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PIGMENT COLOR RESEARCH REPORT

FINAL REPORT

DETERMINATION OF CPC PURITY. AN EVALUATION OF THE RESULTS OF TWO INDEPENDENT METHODS

MARCH 1952 to JUNE 1952.

SUBMITTED BY: A. R. HANKE A R Hanke DATE SUBMITTED: JULY 7, 1952

APPROVED BY: B. H. PERKINS B H Perkins 7/29/52 DATE ISSUED:

I - INTRODUCTION:

Preliminary trials on attempting to correlate % CPC by a spectrophotometric method with percent total copper have shown that even with finished CPC, the correlation is poor. With crude CPC the correlation is understandably poor because of the variable amount of ionic copper present. With finished CPC it is less evident why better correlations cannot be had. One explanation is that the CPC pigment contains copper that in some fashion is organically bound but not bound as CPC and that this type of copper is present in variable amounts so that a copper analysis will not give a true indication of the CPC content.

It was considered of interest to examine a method which first acid pastes the pigment then floods it into water and then filters off the soluble material so produced before determining copper on the CPC residue. This report describes the preliminary work in the development of this method and also compares the results of this method with the results of the spectrophotometric method.

II - CONCLUSIONS:

Two methods of analysis, a spectrophotometric method, and a copper analysis method, give results that are acceptable for most purposes. A statistical treatment of the data derived from the two methods shown that the expected agreement between them is of the order of magnitude of 1 to 2% relative.

III - ESTIMATION OF PURITY BY % Cu ON ACID PASTED CPC

As a prelimimary step to the development of this method, it was considered desirable to see what successive acid pastings would show for both the recovery and the amount of ionic copper extracted. A sample of LB/CPC crude, lot 9032, was acid pasted and flooded into water, filtered and the residue dried and weighed. The filtrate was analyzed for copper. This operation was carried out for a total of six times.

There was a continued decrease in residue weight and some ionic copper was always found in the filtrate although the amount became less with each acid pasting. The technique of acid pasting was not under very good control, hence some acid pastings may have been carried out more efficiently than others, but in spite of this, some significance can be attached to the observation that the rate of change of recovered pigment and percent extracted copper decreased with respect to the number of acid pastings.

TABLE I

No. of Acid Pastings	% Recovery on original Wt. basis.	Cumulative % weight lost	Cumulative % Cu in filtrate on original Wt. basis
0	100.00	0	-
1	82.24	17.76	0.650
2	81.70	18.30	0.676
3	81.48	18.52	0.697
4	81.21	18.79	0.703
5	81.18	18.82	0.707
6	80.90	19.10	0.709

This is illustrated in Figure I and Table I. Table I shows the cumulative percentage loss in weight for each acid pasting as well as the cumulative percent ionic copper found in the filtrate after each acid pasting. Figure I is simply a plot of these data. It is to be observed that although a single acid pasting removes the bulk of the non-CPC copper, even after the sixth acid pasting there is enough copper found in the filtrate (0.002%) to represent the upper limit specified for rubber colors. If the copper found, as a result of these successive acid pastings, is largely due to CPC decomposition, it is surprising that the cumulative percent copper found in the filtrate continues to fall off and doesn't approach a constant slope even after six acid pastings. Then again it has been observed that samples of LB-CPC can be kept dissolved in sulfuric acid for at least 24 hours at 50°C with no loss in optical density at the wave length of maximum optical density 790 mμ. Temperature rises during acid pasting and flooding never amounted to more than a rise from 21°C to 34°C. These are admittedly only thermometer readings and it is conceivable that local temperatures in excess of this may exist for very short periods of time.

The above observations do not conclusively rule out CPC decomposition as an explanation for the continued presence of copper in the acid pasted - flooded filtrate nor do they conclusively prove that there is decomposition. The possibility must still be recognized that acid pasting may be very inefficiently converting a type of organically bound copper that is not CPC copper into ionic copper.

