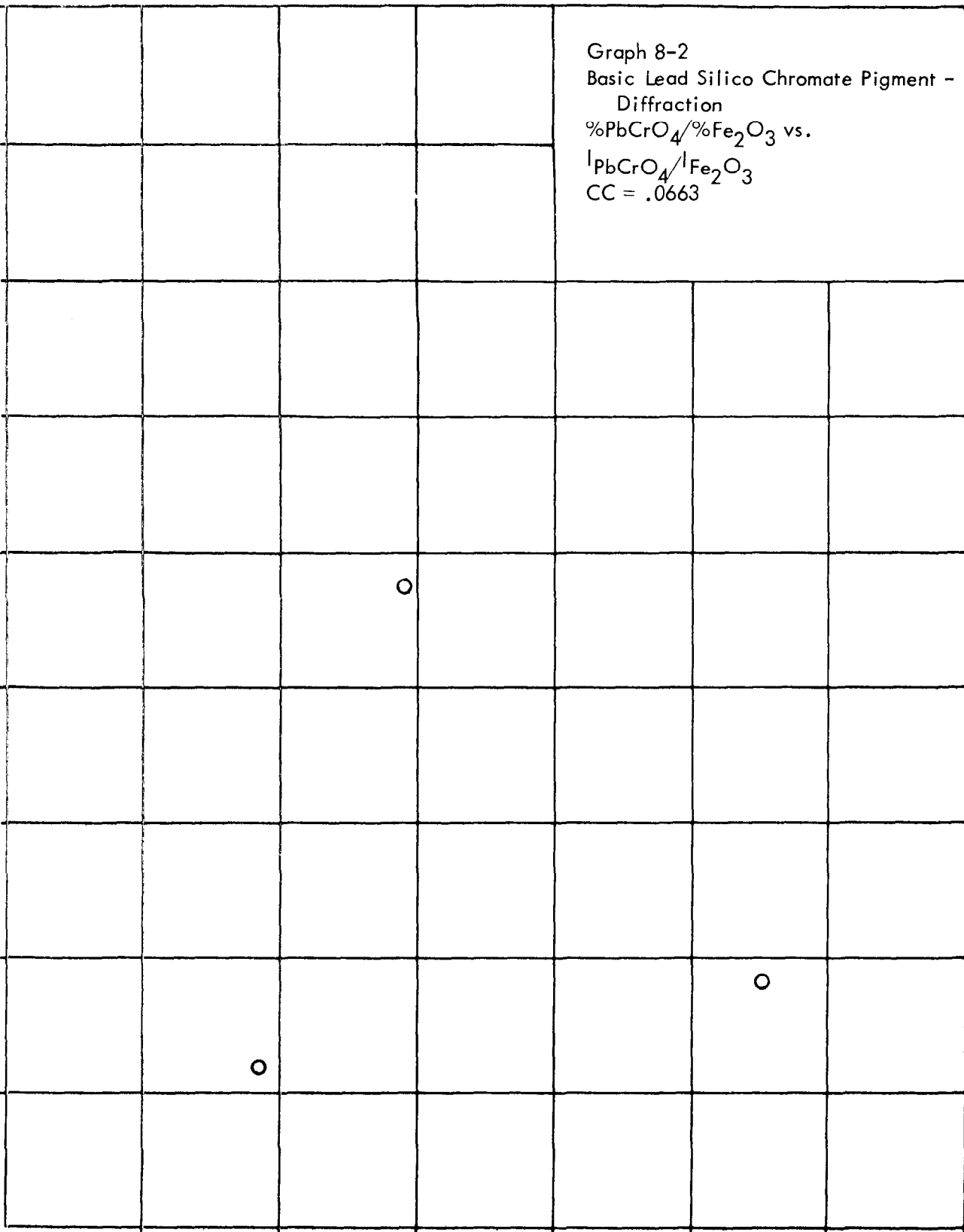
Graph 8-2  
Basic Lead Silico Chromate Pigment -  
Diffraction  
 $\%PbCrO_4/\%Fe_2O_3$  vs.  
 $I_{PbCrO_4}/I_{Fe_2O_3}$   
CC = .0663

$I_{PbCrO_4}/I_{Fe_2O_3}$

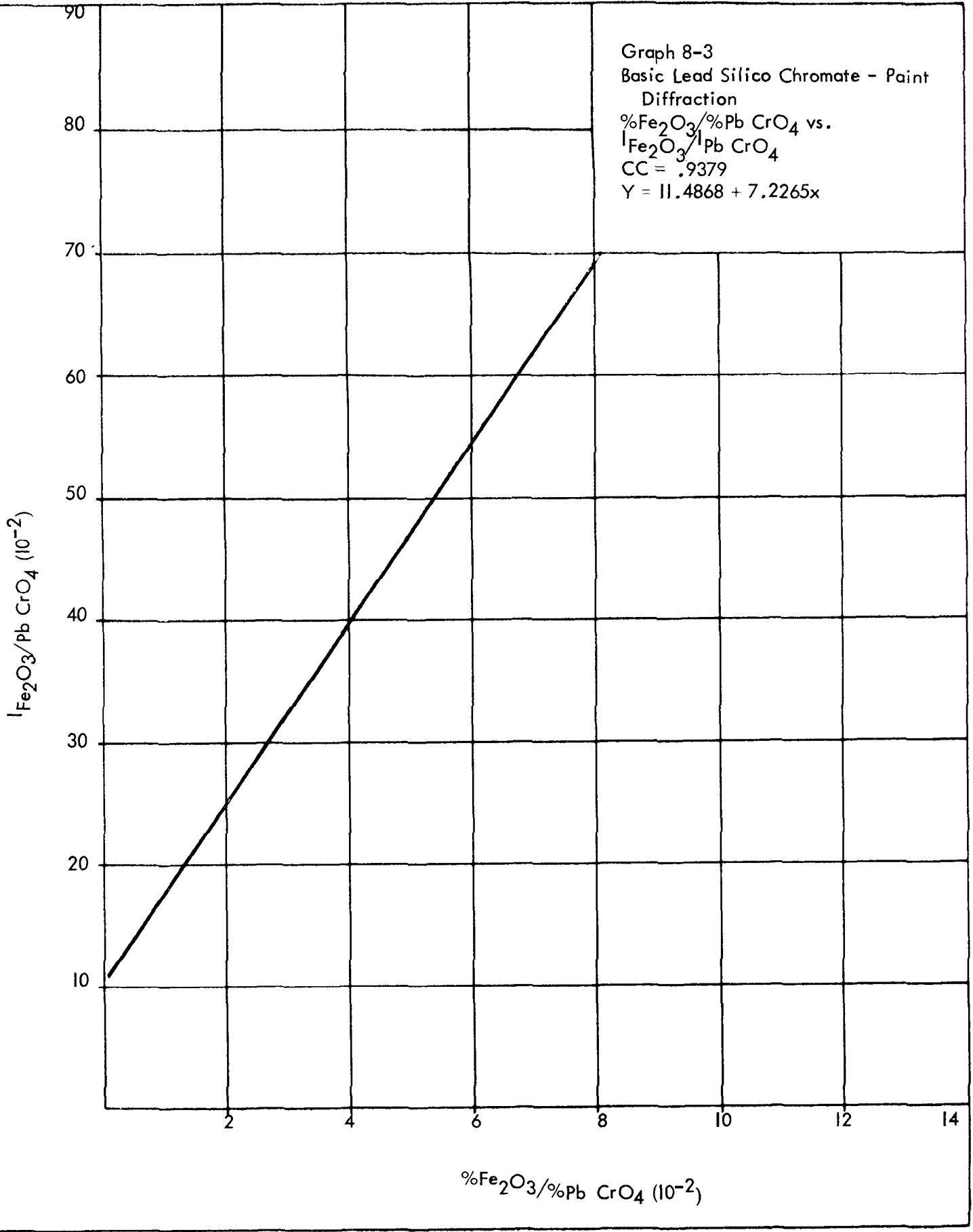
50  
40  
30  
20  
10

5 10 15 20 25 30

$\%PbCrO_4/\%Fe_2O_3$



Graph 8-3  
Basic Lead Silico Chromate - Paint  
Diffraction  
 $\%Fe_2O_3/\%Pb\ CrO_4$  vs.  
 $I_{Fe_2O_3}/I_{Pb\ CrO_4}$   
CC = .9379  
 $Y = 11.4868 + 7.2265x$



Graph 8-4  
Basic Lead Silico Chromate Pigment -  
Diffraction  
 $\%Fe_2O_3/\%PbCrO_4$  vs.  
 $I_{Fe_2O_3}/I_{PbCrO_4}$   
CC = .4900

$I_{Fe_2O_3}/I_{PbCrO_4} (10^{-2})$

08

07

06

05

04

03

02

01

02

04

06

08

.1

.12

.14

$\%Fe_2O_3/\%PbCrO_4 (10^{-2})$

○

○

○

Graph 8-5  
Basic Lead Silico Chromate - Paint  
%Fe/%Pb vs.  $I_{Fe}/I_{Pb}$   
CC = .9662  
 $Y = -3.7399 + 2.8917x$

$I_{Fe}/I_{Pb} (10^{-2})$

40

30

20

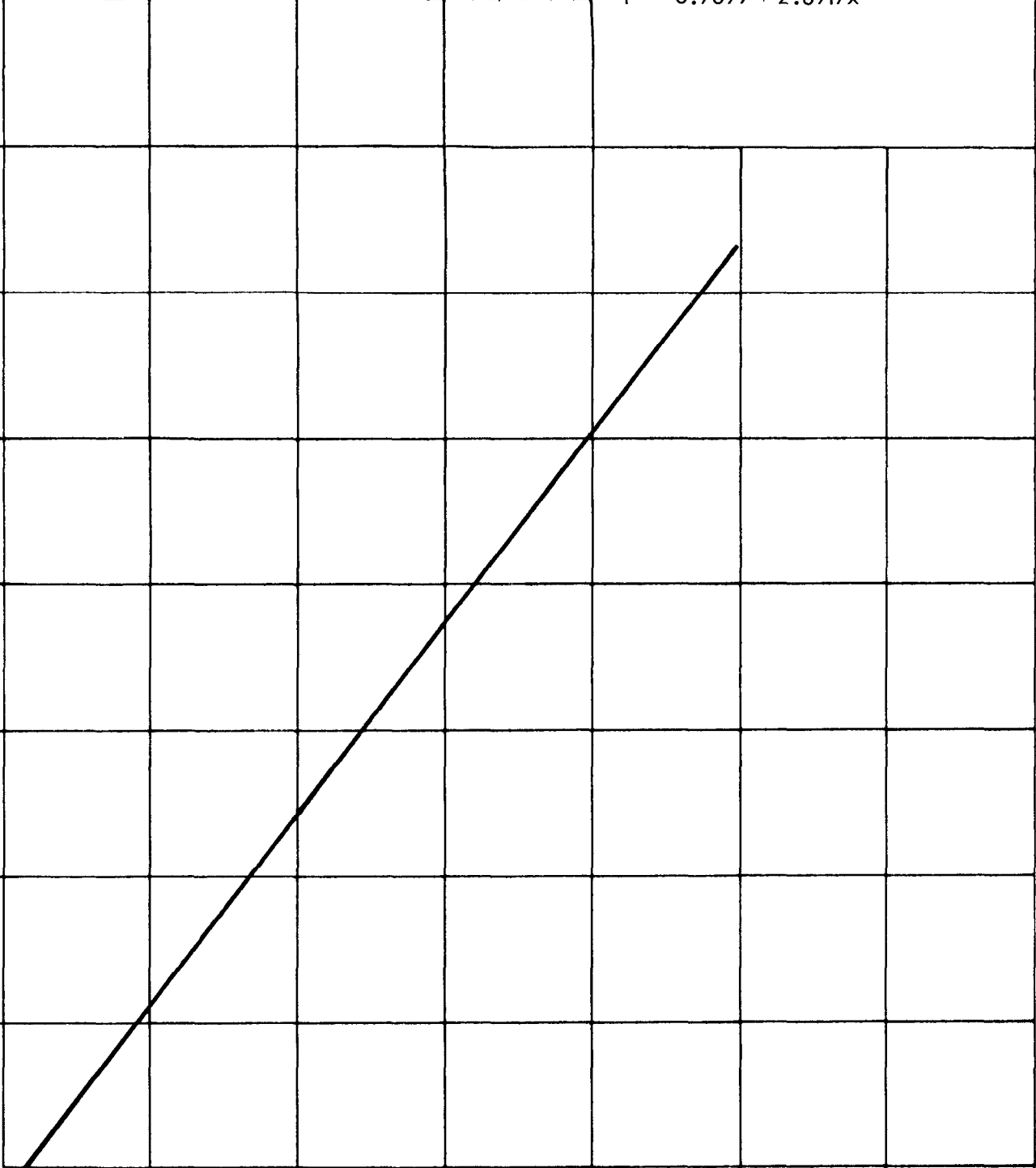
10

5

10

15

%Fe/%Pb ( $10^{-2}$ )



Graph 8-6  
Basic Lead Silico Chromate - Pigment  
%Fe/%Pb vs.  $I_{Fe}/I_{Pb}$   
CC = .9930  
 $Y = -4.305 + 3.5771x$

