



Western Michigan University  
ScholarWorks at WMU

---

Paper Engineering Senior Theses

Chemical and Paper Engineering

---

6-1953

## A More Rapid Routine Control Method for the Estimation of Titanium Dioxide in Paper

Allen E. Uhl  
*Western Michigan University*

Follow this and additional works at: <https://scholarworks.wmich.edu/engineer-senior-theses>



Part of the Wood Science and Pulp, Paper Technology Commons

---

### Recommended Citation

Uhl, Allen E., "A More Rapid Routine Control Method for the Estimation of Titanium Dioxide in Paper" (1953). *Paper Engineering Senior Theses*. 549.  
<https://scholarworks.wmich.edu/engineer-senior-theses/549>

This Dissertation/Thesis is brought to you for free and open access by the Chemical and Paper Engineering at ScholarWorks at WMU. It has been accepted for inclusion in Paper Engineering Senior Theses by an authorized administrator of ScholarWorks at WMU. For more information, please contact [wmu-scholarworks@wmich.edu](mailto:wmu-scholarworks@wmich.edu).



A MORE RAPID ROUTINE CONTROL  
~~METHOD~~ FOR THE ESTIMATION OF  
TITANIUM DIOXIDE IN PAPER

TS  
9999  
434

Senior Thesis

A paper submitted as partial fulfillment  
of graduation requirements in the Pulp and Paper  
Technology Curriculum at Western Michigan College.

Mr. R.T. Elias  
Instructor

Submitted June 1, 1953  
by Allen E. Uhl

//

WESTERN MICHIGAN UNIVERSITY LIBRARY  
KALAMAZOO, MICHIGAN 49001

## TABLE OF CONTENTS

### A More Rapid Routine Control Method for The Estimation of Titanium Dioxide in Paper

	Page
Abstract - - - - -	1
Part I - - A Literature Survey - - - - -	1-13
Use and Advantage of Titanium - - - - -	1
Comparison of Titanium Compounds with Clay- - - - -	2 Tables I-II
Present Methods of Analysis - - - - -	3
Means of Estimating the Concentration of a Solution by its Color - - - - -	4
A Rapid and Accurate Method to Determine Concentration - - - - -	7 Figures 1-2
Apparatus - - - - -	11 Figures 3-4
Literature Cited - - - - -	-13
Outline of Experimental Work - - - - -	-14
Part II - - Laboratory Research - - - - -	16
A. Results Using the TAPPI Volumetric Method - - - - -	-16 Table III Figure 5
B. Brief Account of the TAPPI Colorimetric Method and the Routine Control Method - - - - -	-19
C. Quantitative Estimation Without Ashing the Paper - - - - -	20 Tables IV-V Figures 6-7
Summary - - - - -	-28

## ABSTRACT

A procedure is given for estimation of titanium dioxide in paper, whereby ashing the sample is omitted thru simultaneous destruction of the paper and solution of the titanium. This method is compared with the standard methods of analysis in regard to time required and precision.

# A MORE RAPID ROUTINE CONTROL METHOD FOR THE DETERMINATION OF TITANIUM DIOXIDE IN PAPER

## Literature Survey

### USE AND ADVANTAGE OF TITANIUM

#### High Index of Refraction

Titanium dioxide is widely used in many varieties of paper because of its high index of refraction and high brightness. When it is used in making paper, it gives a white sheet with a high degree of opacity.

Fillers with a high index of refraction have a greater light scattering property and will consequently increase the opacity more than will fillers with a low index. Titanium dioxide has an index of refraction of 2.71 as compared with clay 1.55, talc 1.57 and chalk 1.56<sup>(1)</sup>. If a mineral filler is used which has about the same index of refraction as that of cellulose, namely 1.53, there will not be a great increase in opacity unless large amounts are used. Table I below shows figures giving the relative values of titanium and clay. These figures show that even when over three times the amount of clay was used as compared with titanium, the sheet had a lower brightness and opacity. W. R. Willets stated<sup>(2)</sup> that one pound of titanium dioxide is equivalent to 16 pounds of clay in opacifying effects.

Relative effects of titanium dioxide and clay in paper.

	% Filler	% Opacity	% Brightness
None .....		83.0	82.8
Titanium Dioxide (anatase) 3.0		91.4	86.0
Titanium-barium composit (anatase) 6.5		90.0	86.1
Clay .....	10.0	88.4	84.3

Table I

Taken from Barksdale, J., "Titanium", page 389, The Ronald Press Company, 1949.

Titanium Dioxide (anatase)...	\$.21	~215 per pound
Clay.....	.0088	~0153 per pound
Talc.....	.0125	~015 per pound

Table II(3)

Price of fillers as given in the Paper Trade Journal, October 17, 1952.

### High Cost of Titanium Dioxide

The cost of titanium is considerably higher than that of other fillers. Table II on page 2 shows the cost of fillers per pound. A mill using titanium in their paper may use from between \$50,000 to \$100,000 worth per year. At this cost it would be well worth their while to invest in added laboratory equipment, if necessary, so fast routine control tests could be run. Even though a much smaller amount of titanium than clay can be used to give similar results, an accurate account should be made at all times to prevent the loss of this expensive material.

### PRESENT METHODS OF ANALYSIS

#### Volumetric Method(4)

TAPPI Standard Number T 439 m-44 gives a quantitative method for the determination of titanium in paper. This procedure involves first ashing the sample, then dissolving the titanium, removing any insoluble matter, reducing with the Jones Reductor and titrating with ferric ammonium sulfate.