At any rate, the data strongly indicate that if decomposition of CPC is occurring, it amounts to something in the order of 0.002% Cu per acid pasting. This is the low point reached for the slope of the percent Cu curve of Figure 1. Since 10% Cu is roughly equivalent to 100% CPC, this amounts to only 0.02% CPC per acid pasting. From the point of view of CPC purity, this suggested loss of 0.02% CPC can be neglected. The data further indicate that one acid pasting of a finished CPC is sufficient

to remove enough non-CPC copper so that continued acid pastings will not remove enough additional non-CPC copper to make it worthwhile. From Table I it can be seen that if the first acid pasted product is considered equivalent to finished CPC, and the second acid pasted product is considered equivalent to the acid pasting of a finished pigment, the cumulative percent Cu of four additional acid pastings amounts to only 0.033% Cu. This is equivalent to about 0.3% CPC. This then is the order of magnitude of the systematic error that can exist if finished pigments are given only one acid pasting before copper analysis. This, of course, assumes that the ionic copper found in the filtrates from the acid pasting-flooding steps is due entirely to non-CPC copper and is not due in part to some CPC decomposition. With more efficient acid pasting, the systematic error will be reduced to a still lower figure and it is felt that the acid-pasting technique that was used for this experiment could be improved upon from an analytical point of view by more thoroughly working the pigment particles into the sulfuric acid. In subsequent analyses this was done.

The data show one other thing. The acid-pasting technique cannot be used to determine "ionic copper" in a CPC as we at present understand the term "ionic copper". The amount of copper found in the filtrate even after the sixth acid pasting (0.002%) is admittedly too small to greatly affect the percent purity based on a copper analysis but it is too large when considered in terms of what is obtained in a direct extraction method for ionic copper or what is tolerated by the rubber specification. It may be that the ionic copper found in the aqueous filtrate after acid pasting can have some significance in connection with the use of CPC in rubber or with the problem of CPC purity but at present one must hesitate in calling it "ionic copper". It may have originally been organically bound but released by the acid pasting step.

From an analytical point of view, the practical result of this work is to indicate that at least one acid pasting is necessary before a copper analysis yields a result that can be validly converted to a percent CPC. The analytical method for achieving this is given in detail in Appendix I. The conversion factor in use for converting percent copper into percent CPC is 9.44. This is based on a molecular weight of 599.9 for the LB type CPC.

IV - CPC PURITY BY SPECTROPHOTOMETRIC METHOD

The copper phthalocyanine molecule has a very strong light absorption band in the near infra-red. The position of this band seems to depend to some extent on the type of CPC as far as chlorine content is concerned and for the LB type used for this work, the absorption maximum came at 790 mμ.

To use this method, it is necessary to have a standard of reference that represents 100% CPC or one that represents a CPC of known purity. For this work a specially acid pasted sample was used coded 1226-7B and analyzed for total copper. Assuming a molecular weight of 599.9 for the CPC which gives a conversion factor, % Cu to % CPC of 9.44, gives an analysis of 99.03% for the sample. Using this conversion factor to enable a calculation of the extinction coefficient to a 100% CPC basis will put both the CPC purity by % Cu and the spectrophotometric method on a directly comparable basis. Optical density measurements of H_2SO_4 solutions of this sample calculates to an extinction coefficient on a 100% CPC basis of 373. The unit light path is one centimeter and concentration is expressed as grams per liter.

In carrying out a determination using the spectrophotometric method, it is necessary to work with a very dilute solution (approx. 0.0074 g/l) because the light absorption band is so strong. This means that small sample weights are involved which must be very carefully measured. Dilutions must also be very carefully made. In the early work of this method, weight burettes were used to make the dilutions. This did not prove satisfactory and the present technique is to make the dilution with a 5 ml pipette and a 250 ml volumetric flask. Appendix II gives the method in detail.

V - A CROSS COMPARISON OF THE ANALYTICAL RESULTS OF THE TWO METHODS.

a) Percent Copper on Acid Pasted CPC (Appendix I)
b) Percent Copper by Optical Density of H_2SO_4 Sol'n. (Appendix II)

Six samples of CPC were analyzed by both methods. The code and description of the samples is given below:

BT-304-D, SD-90719 - Standard LB type CPC

1424-16D, SD-90719 - "HF" ground.

$1424\text{-}16D_1$ - Pigment extracted from the borax sludge of above 16-D sample.

CPC/LB Crude, lot 439.

1424-16A - An "HF" grind of the above crude lot 439.

$1424\text{-}16A_1$ - Pigment extracted from the borax sludge of the above 16-A sample.

The samples were analyzed in duplicate and the results of both methods are tabulated in Table 2.