$I_{Fe}/I_{Pb} (10^{-2})$

40

30

20

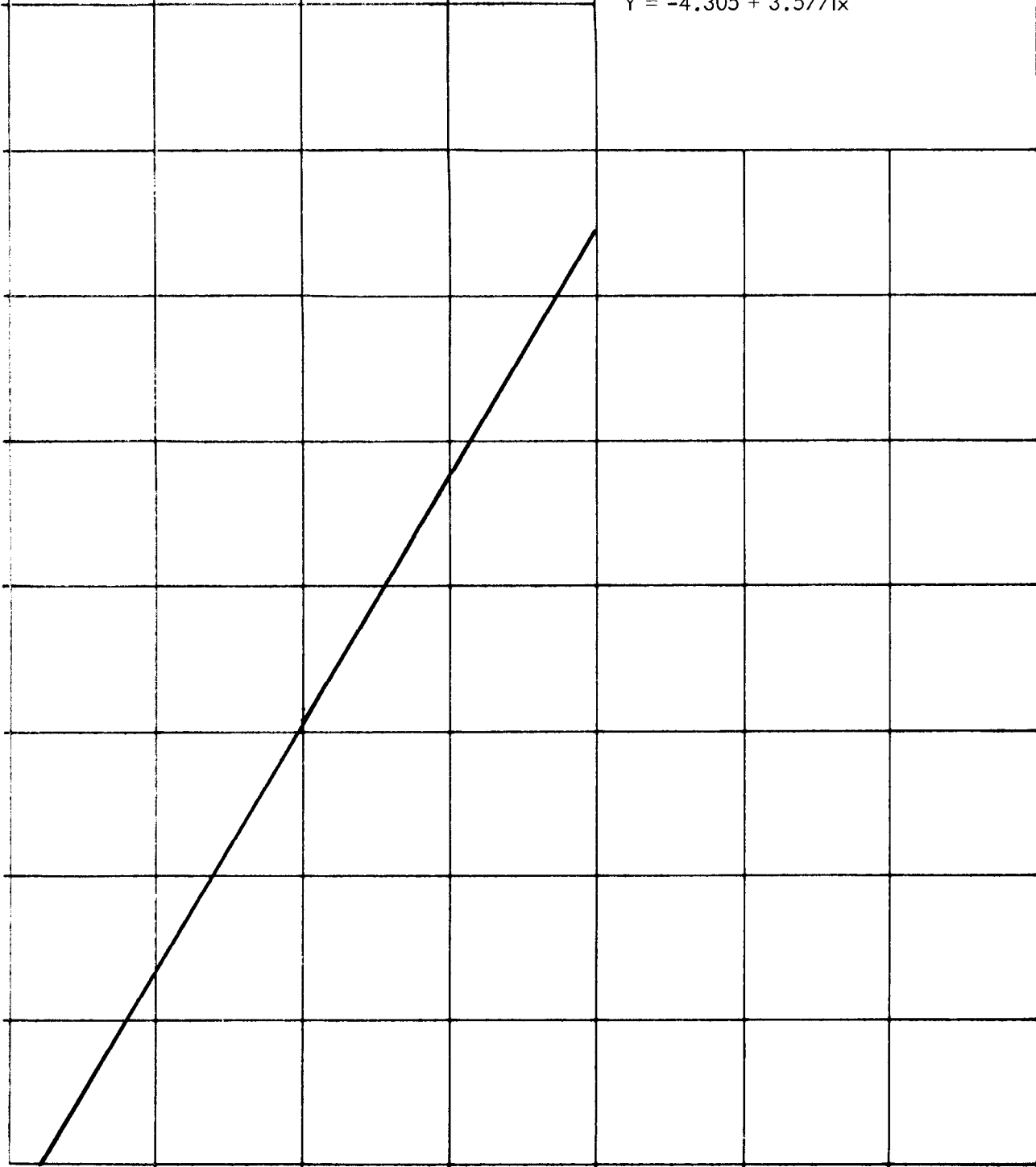
10

5

10

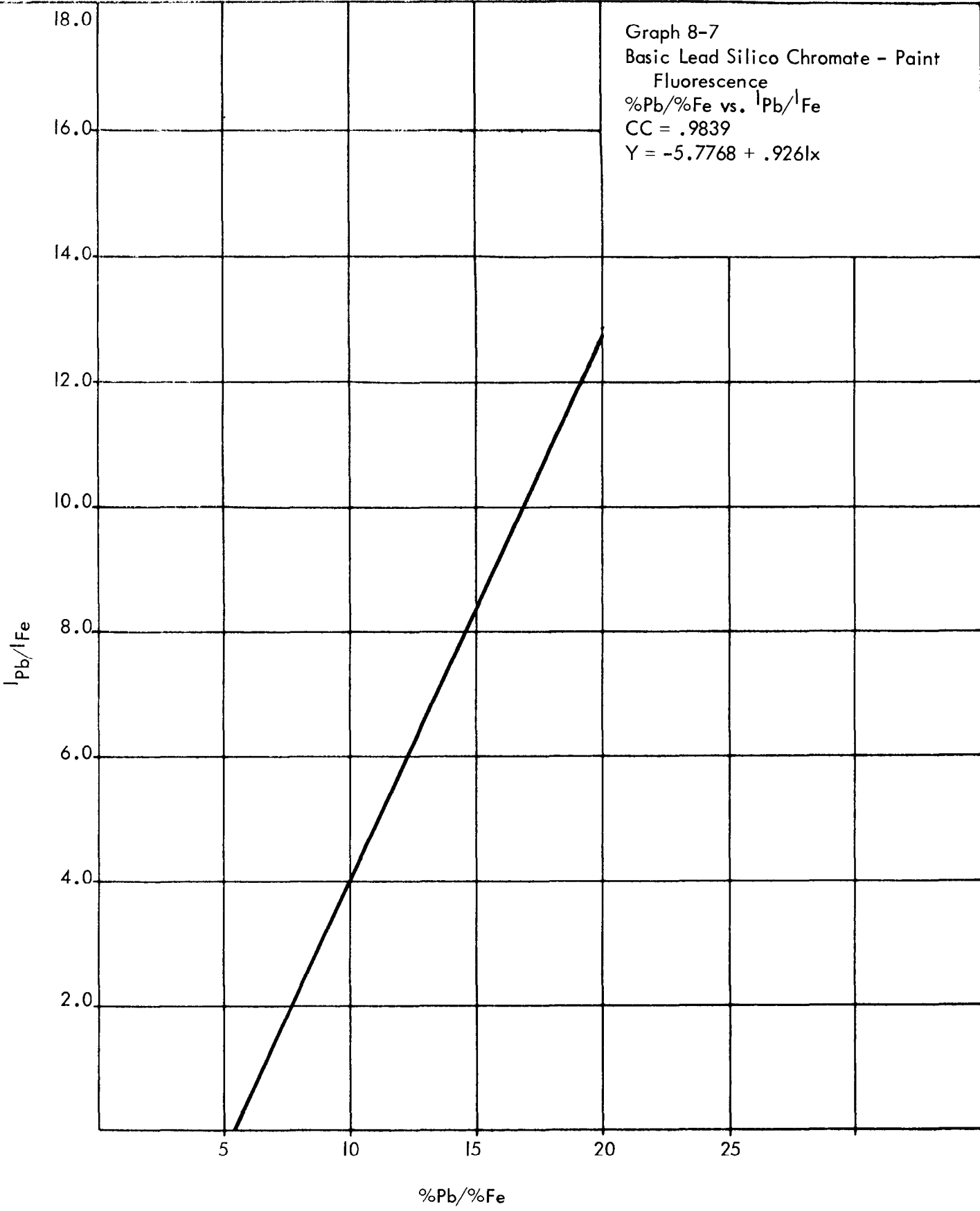
15

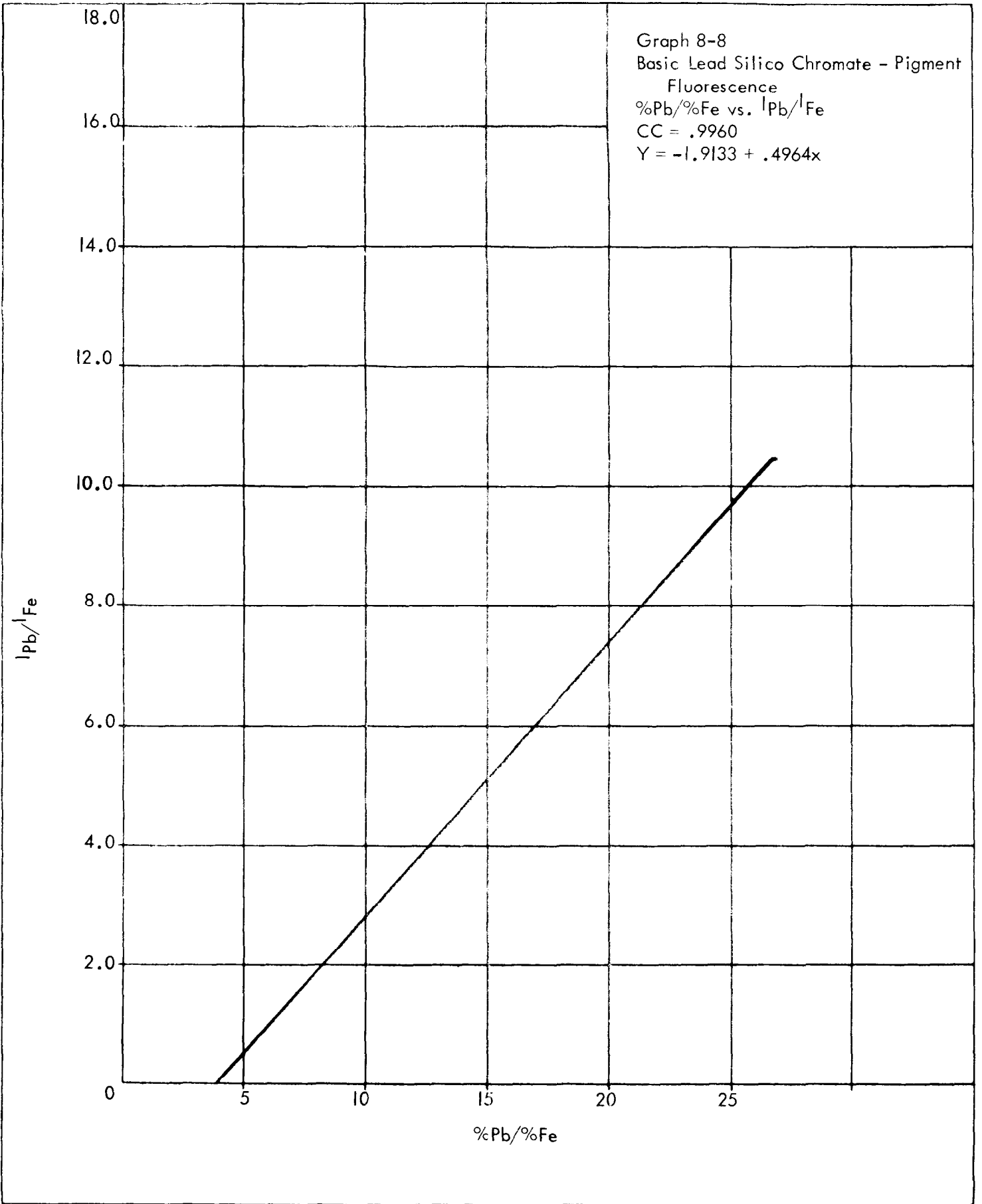
%Fe/%Pb ( $10^{-2}$ )



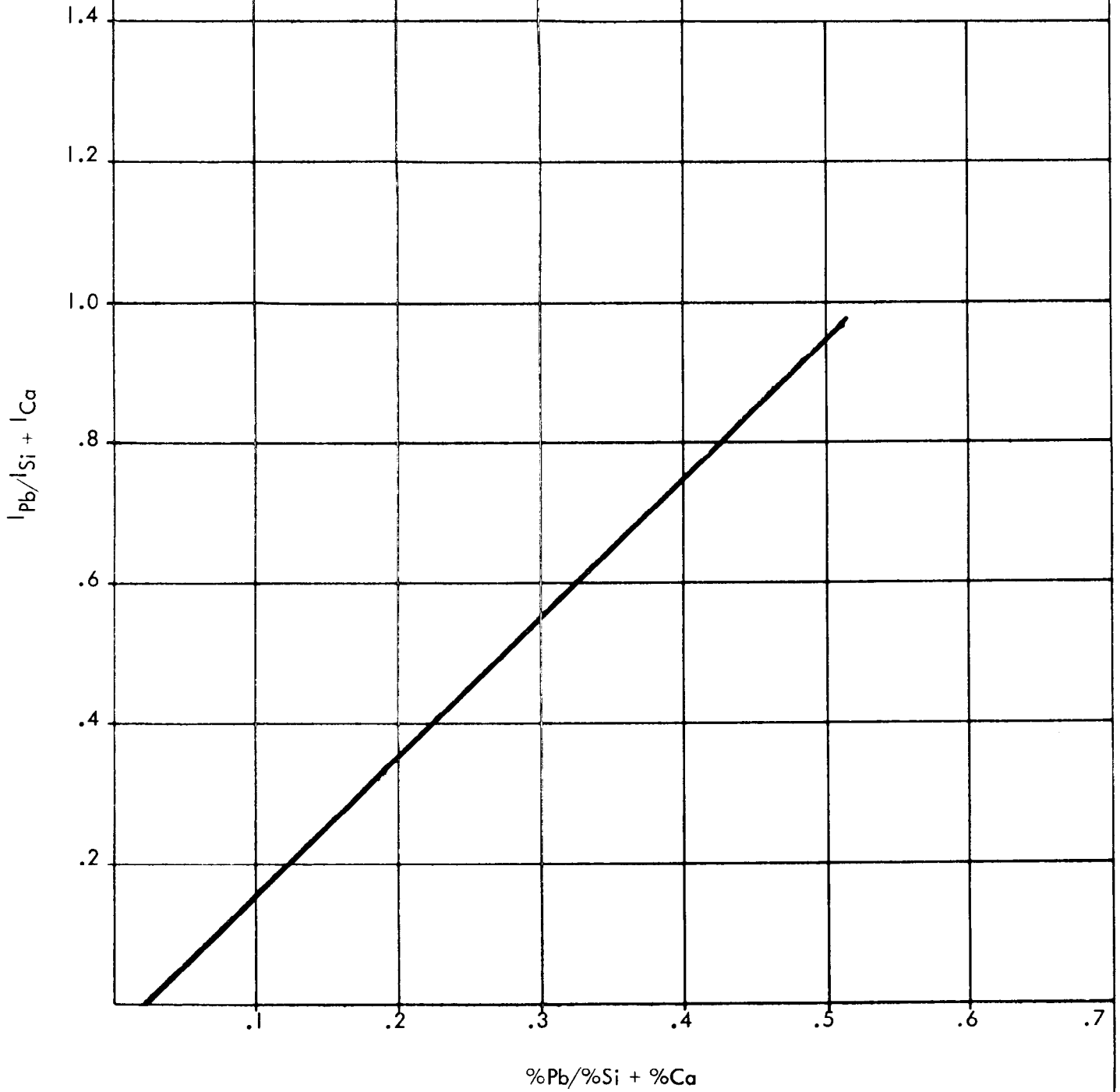


Graph 8-7  
Basic Lead Silico Chromate - Paint  
Fluorescence  
%Pb/%Fe vs.  $I_{Pb}/I_{Fe}$   
CC = .9839  
 $Y = -5.7768 + .9261x$