The degree of accuracy depends upon proper operation of the Jones Reductor and upon titrating the reduced solution as quickly as possible thereby not allowing the titanium to be oxidized by oxygen from the air.

#### Colorimetric Method(5,6)

A colorimetric method as given in TAPPI Standards Number T 627 m-45 is by fusion of an ignited sample with potassium acid sulfate. It is then put into solution

with sulfuric acid. Potassium peroxydisulfate is added to oxidize any iron. Hydrogen peroxide is then added which converts the titanium to <sup>510</sup>tanic oxide which has a yellow color. This is then matched to a known solution.

A faster method is given by TAPPI Routine Control Method RC-42. This suggests first ashing the sample and then heating it for 30 minutes in a concentrated solution of sulfuric acid and ammonium sulfate. The solution is then cooled and diluted with water. Hydrogen peroxide is added and the color is compared with that produced by known amounts of titanium dioxide put through the same procedure. It is necessary to have approximately 9.0 grams of sulfuric acid per 100 ml. of solution.

#### MEANS OF ESTIMATING THE CONCENTRATION OF A SOLUTION BY ITS COLOR

##### Visual

The concentration of a colored solution can be determined by colorimetric analysis, according to Kolthoff and Sandell.<sup>(7)</sup> If the substance to be determined is colorless, it may often be transformed into a colored compound by some suitable chemical reaction. In this way smaller amounts of the substance being tested can be used or traces can be determined in a simple way. Kolthoff and Sandell<sup>(7)</sup> stated that ordinary colorimetric methods, using a Nessler Tube, do not yield an accuracy greater than about 2 per cent. Methodic error also enters in which may be small or /



large.

There are two ways in which to judge the concentration of a colored solution<sup>(7)</sup>: (1) The Standard Series Method and (2) The Balancing Method.

In the Standard Series Method a comparison is made in suitable glass vessels filled with unknown and standard solution to the same height and viewed vertically through the length of the column of liquid. The concentration of the unknown is equal to that of the standard having the same color intensity. As a rule it will be found that the color intensity of the unknown solution will lie between that of two successive standards. In such a case the concentration of the unknown can be approximated if the concentrations of the two standards do not differ too widely.

In the Balancing Method, the height of one or both columns is varied until the color intensities are the same when viewed through the lengths of the columns. Hehner cylinders or the Dubascq colorimeter is used in this method. When the instrument is perfect and Beer's law is obeyed, the following relation holds at equal color intensity.

$$R_x C_x = R_s C_s$$
$$C_x = \frac{R_s C_s}{R_x}$$

where  $R_s$  denotes the reading of the standard, and  $R_x$  that of the unknown at equal intensity.  $C_s$  is the concentration of the standard and  $C_x$  is the concentration of the unknown.

## Sources of Error

Eye fatigue is generally one of the most important factors responsible for irreproducible results. The operator should avoid looking at any bright light and should allow his eyes to rest between successive sets of readings. After a certain number of observations have been made, the eye becomes fatigued and is no longer sensitive to slight differences in intensity or shade.<sup>(7)</sup>

Presence of foreign constituents in the unknown may cause an increase or decrease in the intensity. Interfering ions are molybdenum, vanadium, iron and chromium which produce an orange color with hydrogen peroxide. Fluorides destroy the color and hence must be absent. Phosphoric acid and alkali sulfates also reduce the color intensity. Counteraction to this is accomplished by adding an excess of sulfuric acid. Color intensity is also increased or decreased depending upon the temperature. Therefore, the solution being tested must be at the same temperature as the known solution.<sup>(8)</sup>

Suspended material in the unknown will make it difficult to compare with a clear standard. When this occurs there is a scattering of light and the obedience to Beer's law is not observed, or it is observed only over a limiting range. The finer the particle size, the smaller the deviations. Thus any factor that may effect<sup>Sp</sup> the particle size must be carefully controlled, such as the concentration of the reactants, temperature, time of standing and presence of electrolytes.<sup>(9)</sup>

## A RAPID AND ACCURATE METHOD TO DETERMINE CONCENTRATION

### Photoelectric Colorimetry

A faster and possibly more accurate method than the visual determination of the concentration would be through the use of a photoelectric colorimeter. This would eliminate the personal factor and extend the analysis of a substance to the red and blue ends of the spectrum at which the sensitivity of the eye is extremely low. The precision attainable by routine photoelectric work is about the same (1 or 2 per cent error) as that in careful visual work. In especially favorable cases with attention paid to every source of error, it is possible to attain a precision of a few tenths of one per cent.<sup>(7)</sup>

### Theory of Colorimetry<sup>(7)</sup>

Colorimetry is based upon the laws of Lambert and Beer which states that when a beam of monochromatic light falls on a homogeneous layer of a substance, part of the light is reflected, part is absorbed and part is transmitted.

Lambert investigated the relation between the intensities and the thickness of the layer and stated that the intensity of the transmitted light decreases in geometric progression when the thickness of the layer increases in arithmetic progression. This is given by

$$I_t = I_0 e^{-kl}$$

where  $I_t$  equals the transmitted light,  $I_0$  equals the intensity of the original light,  $l$  is the depth of the layer and  $k$  is a proportionality factor.

Beer studied the influence of the concentration of the colored substance in the solution upon the light transmission or absorption. The same relation was found between transmission and concentration that Lambert found between transmission and thickness. This is given by

$$I_t = I_0 e^{-kc}$$

where  $c$  equals the concentration of the solution.