TABLE II

Sample	Y % CPC by % Cu on Acid Pasted Residue	X % CPC by Spectrophotometric method	Y/X
BT-340-D, SD-90719	94.49	94.64	0.9984
LB/CPC Crude, lot 439	88.45	89.28	0.9907
1424-16-D	88.17	88.74	0.9936
1424-16-A	87.04	87.13	0.9990
$1424\text{-}16\text{-}D_1$	85.62	84.99	1.0074
$1424\text{-}16\text{-}A_1$	82.69	83.11	0.9949

To observe the correlation in the data of Table II, the results by % Cu are plotted as ordinate and the results by the spectrophotometric method are plotted as abscissa in Figure 2. This figure also shows the line of best fit drawn through these points that would also pass through the origin. The slope "m" for this line is calculated from the equation $m = \frac{\Sigma Yi}{\Sigma Xi}$ and comes to 0.9973 which makes the equation for the line $Y = 0.9973\ X$. Since the line passes through the origin, it is possible to calculate individual estimates of the value for the slope by dividing "Y" by "X" for each pair of values. This is shown in the last column of Table II. These values can then be treated statistically. The first question that can be asked is, "Is the calculated slope of 0.9973 far enough away from 1.000 to be significant?". This is the same as asking, "Do the two methods give the same answer?". To attempt an answer to this question, first a standard deviation "s" is calculated from the formula -

$$s = \frac{\Sigma m_1 \left\{\frac{\Sigma m_1}{n}\right\}^2}{n - 1}$$

Where "s" = estimate of the standard deviation of a single estimate of the slope Y/X.

"m_1" = each individual estimate of the slope Y/X.

"n" = number of such estimates.

Applying the formula to these data yields a standard deviation "s" of 0.00582. Since there are six (n = 6) estimates of the slope Y/X, the standard deviation of the average slope is -

$$s_{ave.} = \frac{0.00582}{\sqrt{6}} = 0.002373$$

Applying the "t" tables, one obtains the "t" value of 2.571 for 5 degrees of freedom (n-1) at a probability level of 5%.

Confidence limit = 2.57 x 0.002373 = 0.0061.

The significance of the value 0.0061 is that the average slope must deviate from 1.0000 by an amount equal to or greater than 0.0061 before we can say that the deviation is significant. The 5% probability level implies that in making the foregoing statement, we would be wrong only five times out of every 100. Since 0.0061 is larger than the observed differences of 0.0027, we are not allowed to say that the deviation of the calculated slope from unity is significant. In other words, if there is, in truth, a difference between the two methods, our statistical data are not extensive enough to detect it. Since 0.0061 represents a deviation from the average slope of about 0.6% relative, we can say that even if more extensive data would show a difference between the two methods, this difference can be no greater than the order of magnitude of 0.6% relative and is probably less.

Now that it has been established that the two methods give very nearly the same result, another question that can be asked is, "What sort of variation can be expected between an estimate of CPC purity by % Cu and an estimate of CPC purity by the spectrophotometric method". To answer this question, the standard deviation of a single estimate of the slope is used rather than the average slope. The standard deviation of such a single estimate is s = 0.00582. Using the same "t" value as before, namely 2.571, gives a confidence limit of 2.571 x 0.00582 = 0.015. This means that the ratio of the results by the two methods of analysis should be no further away from unity than 0.015, which means about 1.5% relative. The true limiting differences may actually be less than this but because of the limited amount of data (5 degrees of freedom) we cannot say how much less but we can say that it won't be more. The significance of the 5% probability point again is simply that we will be wrong only five times out of every hundred such decisions we make.

If the importance of the decision is such that we feel we must be right more often than this, let us say 99 times out of every 100 such decisions, we would use the 1% probability point which the "t" tables gives as 4.032.

Confidence limit = 4.032 x 0.00582 = 0.0235.

This means that to play it safe and be wrong only once in one hundred times, our limiting difference must be set up to 2.5%. The true difference is still the same, of course, but we have to set the upper limit up to 2.4% in order to make the statement that the present difference between determinations using the two methods will be <u>no greater</u> than 2.4% relative.

This statistical analysis can be roughly summed up by saying that the determination by the two methods will compare to within 1% to 2% relative. This is quite acceptable from a determination point of view when one bears in mind that the comparison estimate is weighted by the fact that we only have a limited amount of data to treat statistically.