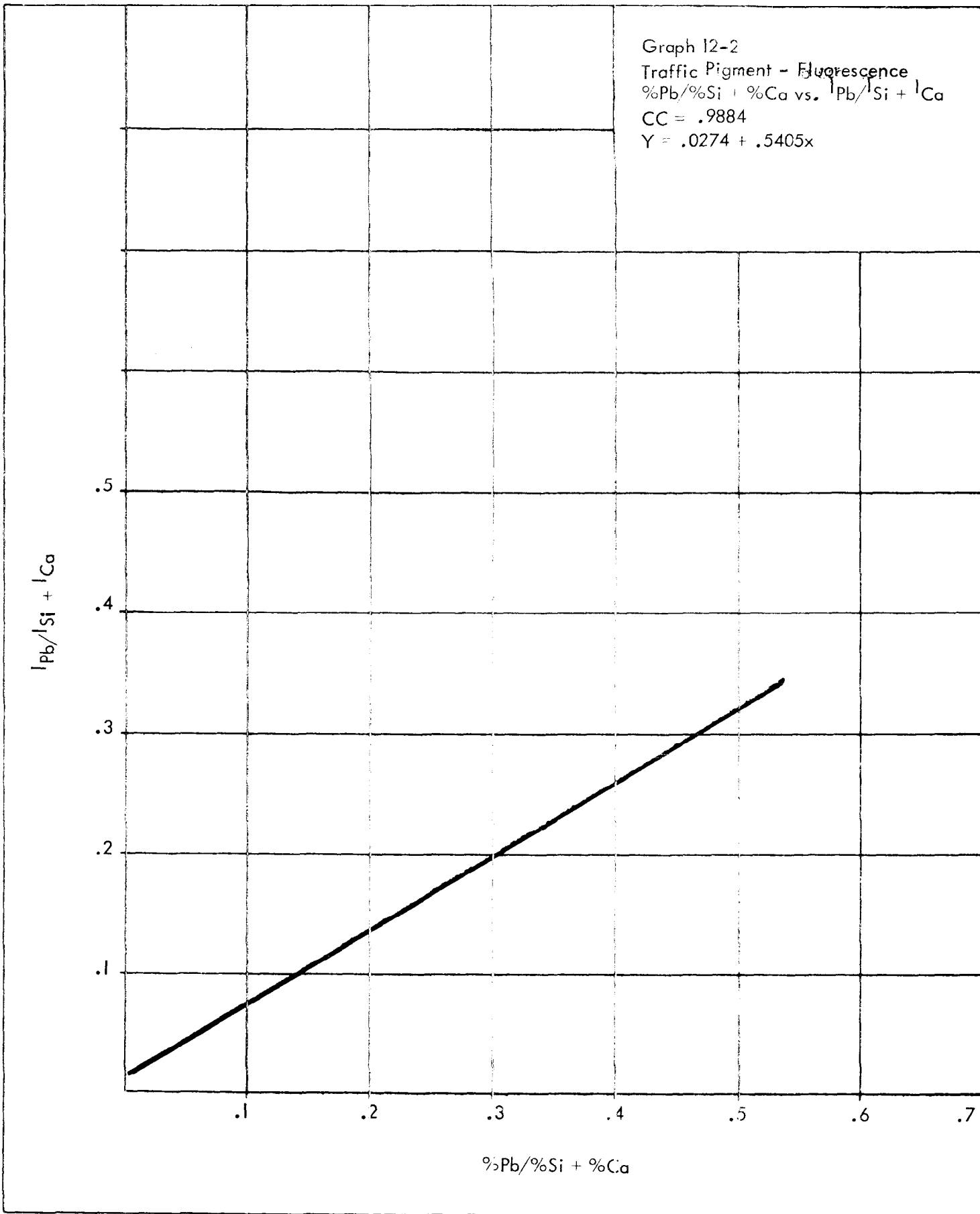




Graph 12-1  
Traffic Paint - Fluorescence  
%Pb/%Si + %Ca vs.  $I_{Pb}/I_{Si} + I_{Ca}$   
CC = .9903  
 $Y = -.0340 + 2.0207x$



Graph 12-2  
Traffic Pigment - Fluorescence  
%Pb/%Si + %Ca vs.  $I_{Pb}/I_{Si} + I_{Ca}$   
CC = .9884  
 $Y = .0274 + .5405x$



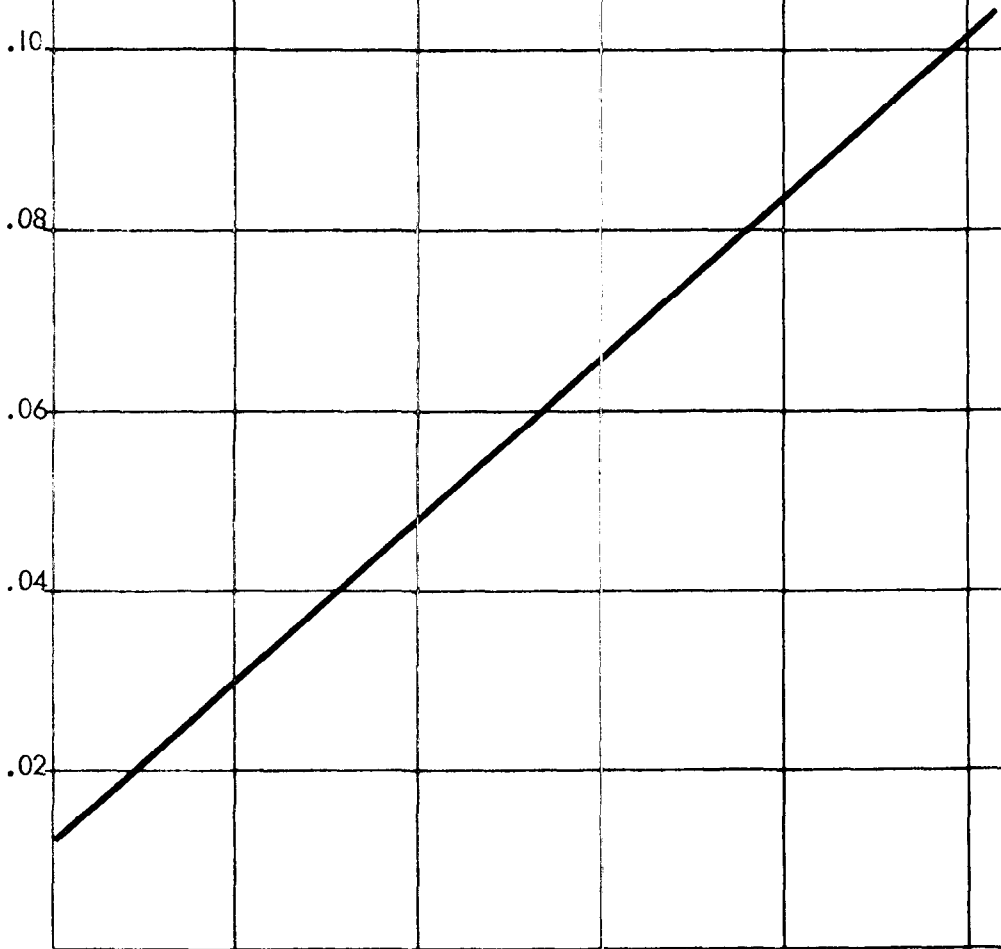
Graph 12-3  
Traffic Paint - Fluorescence  
%Si/%Ca + %Pb vs.  $I_{Si}/I_{Ca} + I_{Pb}$   
CC = .9519  
 $Y = .0066 + .4179x$

$I_{Si}/I_{Ca} + I_{Pb}$

.10  
.08  
.06  
.04  
.02

.05 .1 .15 .20

%Si/%Ca + %Pb



Graph 12-4  
Traffic Pigment - Fluorescence  
%Si/%Pb + %Ca vs.  $I_{Si}/I_{Pb} + I_{Ca}$   
CC - .9889  
 $Y = 1.3125 + 2.0625x$

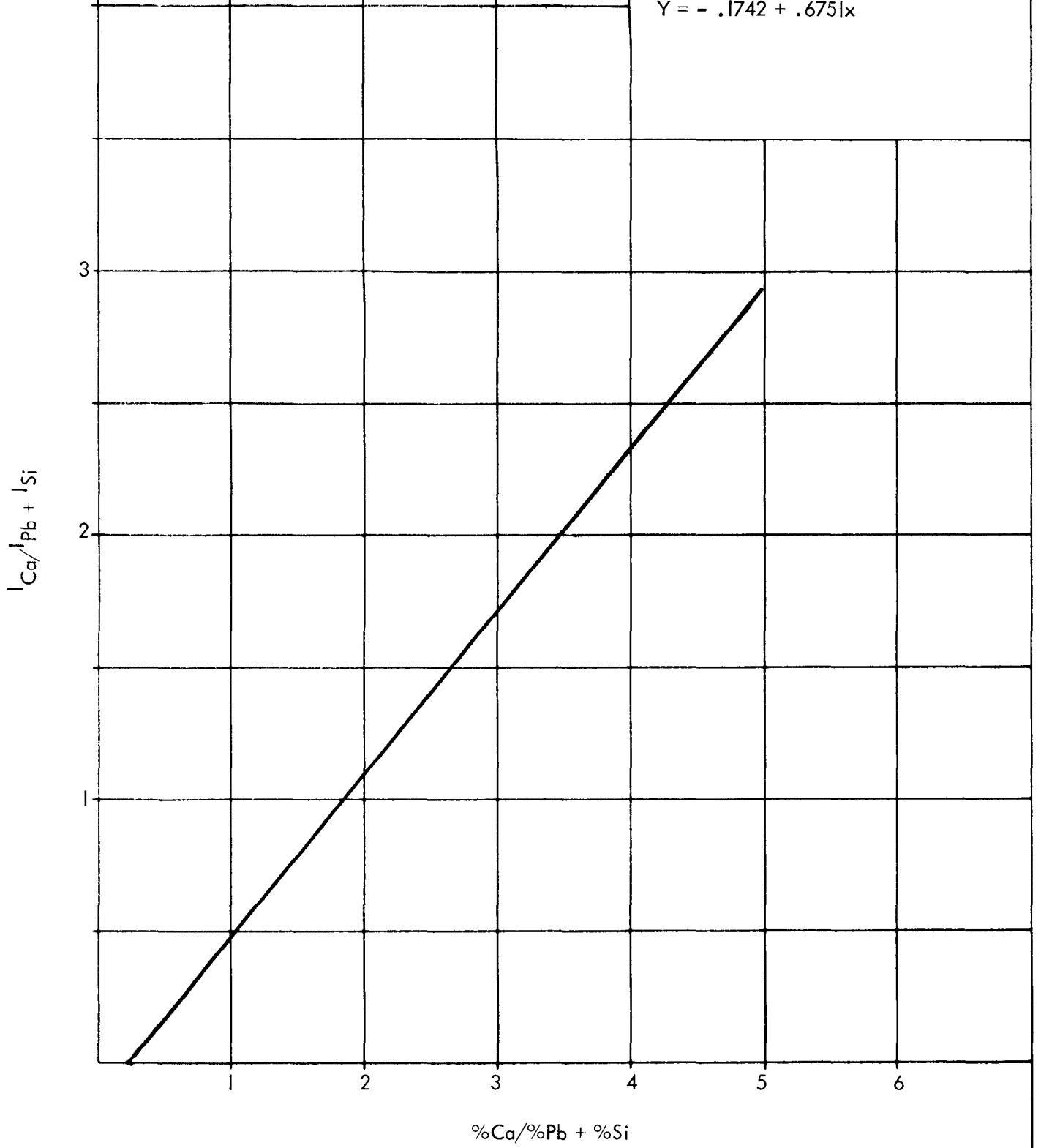
$I_{Si}/I_{Pb} + I_{Ca} (10^{-2})$

50  
40  
30  
20  
10

5 10 15 20

%Si/%Pb + %Ca ( $10^{-2}$ )

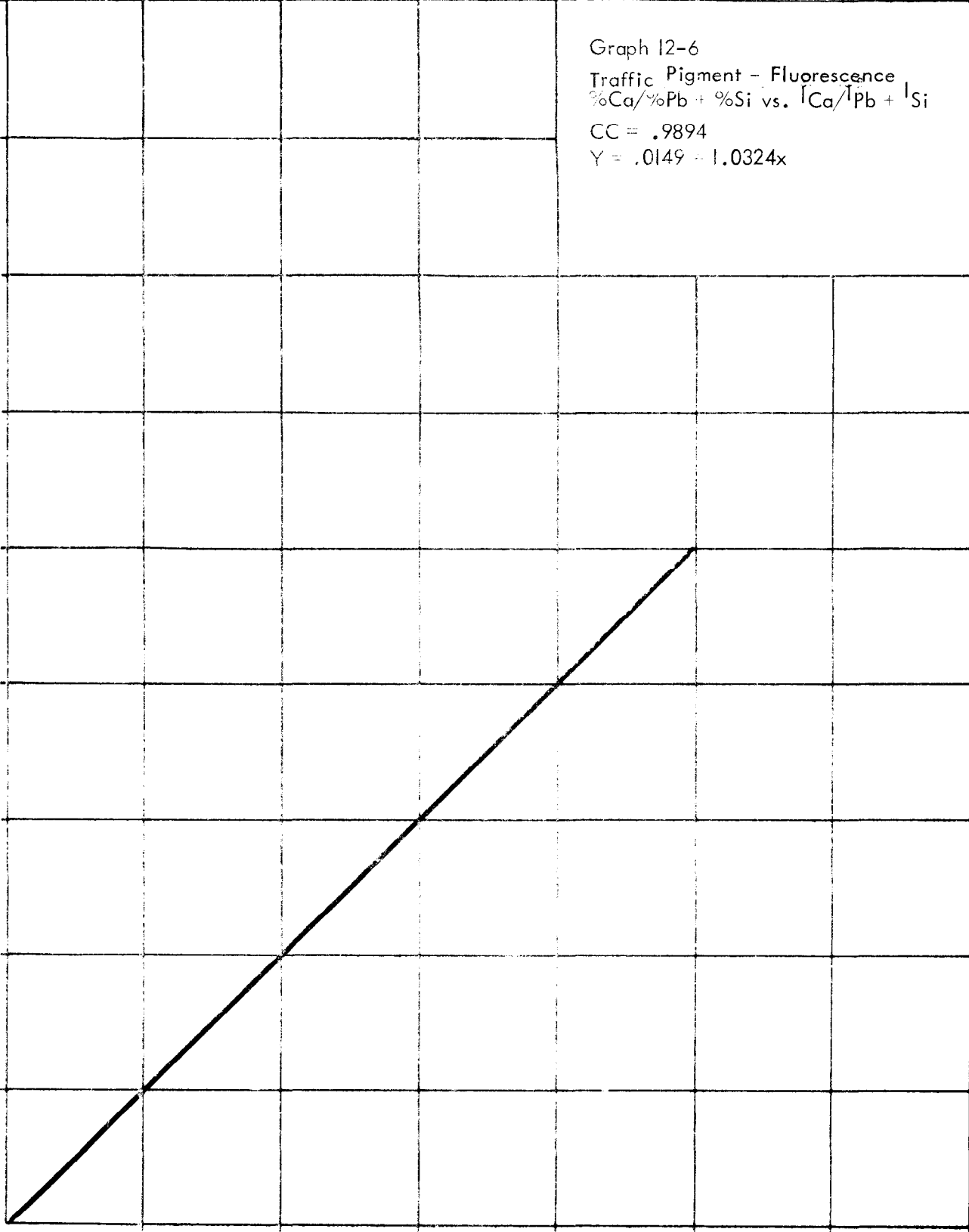
Graph 12-5  
Traffic Paint - Fluorescence  
%Ca/%Pb + %Si vs.  $I_{Ca}/I_{Pb} + I_{Si}$   
CC = .9757  
 $Y = -.1742 + .6751x$