Combination of the two laws gives

$$I_t = I_0 e^{-klc}$$

### Choice of Concentration(7,9)

If the photo current is the measured quantity, the limiting factor is the uncertainty in the galvanometer reading,  $dG$ . If there is a steady light and a linear relation between intensity and galvanometer deflection, then

$$G = kI_t \text{ and } dG = k \cdot dI_t$$

Applying the Lambert and Beer law which was given above,

$$\ln \frac{I_0}{I_t} = E, \text{ the extinction point}$$

$$\text{then } E = \ln I_0 - \ln I_t = \ln I_0 - \ln I_0 e^{-E}.$$

The error of the determination is given by

$$\frac{dc}{c} = \frac{dE}{E} = - \frac{dI}{I \cdot e^{-E}}$$

which is a minimum when  $E \cdot e^{-E}$  is a maximum. This occurs when  $E = 1$  or  $\log \frac{I_t}{I_0} = 0.4343$  which corresponds to 36.8 per cent transmission.

Relative accuracy depends upon the particular instrument being used. Figure 1, below, shows the relative error in concentration determination as the per cent transmission varies. Also, errors will enter in the same as in visual determination if there are interfering ions present.

#### Preparing for Routine Tests (10)

In photoelectric estimation of concentrations of unknown solutions, the first step is to prepare a calibration curve, i.e., a plot of  $\log \frac{I_t}{I_0}$  vs. concentration. For this, several solutions of known concentration of the substance to be analyzed are prepared, their intensity ratios are read with the colorimeter and a graph is drawn. Then, when the intensity ratio of an unknown solution is measured, the concentration corresponding to this value is read off the curve. Figure 2, below, shows a typical  $\log I_t/I_0$  vs. concentration calibration plot. The straight line shows that the solution obeys Beer's law.

If the photoelectric colorimeter can be used for the determination of the concentration of titanium dioxide in a solution, it would provide a simple and fast control method, especially if the paper could be dissolved as is rather than ashing it first.

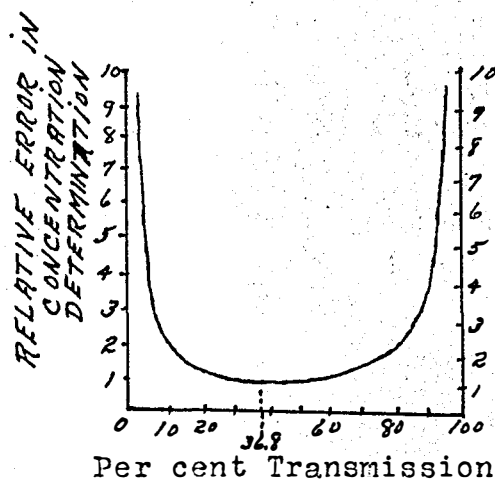


Fig. 1(7) Relative error in photo-electric photometry at different percentages transmission.

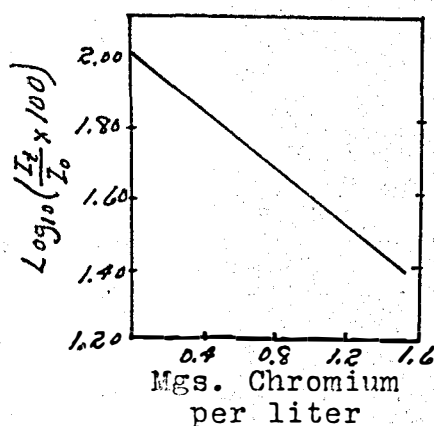


Fig. 2(10) Spectrophotometer calibration for chromium phenyl-carbazide method.

## APPARATUS

### The Jones Reductor

Amalgamated zinc is used for the reduction process. Care must be taken so as not to expose the reduced solution to air. Greatest accuracy is obtained when the solution being titrated is not shaken until the end point has been almost reached. Reduced solutions are unstable to air and hence must be protected from oxidation by the air after reduction has been done. The final end point is a light straw color. The percentage is then calculated. In the case of titanium, 1 ml. of .1N ferric alum equals .00801 grams titanium dioxide. For complete operation see TAPPI Standard Number T 439 m-44. Figure 3 on the following page shows a general description of the reductor.

### The Photoelectric Colorimeter

The photoelectric colorimeter is an instrument used for measuring absolute or relative light intensities. The solution being tested is placed either before or behind the slit of the monochromator and the current or voltage developed is measured. The photocell gives a response which is directly proportional to the light intensity levels used for the measurement. A very simplified diagram of the photoelectric circuit is given in Figure 4 on the following page.