Graph 12-6  
Traffic Pigment - Fluorescence  
%Ca/%Pb + %Si vs.  $I_{Ca}/I_{Pb} + I_{Si}$   
CC = .9894  
 $Y = .0149 + 1.0324x$

$I_{Ca}/I_{Pb} + I_{Si}$

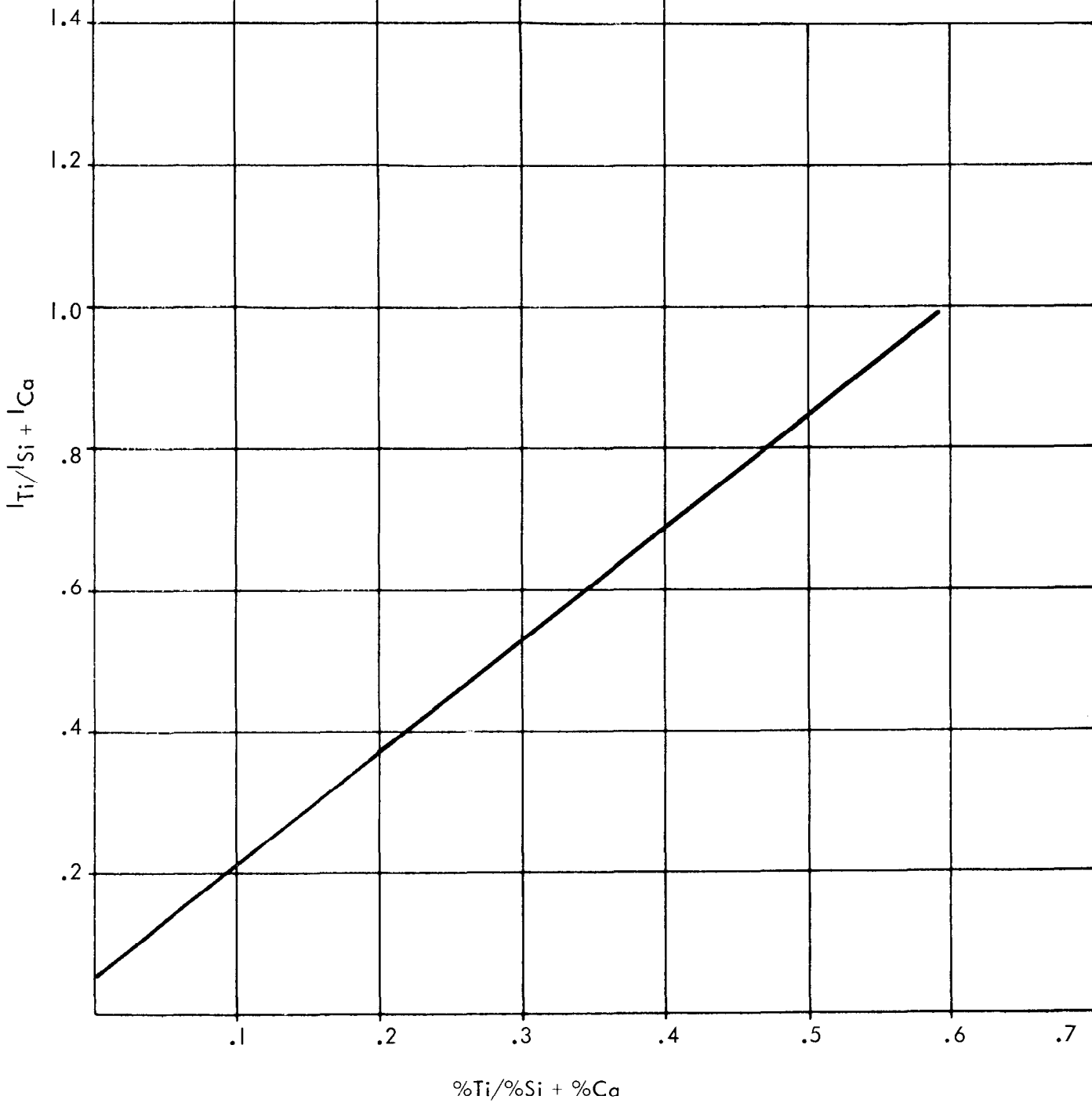
6  
5  
4  
3  
2  
1



%Ca/%Pb + %Si



Graph 12-7  
Traffic Paint - Fluorescence  
%Ti/%Si + %Ca vs. I<sub>Ti</sub>/I<sub>Si</sub> + I<sub>Ca</sub>  
CC = .9504  
Y = .0886 + 1.3552x



Graph 12-8  
Traffic Pigment - Fluorescence  
%Ti/%Si + %Ca vs. I<sub>Ti</sub>/I<sub>Si</sub> + I<sub>Ca</sub>  
CC = .9597  
Y = .0448 + .2632x

I<sub>Ti</sub>/I<sub>Si</sub> + I<sub>Ca</sub>

.4

.3

.2

.1

.1

.2

.3

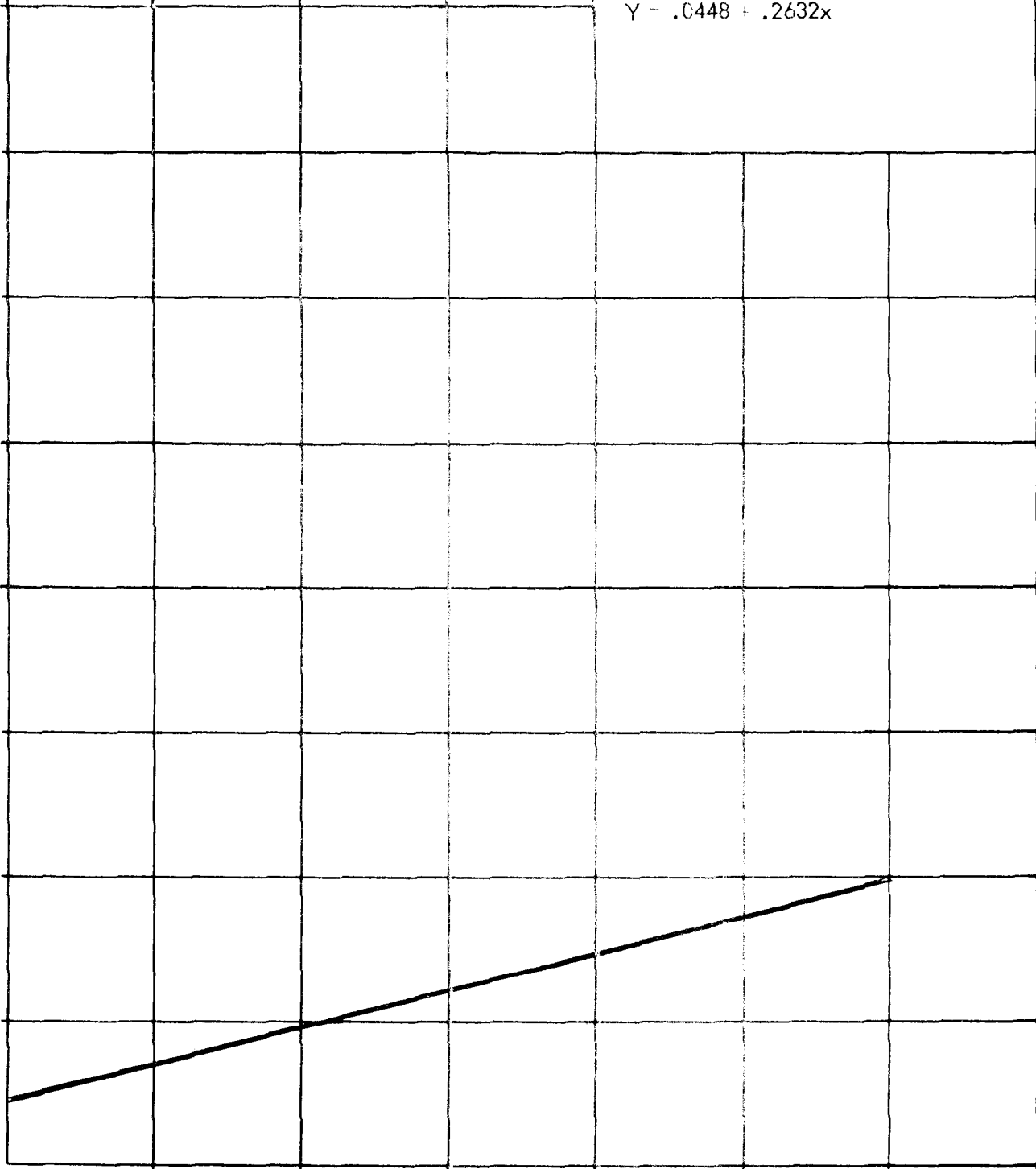
.4

.5

.6

.7

%Ti/%Si + %Ca



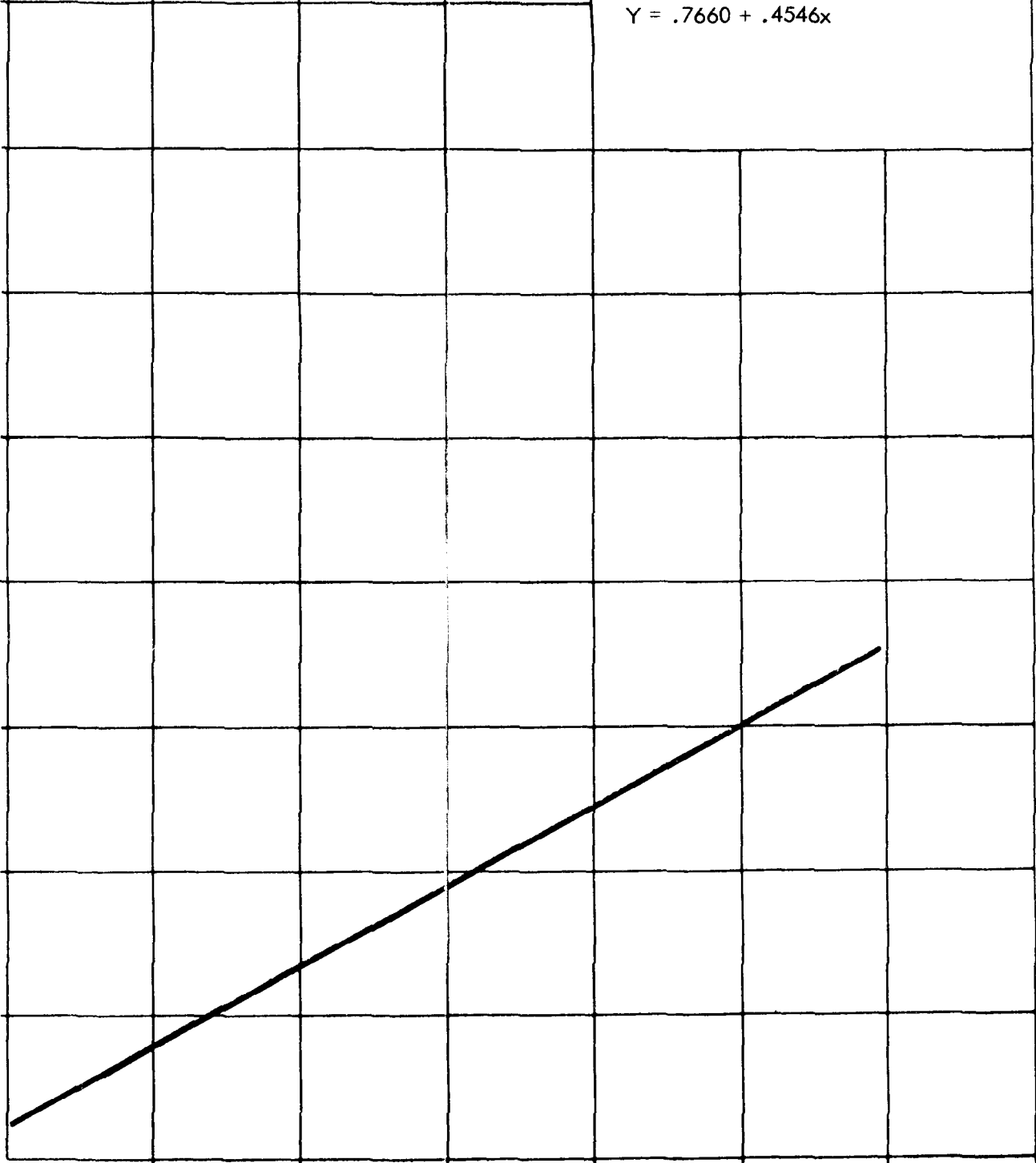
Graph 12-9  
White Traffic Paint - Fluorescence  
%Si/%Ti + %Ca vs.  $I_{Si}/I_{Ti} + I_{Ca}$   
CC = .9920  
 $Y = .7660 + .4546x$