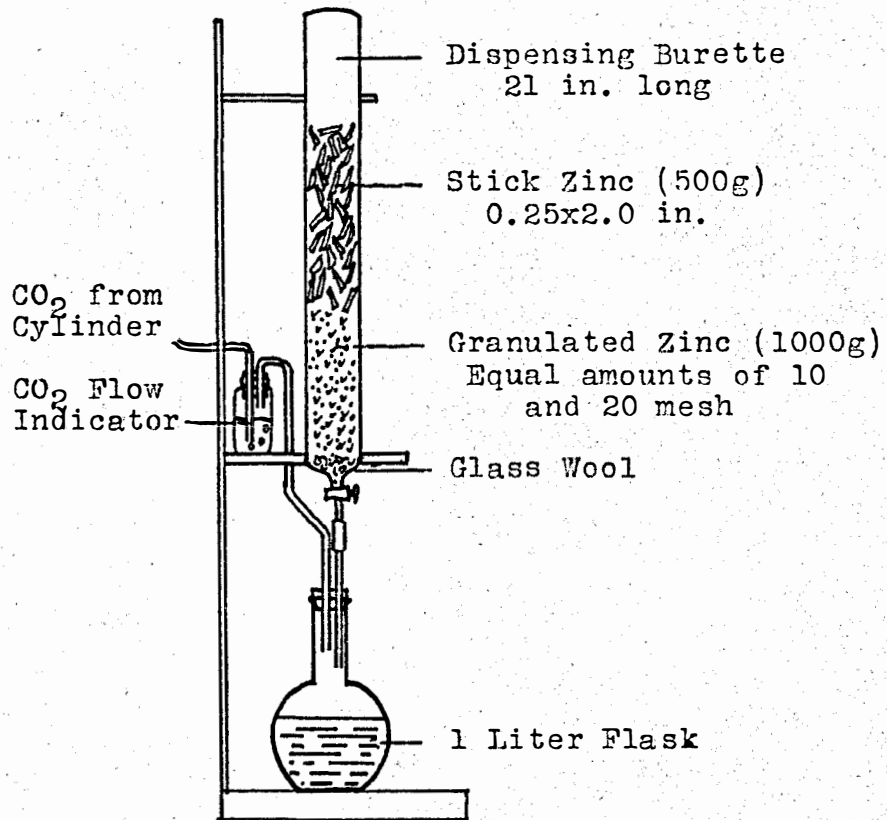


Fig. 3(4)

# THE JONES REDUCTOR

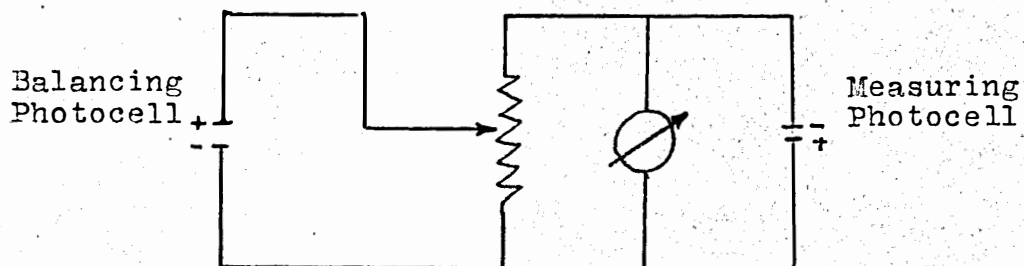


Fig. 4 Simplified diagram of the photoelectric circuit as given in the operating instructions for the Lumetron Photoelectric Colorimeter, page 859-2.



## LITERATURE CITED

- (1) Barksdale, J., "Titanium", first ed., pages 387-392, The Ronald Press Co., 1949.
- (2) Willets, W. R., Paper Trade J. 97, No. 10:28-30, (Sept. 7, 1933); World's Paper Trade Rev., 102, No. 9:642, 644, 682, 684, (Aug. 31, 1934).
- (3) Paper Trade J., 135, No. 16: , (Oct. 17, 1952)
- (4) TAPPI Standard Number T 439 m-44.
- (5) TAPPI Standard Number T 627 m-45.
- (6) TAPPI Routine Control Method RC-42.
- (7) Kolthoff, I.M. and Sandell, E.B., "Textbook of Quantitative Inorganic Analysis", Revised ed., pages 645-668, New York, N.Y., MacMillan Co, 1947.
- (8) Scott, W.W., "Scotts Standard Methods of Analysis", Furman, N.H. editor, 7th ed., pages 987-988, Lancaster Press, Inc., 1939.
- (9) Kirk, P.L., "Quantitative Ultramicroanalysis", First ed., pages 70-71, New York, N.Y., John Wiley & Sons, Inc., 1950.
- (10) Prutton, F.C., and Maron, S.H., "Fundamental Principles of Physical Chemistry", Revised ed., page 758, New York, N.Y., The MacMillan Co., 1951.
- (11) Operating Instructions for the Lumetron Photoelectric Colorimeter, page 859-2, Photovolt Corp., New York, N.Y.

## Outline of Laboratory Experimentation

The laboratory work in connection with this thesis will consist of first comparing results obtained by analyzing solutions of known concentrations of titanium dioxide using the three TAPPI methods: T 439 m-44, T 627 m-45 and RC-42. The temperature and concentration shall be varied in the colorimetric determinations to see if there are any deviations due to these effects.

The method I propose shall employ the use of a suitable oxidizing agent, so ashing will not be necessary, and a solvent for the titanium. These should be such as not to interfere with the color reaction given by hydrogen peroxide and titanium. The concentration shall be evaluated through the use of the photoelectric colorimeter.

### I Comparison of standard tests

A Solutions of known concentrations shall be analyzed

- 1 TAPPI T 439 m-44
- 2 TAPPI T 627 m-45
- 3 TAPPI RC-42

B The above colorimetric determinations shall be conducted varying the temperature and concentration

C The amount of time for each determination shall be recorded

D Deviations from the actual concentration shall be calculated and plotted

## II Proposed method which will eliminate ashing

A Oxidizing agents for carbon

1 Hydrogen peroxide

2 Nitric acid

B Solvent for titanium

1 Concentrated sulfuric acid

C Hydrogen peroxide added to produce color reaction

1 Color intensity evaluated with a photo-electric colorimeter

## III Comparison of methods

A Accuracy

B Time required

## LABORATORY RESEARCH

The objective of this study was to find a method of analysis which can be used for control purposes. The method must be fast and have a reasonable degree of precision so the answers can be obtained while the paper is still on the machine, and so changes can be made before an appreciable loss has occurred. This method was to be compared with the standard methods of analysis in regard to the time required. The degree of precision was also to be determined.