$I_{Si}/I_{Ti} + I_{Ca} (10^{-2})$

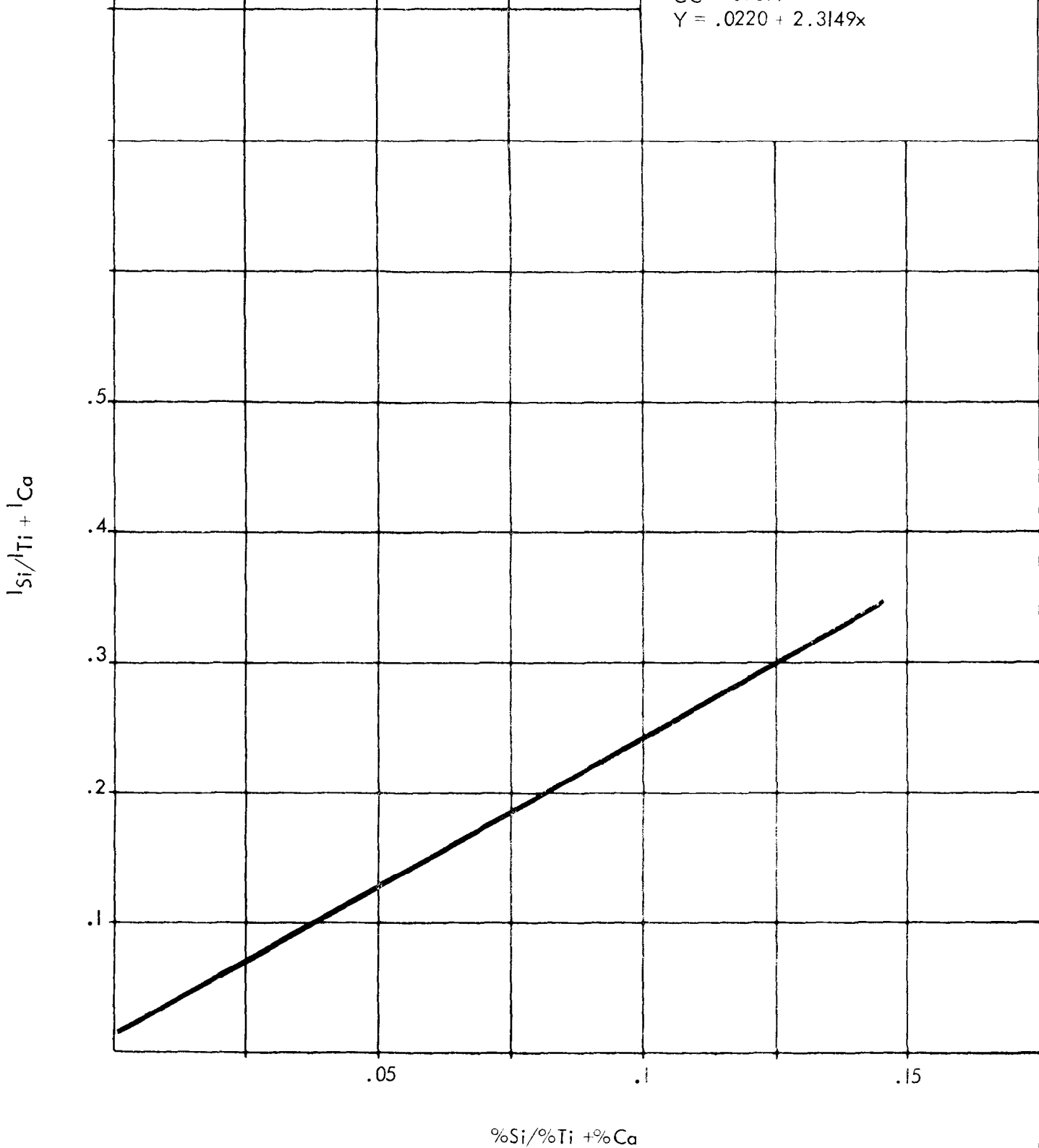
10  
8  
6  
4  
2

5 10 15

%Si/%Ti + %Ca(10<sup>-2</sup>)



Graph 12-10  
White Traffic Pigment - Fluorescence  
%Si/%Ti + %Ca vs. I<sub>Si</sub>/I<sub>Ti</sub> + I<sub>Ca</sub>  
CC = .9691  
Y = .0220 + 2.3149x



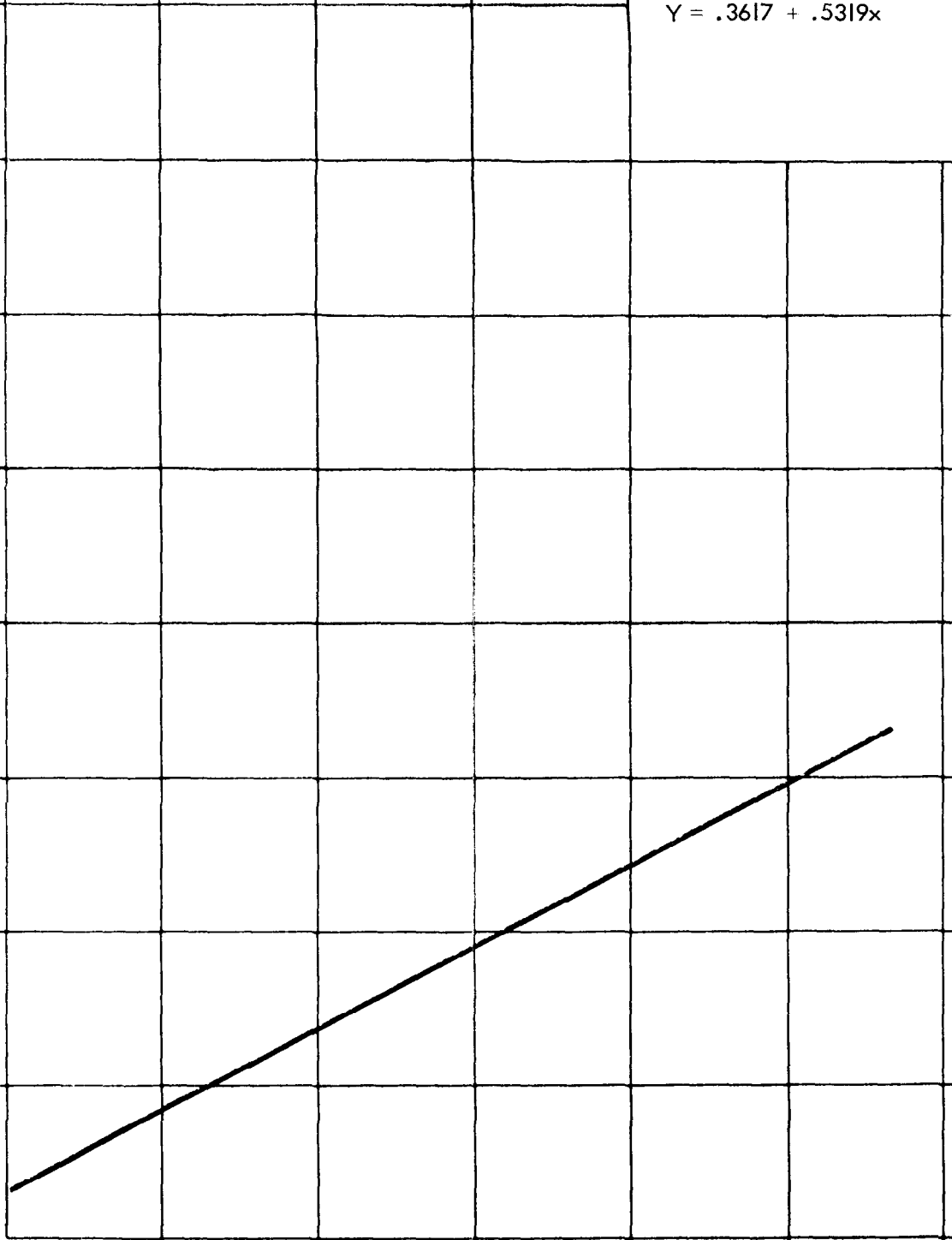
Graph 12-11  
White Traffic Paint - Fluorescence  
%Ca/%Ti + %Si vs. I<sub>Ca</sub>/I<sub>Ti</sub> + I<sub>Si</sub>  
CC = .9888  
Y = .3617 + .5319x

I<sub>Ca</sub>/I<sub>Ti</sub> + I<sub>Si</sub>

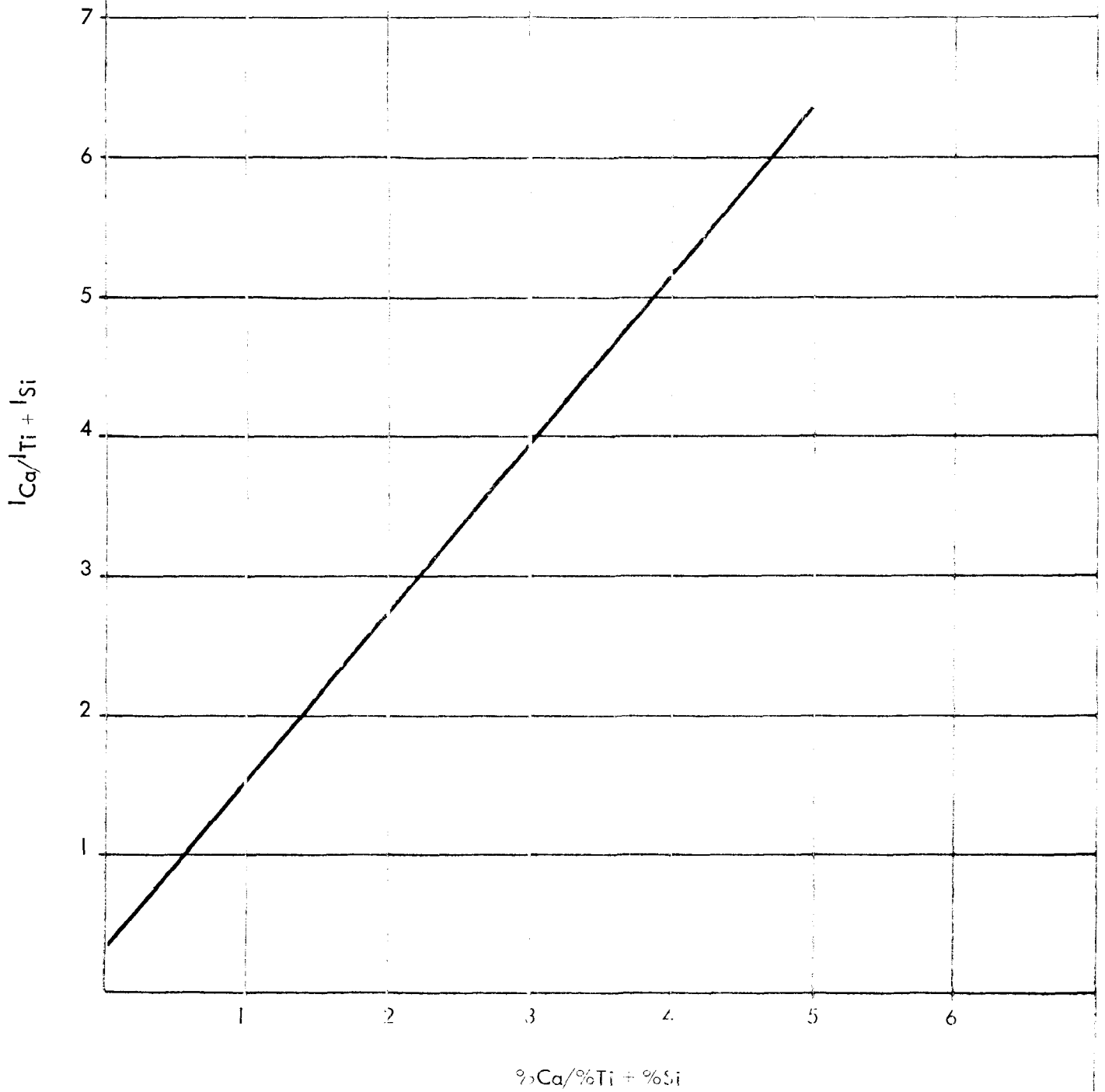
5  
4  
3  
2  
1

1 2 3 4 5 6

%Ca/%Ti + %Si



Graph I2-12  
White Traffic Pigment - Fluorescence  
%Ca/%Ti + %Si vs.  $I_{Ca}/I_{Ti} + I_{Si}$   
CC = .8612  
 $Y = .4943 + 1.0862x$



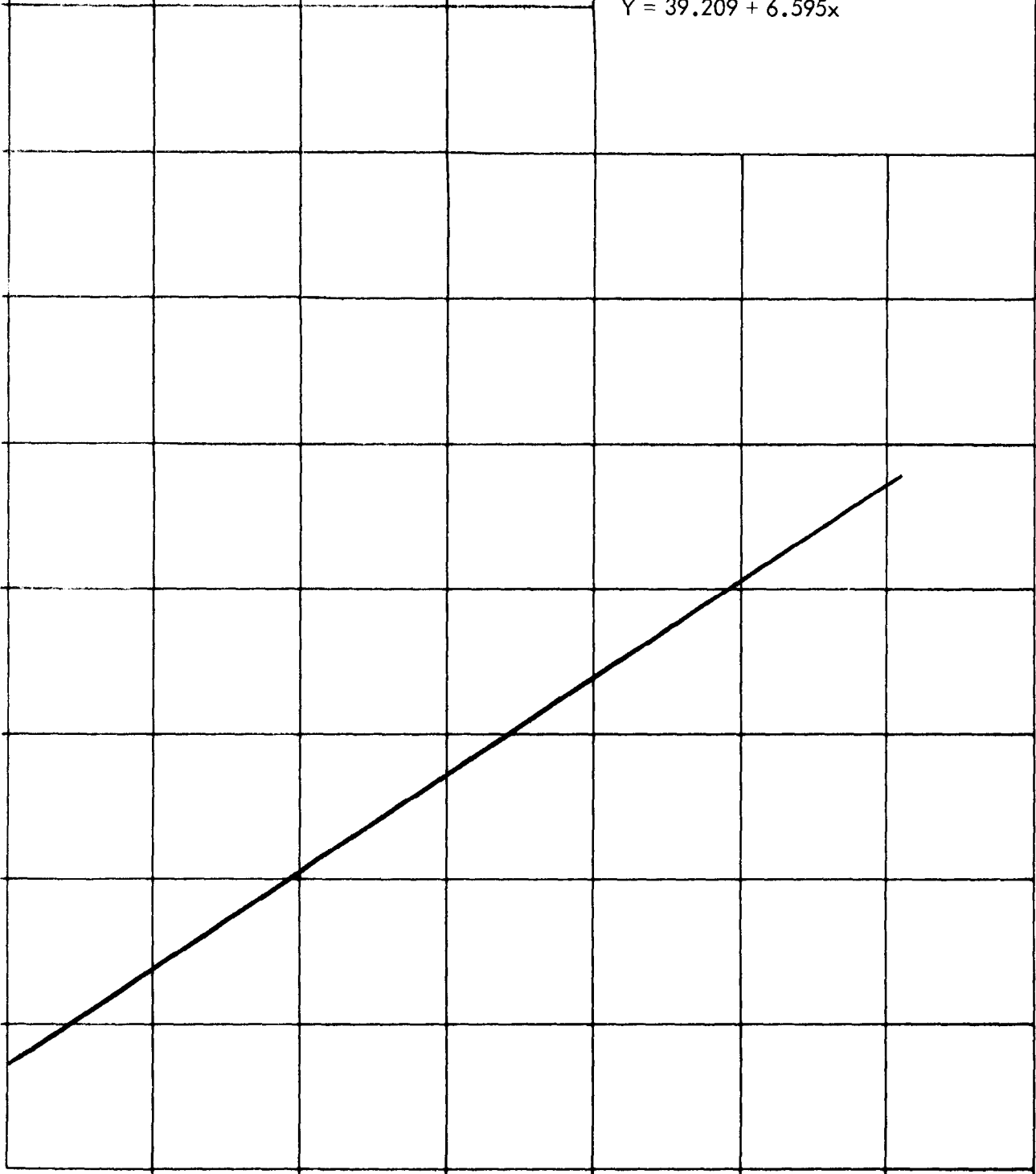
Graph 13-1  
Yellow Traffic Paint - Diffraction  
%Chrome Yellow/<sup>I</sup>Chrome Yellow  
CC = .9703  
 $Y = 39.209 + 6.595x$

<sup>I</sup>Chrome Yellow ( $10^{-2}$ )

300  
250  
200  
150  
100  
50

10 20 30

%Chrome Yellow



Graph 13-2  
Yellow Traffic Pigment - Diffraction  
%Chrome Yellow vs.  $I_{\text{Chrome Yellow}}$   
CC = .98  
 $Y = -6.27 + 6.984x$

$I_{\text{Chrome Yellow}} (10^{-2})$

300

200

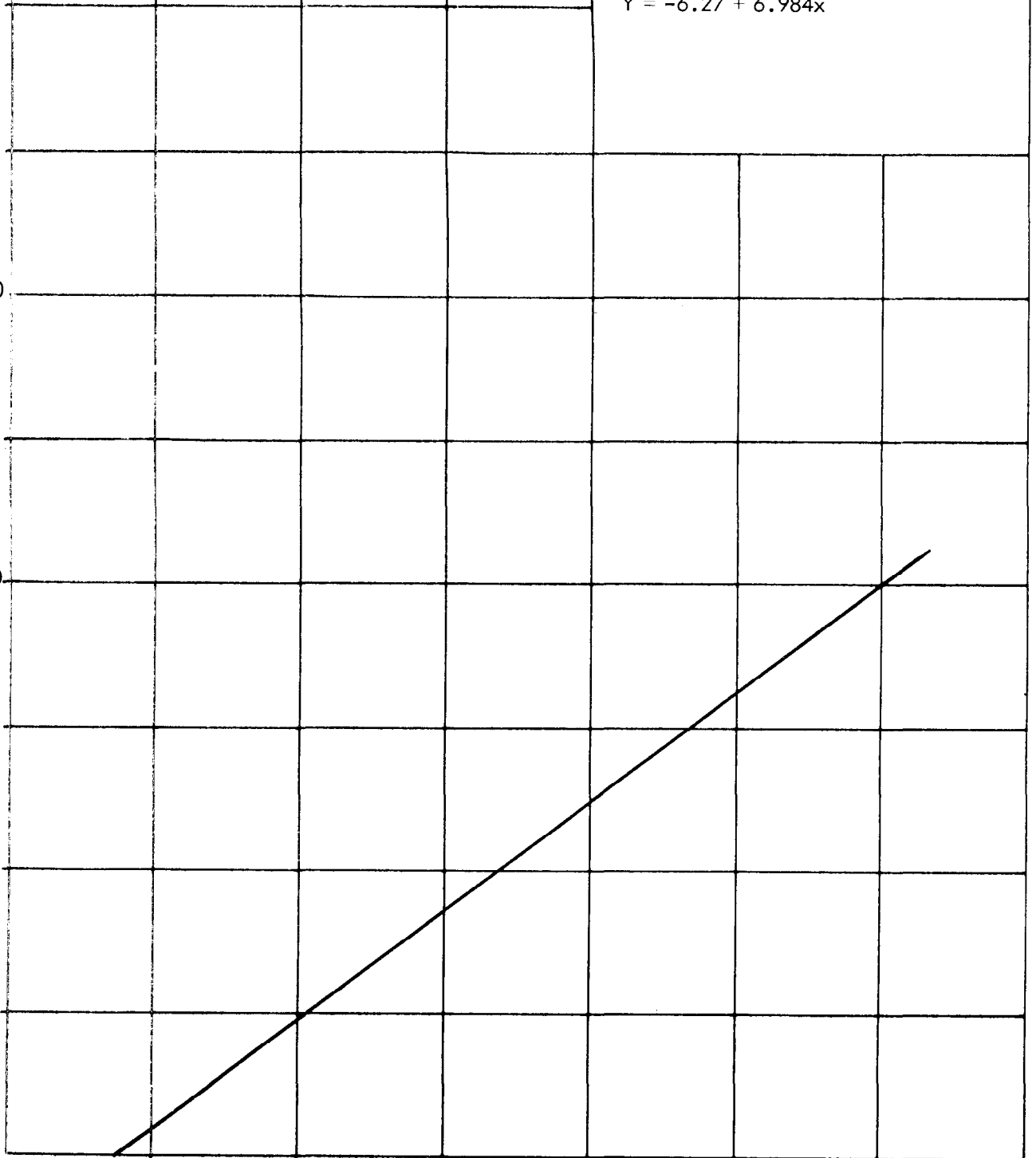
100

10

20

30

%Chrome Yellow





Graph 13-3

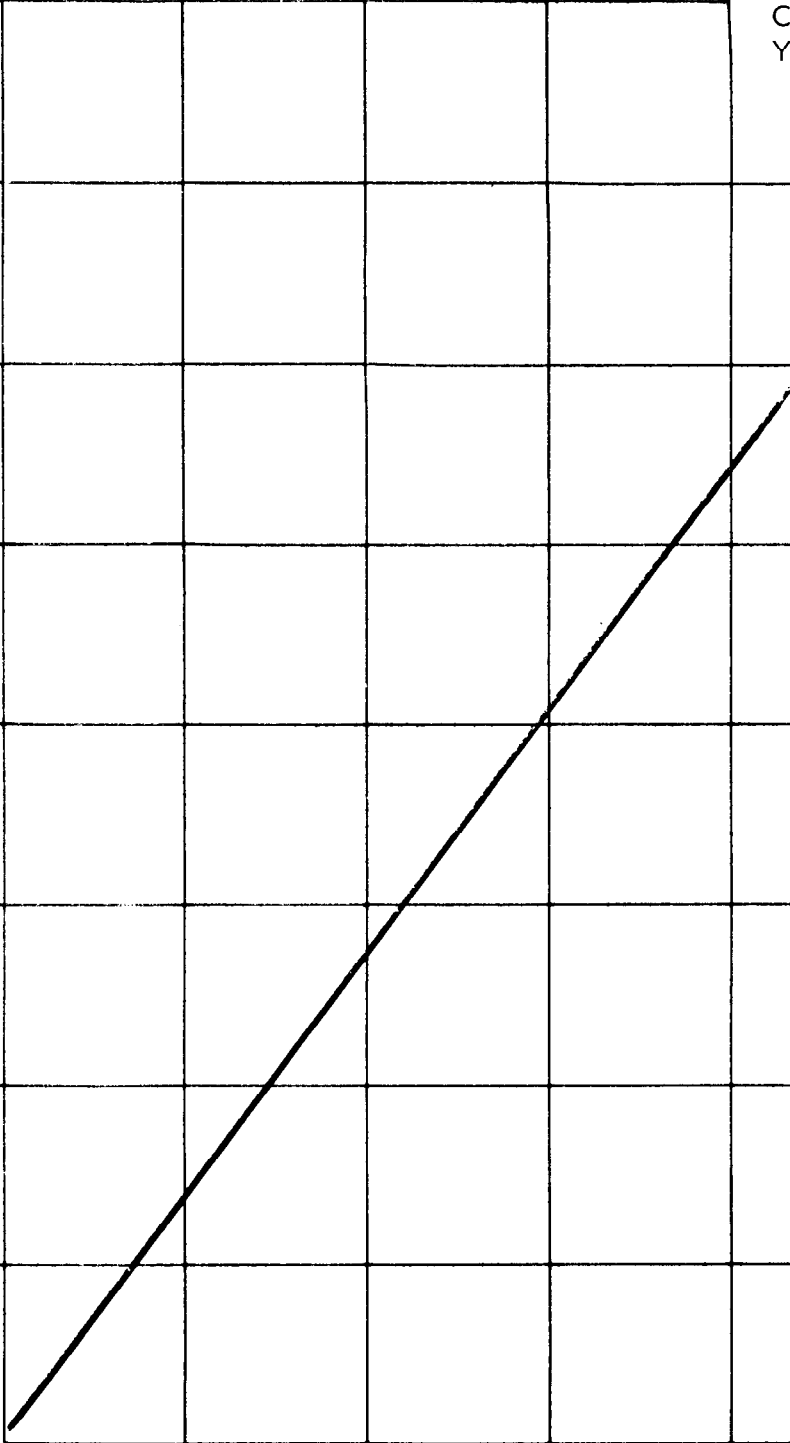
White Traffic Paint - Diffraction  
 $\%TiO_2/\%Talc + \%CaCO_3$  vs.  
 $I_{TiO_2}/I_{Talc + CaCO_3}$   
CC = .9951  
 $Y = 0.0157 + 1.3125x$

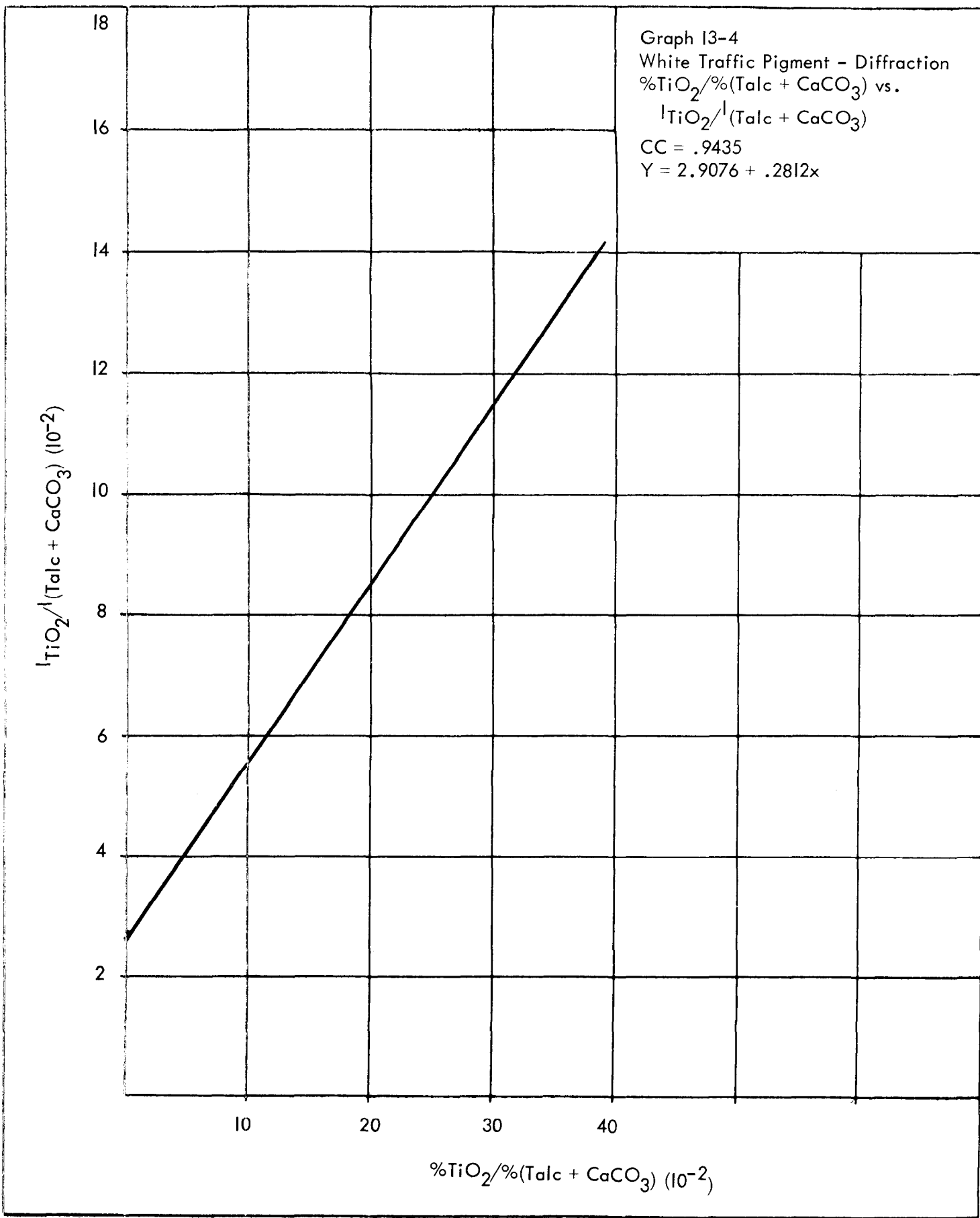
$I_{TiO_2}/I_{Talc + CaCO_3}$

.6  
.5  
.4  
.3  
.2  
.1

.1 .2 .3 .4 .5 .6

$\%TiO_2/\%Talc + \%CaCO_3$





Graph I3-5

White Traffic Paint - Diffraction

%Talc/%TiO<sub>2</sub> + %CaCO<sub>3</sub> vs.  
I<sub>Talc</sub>/I<sub>TiO<sub>2</sub></sub> + I<sub>CaCO<sub>3</sub></sub>