### Results Using The TAPPI Volumetric Method

To check the degree of precision obtainable by the Jones Reductor method and to estimate the time required, a series of 13 tests were run using aliquot samples from the same  $\text{TiO}_2$  solution. The following two pages show data and a graph of the results. On the average, the error was .57% based on the amount of  $\text{TiO}_2$  per sample. The maximum error was -1.08% and the minimum error was +.08%. The errors indicated may have been caused by one or more of several sources: Incomplete reduction in the Jones Reductor, incomplete washing of the sample from the reductor, oxidation of the titanium by the air while titrating, inaccurate burette readings, inaccurate end point and inaccurate determination of the normality of the

DATA FROM ESTIMATIONS FOLLOWING  
TAPPI T627 m-45  
(Volumetric)

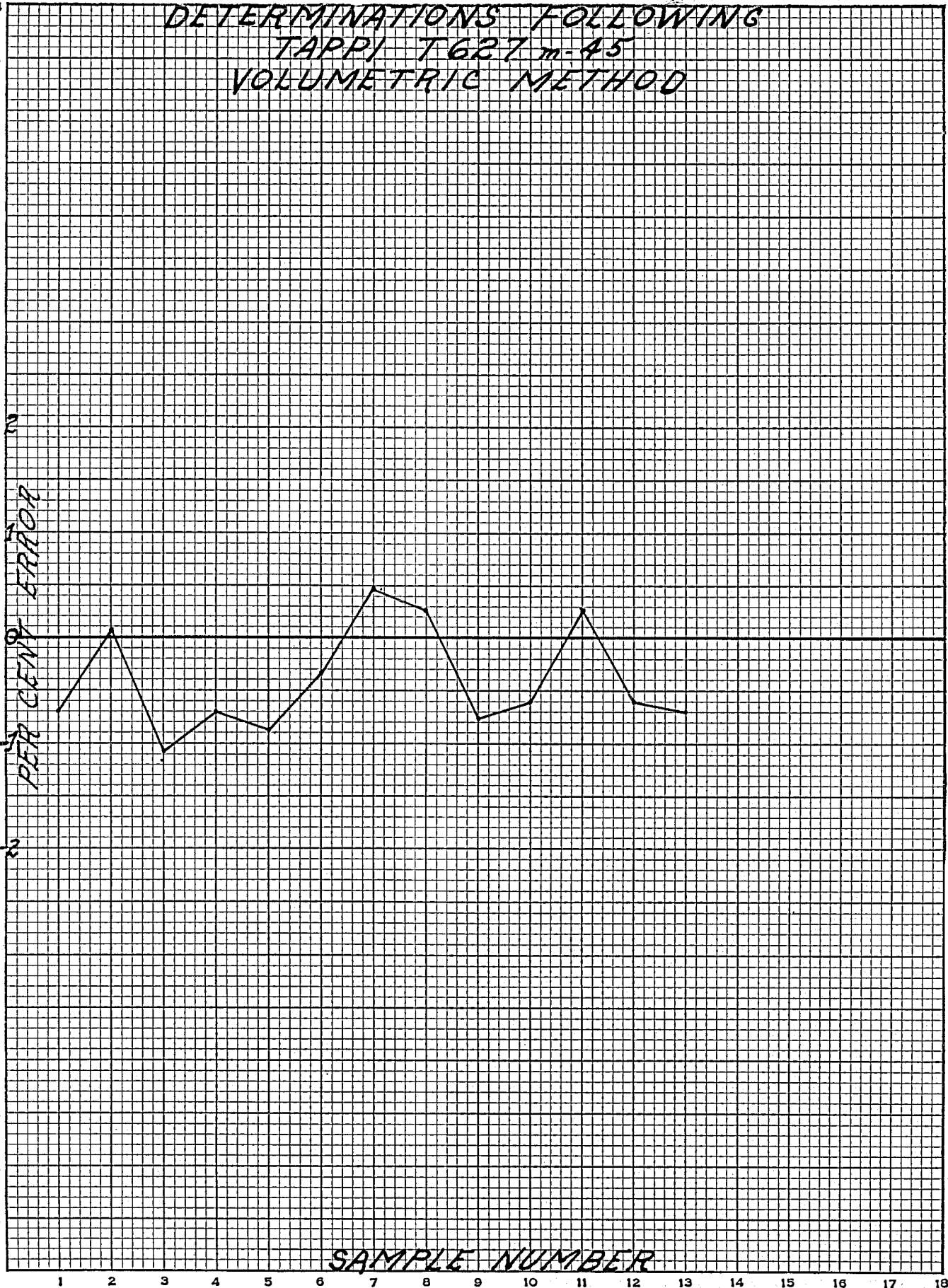
All Samples Contained .2500 g  $\text{TiO}_2$

Sample	Weight as determined	Per cent error
1	.2483	- .68
2	.2502	+ .08
3	.2473	-1.08
4	.2483	- .68
5	.2478	- .88
6	.2492	- .32
7	.2512	+ .48
8	.2507	+ .28
9	.2481	- .76
10	.2486	- .56
11	.2507	+ .28
12	.2485	- .60
13	.2483	- .68

Table III

DETERMINATIONS FOLLOWING  
TAPPI T627 m-45  
VOLUMETRIC METHOD

PER CENT ERROR



ferric ammonium sulphate. While it is recognized that an operator with long experience may obtain greater precision, it is also true that paper testers in mills probably do not have the experience to obtain these levels of accuracy.