CC = .9852

Y = 0.0004 + 0.5025x

I<sub>Talc</sub>/I<sub>TiO<sub>2</sub></sub> + I<sub>CaCO<sub>3</sub></sub>

.16

.14

.12

.10

.08

.06

.04

.02

.1

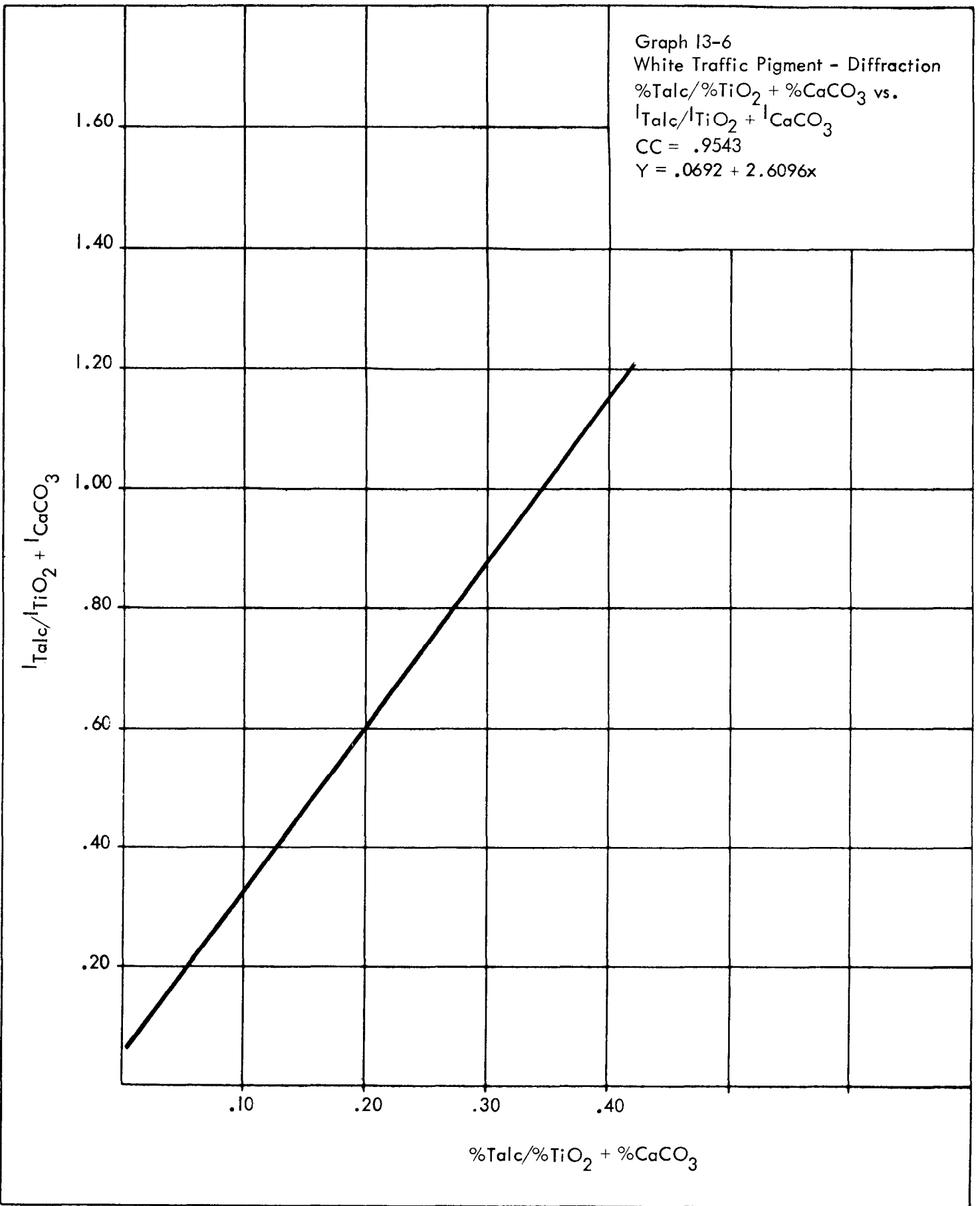
.2

.3

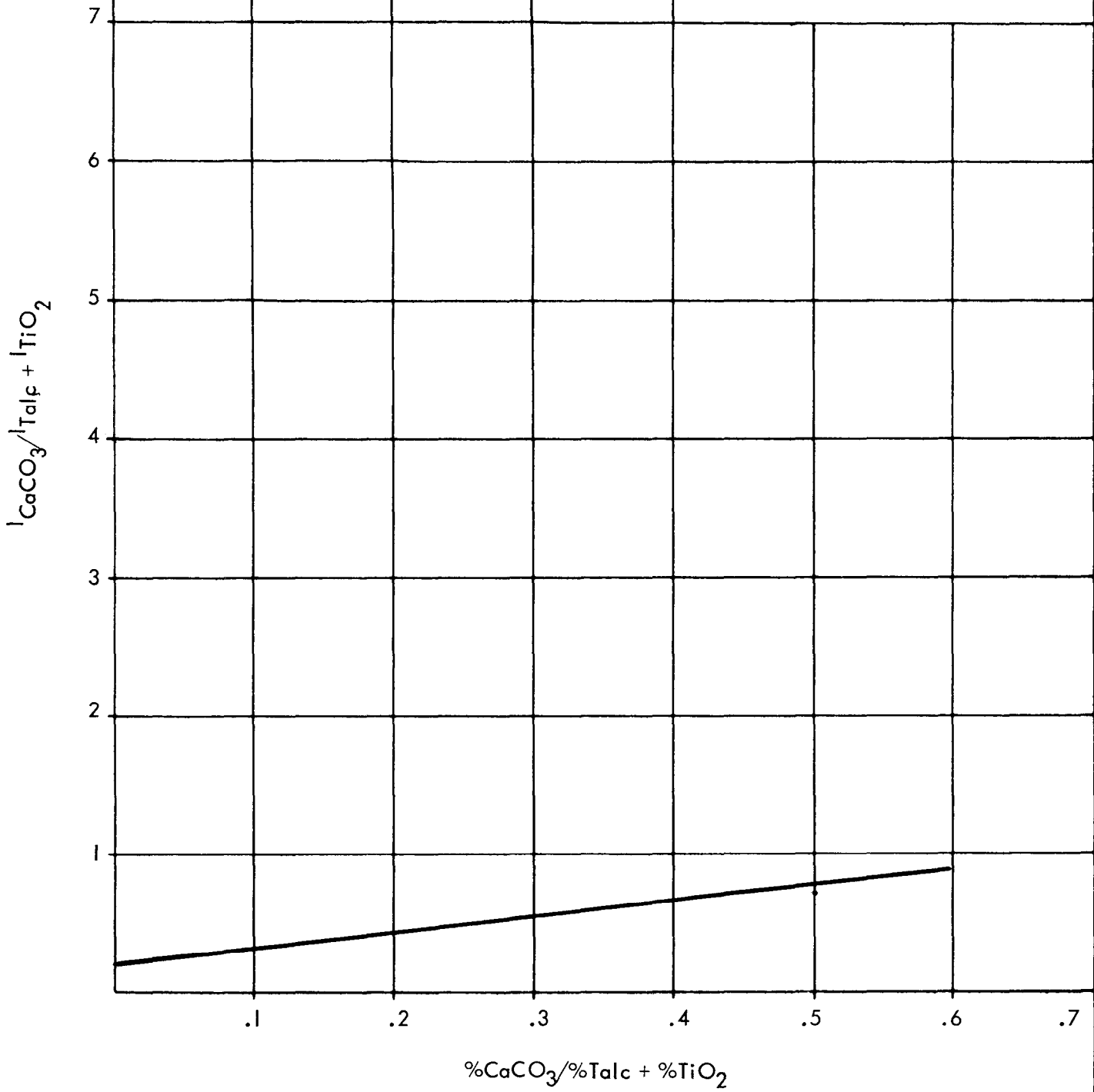
.4

%Talc/%TiO<sub>2</sub> + %CaCO<sub>3</sub>

Graph 13-6  
White Traffic Pigment - Diffraction  
%Talc/%TiO<sub>2</sub> + %CaCO<sub>3</sub> vs.  
 $I_{\text{Talc}}/I_{\text{TiO}_2} + I_{\text{CaCO}_3}$   
CC = .9543  
 $Y = .0692 + 2.6096x$



Graph 13-7  
White Traffic Paint - Diffraction  
 $\%CaCO_3/\%Talc + \%TiO_2$  vs.  
 $I_{CaCO_3}/I_{Talc} + I_{TiO_2}$   
CC = .9812  
Y = 0.2121 + 0.9561x



Graph 13-8  
White Traffic Pigment - Diffraction  
%CaCO<sub>3</sub>/%TiO<sub>2</sub> + %Talc vs.

$$I_{CaCO_3} / I_{TiO_2} + I_{Talc}$$

$$CC = .9042$$

$$Y = .4380 + .5581x$$

$I_{CaCO_3} / I_{TiO_2} + I_{Talc}$

4.0

3.0

2.0

1.0

.10

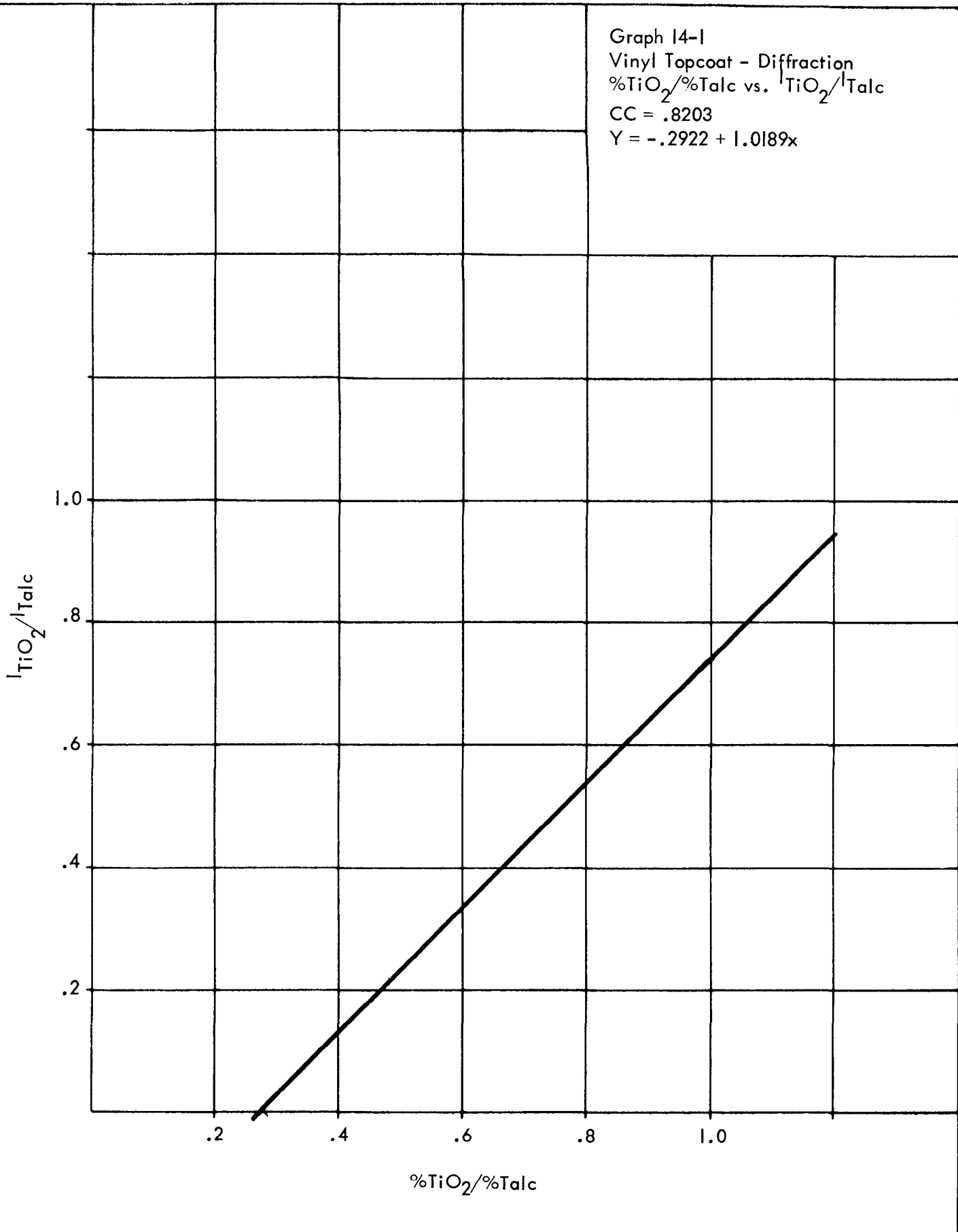
.20

.3

.4

%CaCO<sub>3</sub>/%TiO<sub>2</sub> + %Talc

Graph 14-1  
Vinyl Topcoat - Diffraction  
%TiO<sub>2</sub>/<sub>%Talc vs. I<sub>TiO<sub>2</sub></sub>/I<sub>Talc</sub>  
CC = .8203  
Y = -.2922 + 1.0189x</sub>



Graph 14-2  
Vinyl Topcoat - Diffraction  
%Talc/%TiO<sub>2</sub> vs. I<sub>Talc</sub>/I<sub>TiO<sub>2</sub></sub>  
CC = .8719  
Y = -1.2884 + 2.5773x

I<sub>Talc</sub>/I<sub>TiO<sub>2</sub></sub>

3

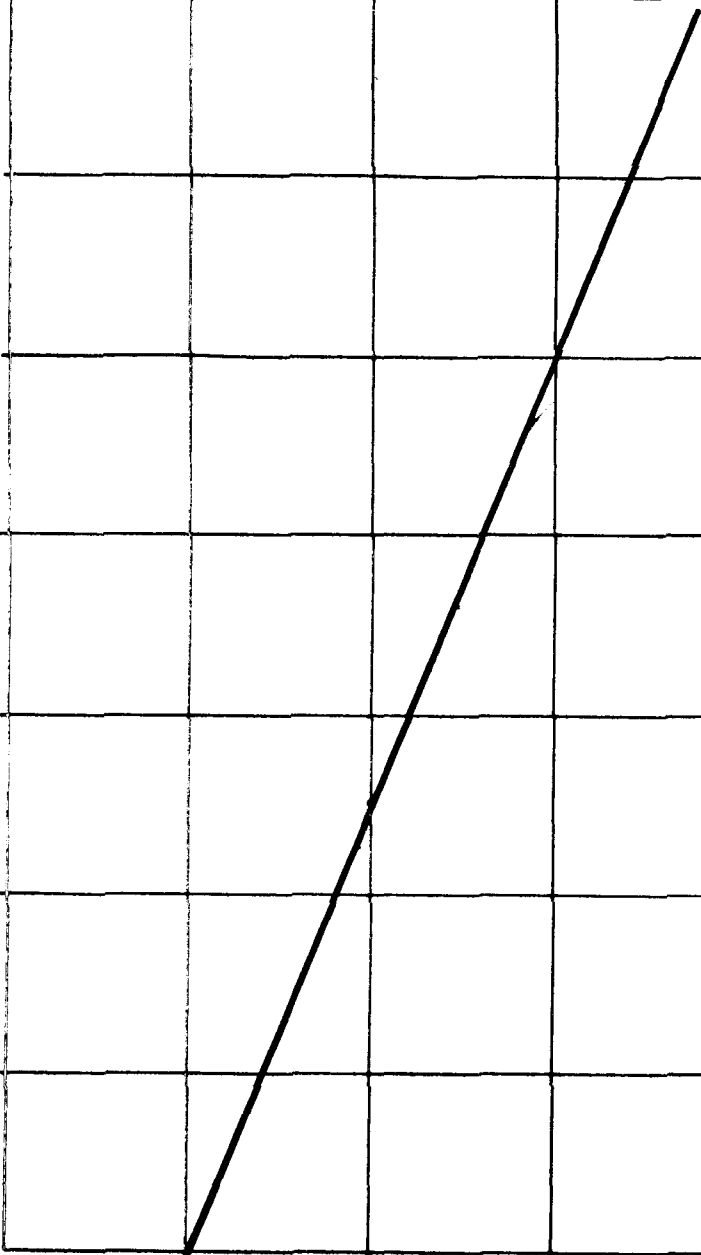
2

1

1

2

%Talc/%TiO<sub>2</sub>





Graph 15-1  
Vinyl Topcoat - Fluorescence  
%Ti/%Mg vs.  $I_{Ti}/I_{Mg}$   
CC = .3850

$I_{Ti}/I_{Mg}$

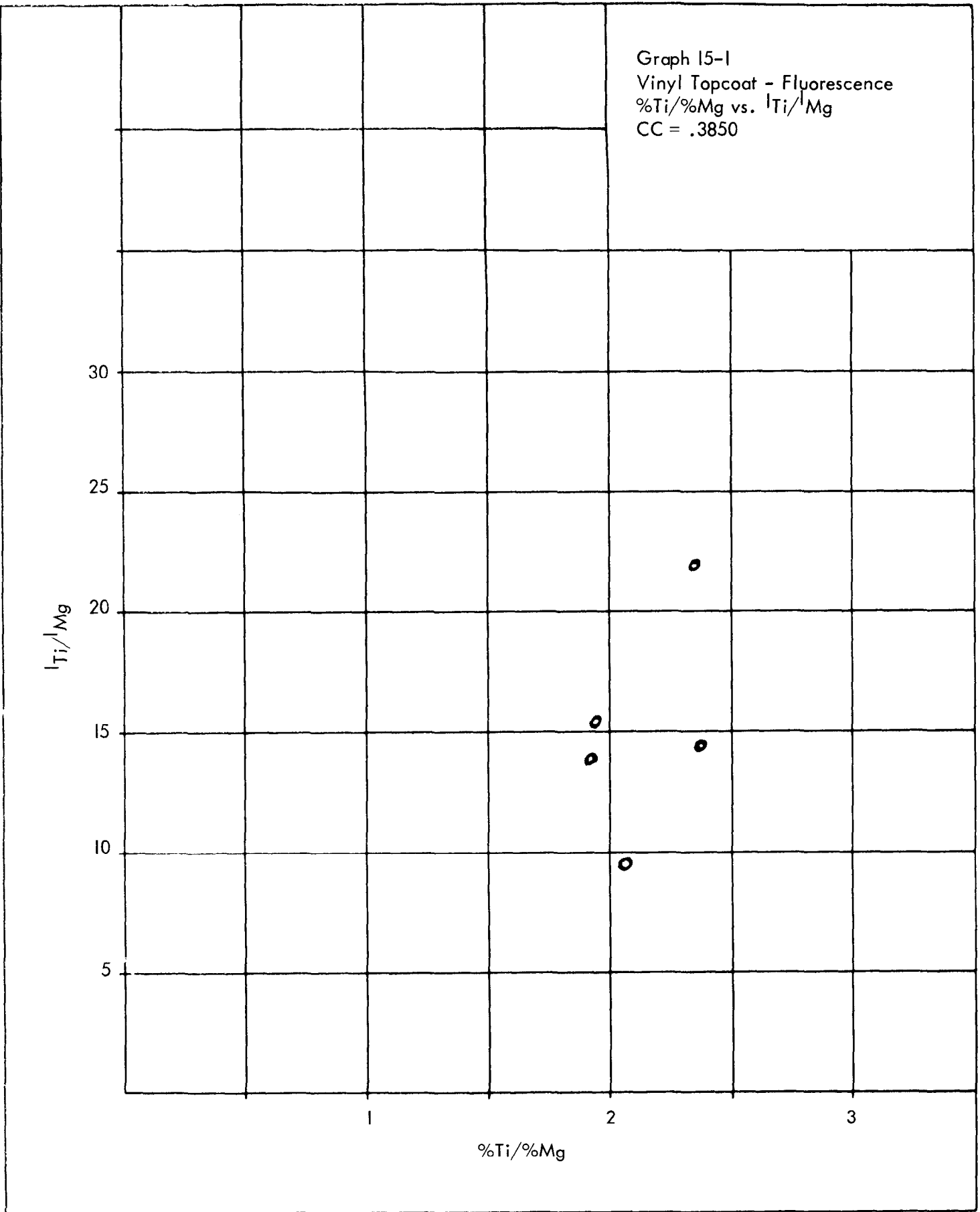
30  
25  
20  
15  
10  
5

%Ti/%Mg

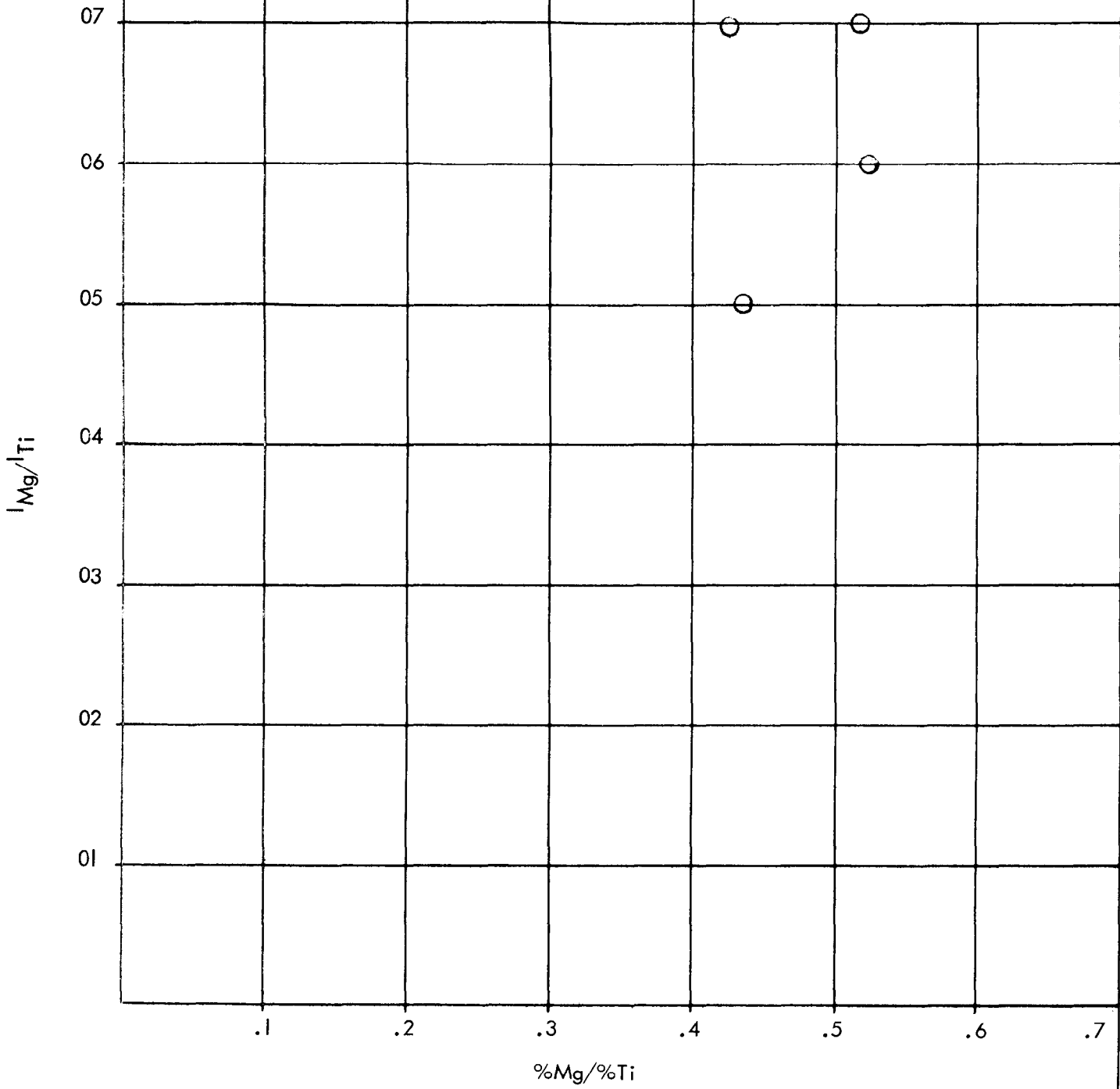
1

2

3



Graph 15-2  
Vinyl Topcoat - Fluorescence  
%Mg/%Ti vs.  $I_{Mg}/I_{Ti}$   
CC = 0.00



## FIGURES

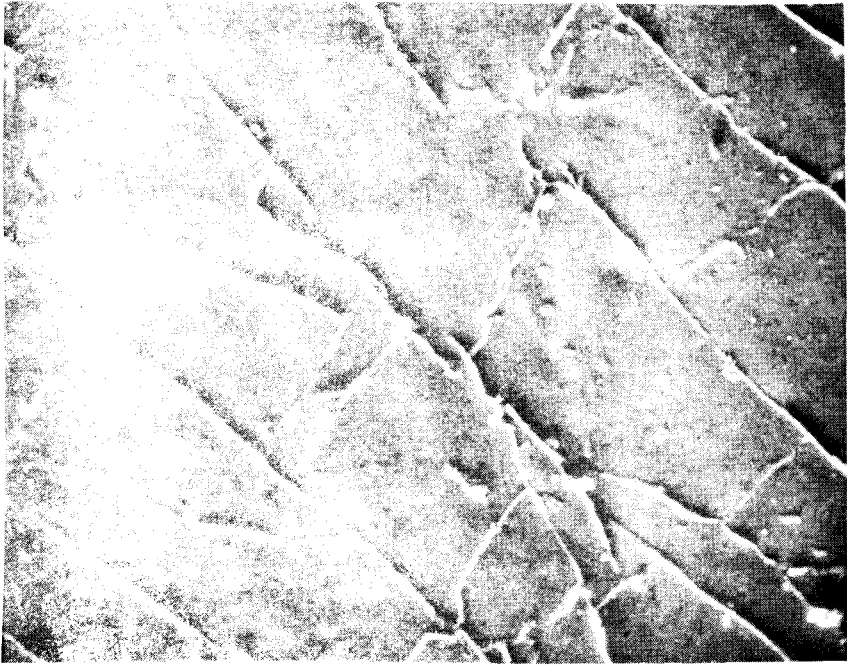


Fig. 1. Surface of 45, Tape Side, x100

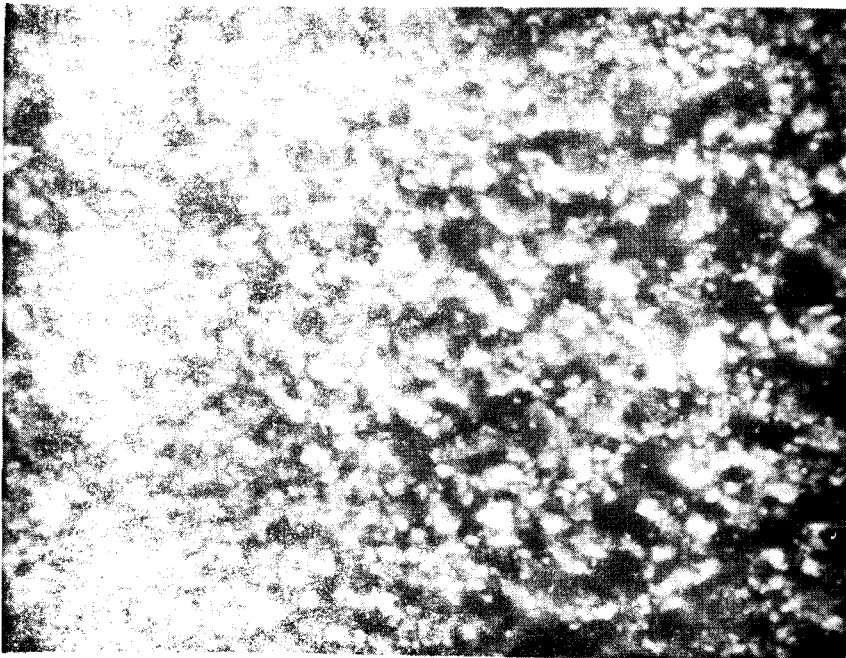


Fig. 2. Surface of 45, Tape Side, x2000



Figure 3. Mixture 48, Air Side, x100

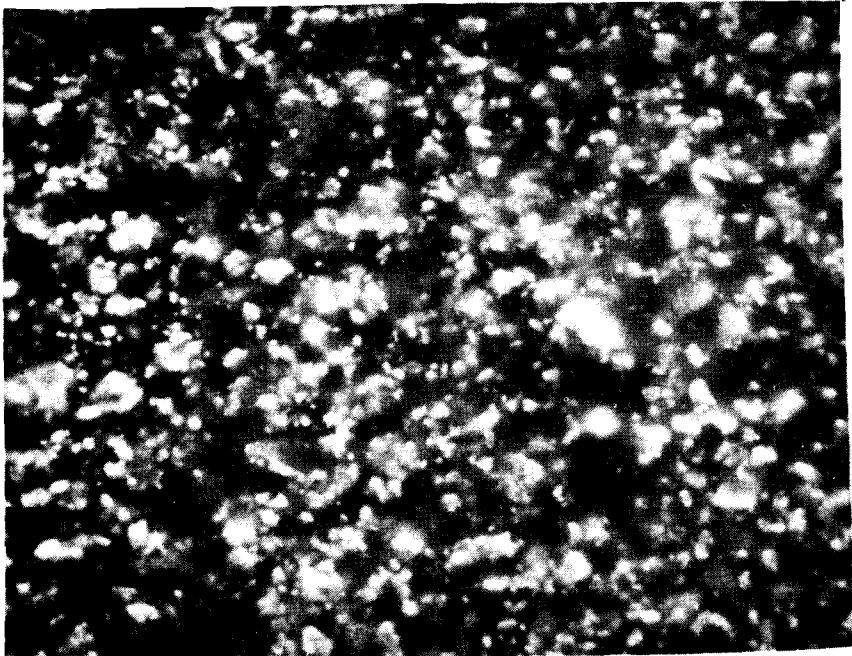


Figure 4. Mixture 48 Air Side, x2000

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