The time required for this determination is about 2 hours in addition to the time necessary for ashing a sufficiently large sample. From this standpoint, the method is not adaptable for control purposes.

A Brief Account of the TAPPI Colorimetric Method and The Routine Control Method

The TAPPI T627 m-45 colorimetric method involves ashing the sample, fusing it with potassium acid sulphate, dissolving it in sulphuric acid, developing a color with hydrogen peroxide and comparing the intensity of the color with that of a known solution put through the same procedure. This method requires about one hour to run and may encounter personal error unless careful comparison of the color intensities is made. All insoluble material must be filtered out or a comparison with a clear solution will be difficult. This method, also, requires too much time to be used for a control check.

The TAPPI RC-42 method is similar to the T627 m-45 colorimetric analysis with the exception that smaller amounts are used in order to reduce the time required and instead of first fusing the ashed sample and then dissolving it, <sup>it is done in one step using</sup> ~~it is~~ a solution of ammonium sulphate and sulphuric acid.

This step alone requires 30 minutes which is more time than any complete control test should take. The precision again, depends upon the comparison of the color intensity with that of a known solution using the same procedure and the removal of all suspended material.

#### QUANTITATIVE DETERMINATION WITHOUT ASHING THE PAPER

This method eliminates the step of ashing the sample. It includes the use of an oxidizing agent which completely destroys the paper while the titanium is being dissolved, and was suggested by Mr. R.T. Elias, instructor in the Pulp and Paper Department at Western Michigan College. In this method smaller samples are used than in the TAPPI Routine Control Method to shorten the time required, and to confer accuracy the photoelectric colorimeter is used.

#### Reagents:

- A. Hydrogen Peroxide Solution, 10%.
- B. Solution of Sulfuric Acid and Potassium Nitrate.  
Dissolve 30 grams of potassium nitrate in 100ml.  
of concentrated sulfuric acid.

#### Equipment:

Lumetron Photoelectric Colorimeter or its equivalent.

#### Procedures:

Weigh out accurately to four decimal places enough of the sample of paper to contain between 1.2 and 2.2 milligrams of titanium dioxide. This would be equivalent



to about a 35 mg. sample if the paper contained 5%  $\text{TiO}_2$  or a 55 mg. sample if the paper contained about 3%  $\text{TiO}_2$ . Place in a 125 ml. beaker and add 5 ml. of the solution of potassium nitrate in sulphuric acid. Heat slowly under a hood until the paper has disintegrated, and then raise to almost boiling for 5 minutes. Allow to cool until water can be introduced without spattering. (Add about 10 ml.) Transfer the contents of the beaker to a 50 ml. volumetric flask. Completely rinse contents of the beaker into the flask by using small portions of water. Add about 5ml. of the 10% hydrogen peroxide. Cool rapidly to about room temperature, dilute to exactly 50 ml. and obtain the percentage of transmission using the photo-electric colorimeter with a filter wave length of 440 millimicrons. If the solution contains any suspended material, it must first be filtered. The filter should not be washed. The concentration is then taken from the reference curve, and the percentage  $\text{TiO}_2$  in the sample is calculated. The concentration from the graph should be taken to three significant figures.

#### Reference Curve:

The reference curve is determined by analyzing solutions of titanium dioxide of known concentrations put through the above procedure. A plot is then drawn of the percentage transmission versus the concentration on semi-logarithmic paper. Data and a graph are shown on the following two pages.

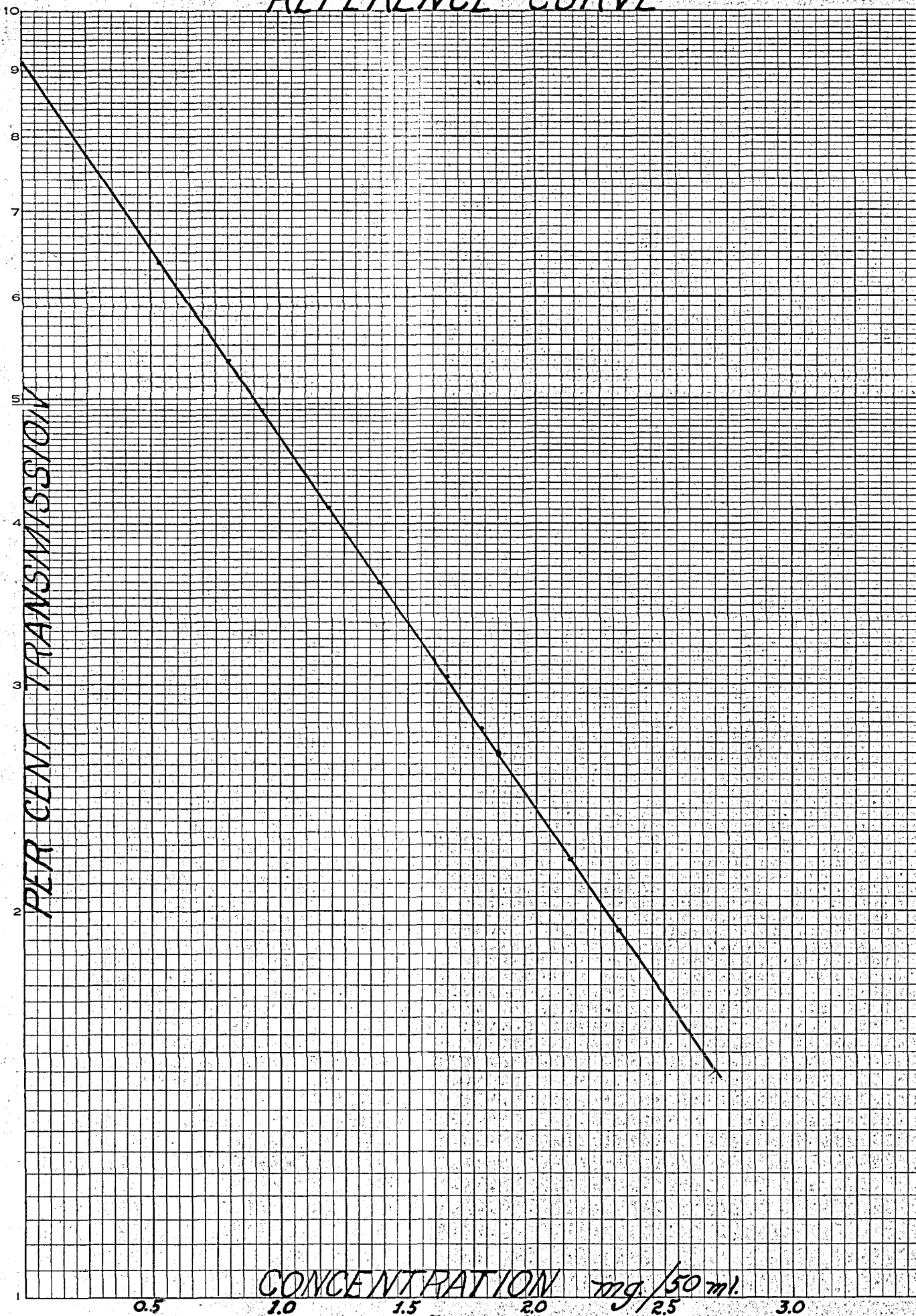
Evaluation of Solutions of Titanium Dioxide Using  
Known Concentrations to Determine the Relationship  
between Concentration and Percent Transmission

Solutions were analyzed with a Lumetron Photo-  
electric Colorimeter using a blue filter with a wave length  
of 440 millimicrons. All solutions were diluted to  
50 ml.

Sample	Weight $\text{TiO}_2$ (g)	%Transmission
1	.00000	91.20
2	.00053	63.90
3	.00080	53.20
4	.00093	49.00
5	.00119	41.10
6	.00139	36.00
7	.00165	30.40
8	.00178	27.20
9	.00185	26.50
10	.00213	21.90
11	.00232	19.30

Table IV

# REFERENCE CURVE



CONCENTRATION mg./50 ml.

To eliminate much error in weighing excessively small amounts of titanium dioxide, a larger portion is weighed and dissolved. Aliquot samples are then taken through the range of .5 to 2.5 milligrams per 50 ml.

#### Precision of The Determination:

A series of 17 tests were run to check the degree of precision obtainable using this method. Table V and Figure 7 show the results. Determinations were made on Plover Bond Opaque Paper using approximately 60 mg. samples. The maximum percentage  $TiO_2$  was 3.76, <sup>and the minimum was 3.63.</sup> Taking the average, 3.67, as the true percentage of  $TiO_2$  in the paper, the maximum per cent error in the determinations was 2.46, based on the amount of  $TiO_2$  present in the sample.

The precision of the determination depends upon several factors: Precision in weighing, precision in diluting the sample, absence of foreign material which will cause a deviation of the color intensity, adjustment of the temperature, a true reference curve and a steady light source in determining the percentage transmission.

Deviations in the temperature from sample to sample will cause a change in the volume such that the dilution will vary and consequently the intensity of the color will be altered. Therefore, all samples, including those for the determination of the reference curve must be cooled to approximately the same temperature.

Data from Determinations of Titanium Dioxide  
in Paper without Ashing the Sample.

Solutions were analyzed with a Lumetron Photo-  
electric Colorimeter using a blue filter with a wave  
length of 440 millimicrons. All solutions were diluted  
to 50 ml.

Sample	Weight(g)	%Transmission	Weight TiO <sub>2</sub> per 50 ml.	%TiO <sub>2</sub>
1	.0579	21.90	.00212	3.68
2	.0581	21.75	.00214	3.68
3	.0595	21.40	.00217	3.64
4	.0587	22.00	.00213	3.63
5	.0572	22.50	.00209	3.65
6	.0592	21.70	.00215	3.63
7	.0589	21.75	.00214	3.63
8	.0563	23.20	.00205	3.64
9	.0566	23.10	.00206	3.64
10	.0578	22.40	.00210	3.63
11	.0618	19.95	.00227	3.67
12	.0573	21.80	.00214	3.73
13	.0585	21.30	.00217	3.71
14	.0593	21.20	.00218	3.68
15	.0601	20.50	.00223	3.71
16	.0574	21.80	.00213	3.71
17	.0596	21.35	.00448	3.76

Table V

ESTIMATION OF  $TiO_2$  CONTENT  
WITHOUT ASHING THE SAMPLE

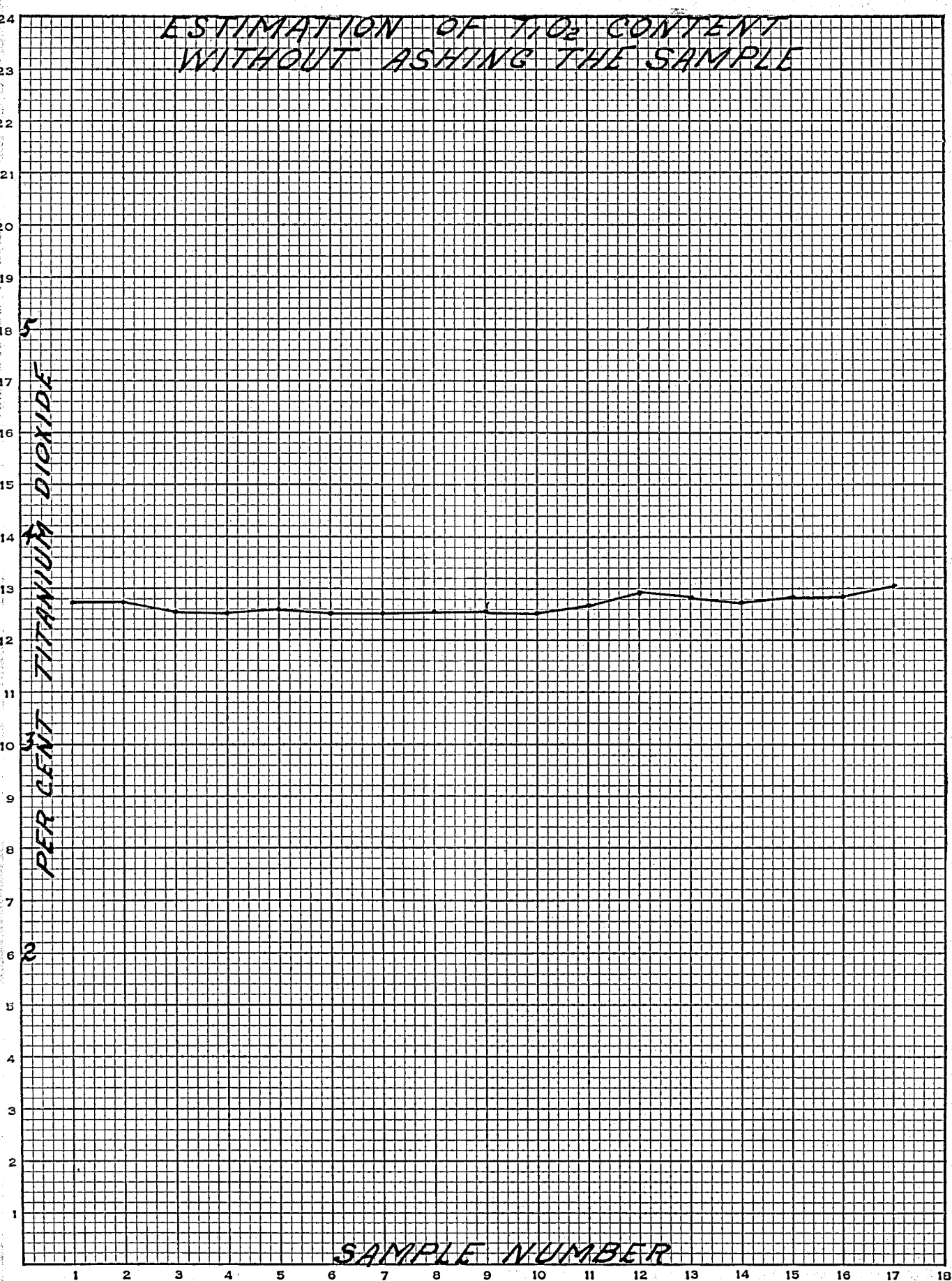


Figure 7  
-26-

### Time Required:

The amount of time required for a determination can be brokenf down into the following steps:

Weighing of the sample	2 minutes
Dissolving the $TiO_2$	7 "
Diluting and Cooling	5 "
Filtering (if necessary)	5 "
Evaluating	<u>3</u> "
Total	22 "

As can be seen from these figures, the amount of time has been greatly reduced in comparison with the standard methods. This has been accomplished through the use of small samples. Ashing a large sample is not necessary as it is in the TAPPI procedure; an oxidizing agent will quickly disintegrate a smaller sample. The use of small volumes reduces the amount of time necessary to heat the solution to the boiling point and to cool again to room temperature. Cooling can be accomplished very rapidly by using a cold water bath. Another means of saving time is in filtering. It is necessary to filter only enough to fill the cubette in the colorimeter. After the percentage transmission has been determined, the concentration of the solution can be quickly read from the reference curve and the per cent  $TiO_2$  in the paper calculated. Several determinations can be run simultaneously with a small amount of time necessary in excess of what it takes for a single sample.

Summary:

This method of analysis has two distinct features: It is fast and has a reasonable degree of precision. The use of small samples makes it possible to use an oxidizing agent such as potassium nitrate to destroy the cellulose, and the colorimeter makes it possible to make an accurate estimation of the concentration of the  $TiO_2$  in the solution. If a rapid method such as this were used for a control test, much loss due to the under addition or over addition of titanium dioxide could be prevented.

*Respectfully submitted,  
Allen E. Vahl*