VI - PATENT SITUATION :

Nothing of a patentable nature disclosed.

VII - REFERENCES:

N.B. 1355-7
N.B. 1410-22
N.B. 1427-10, 61, 62
N.B. 1397-4, 9

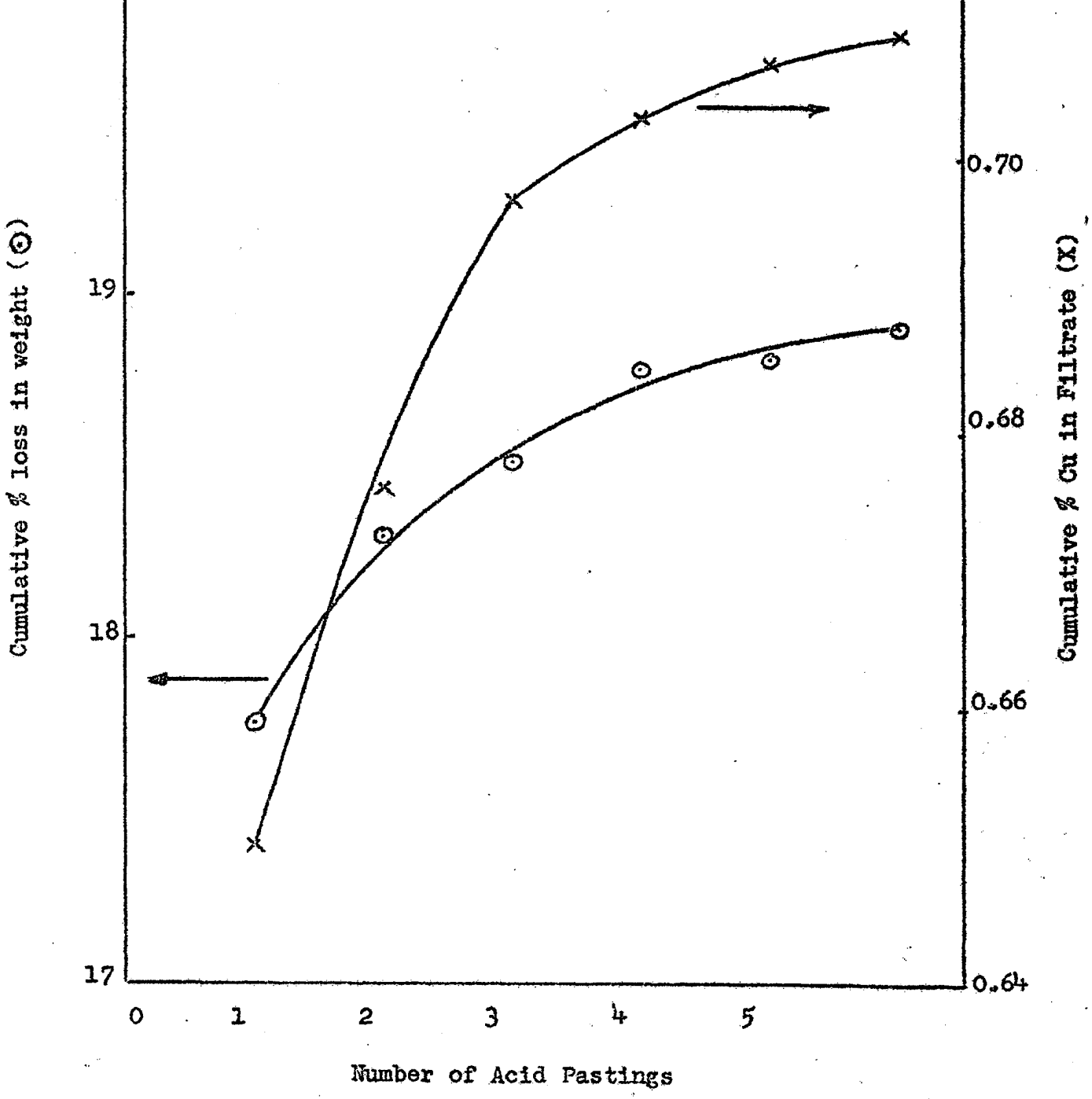
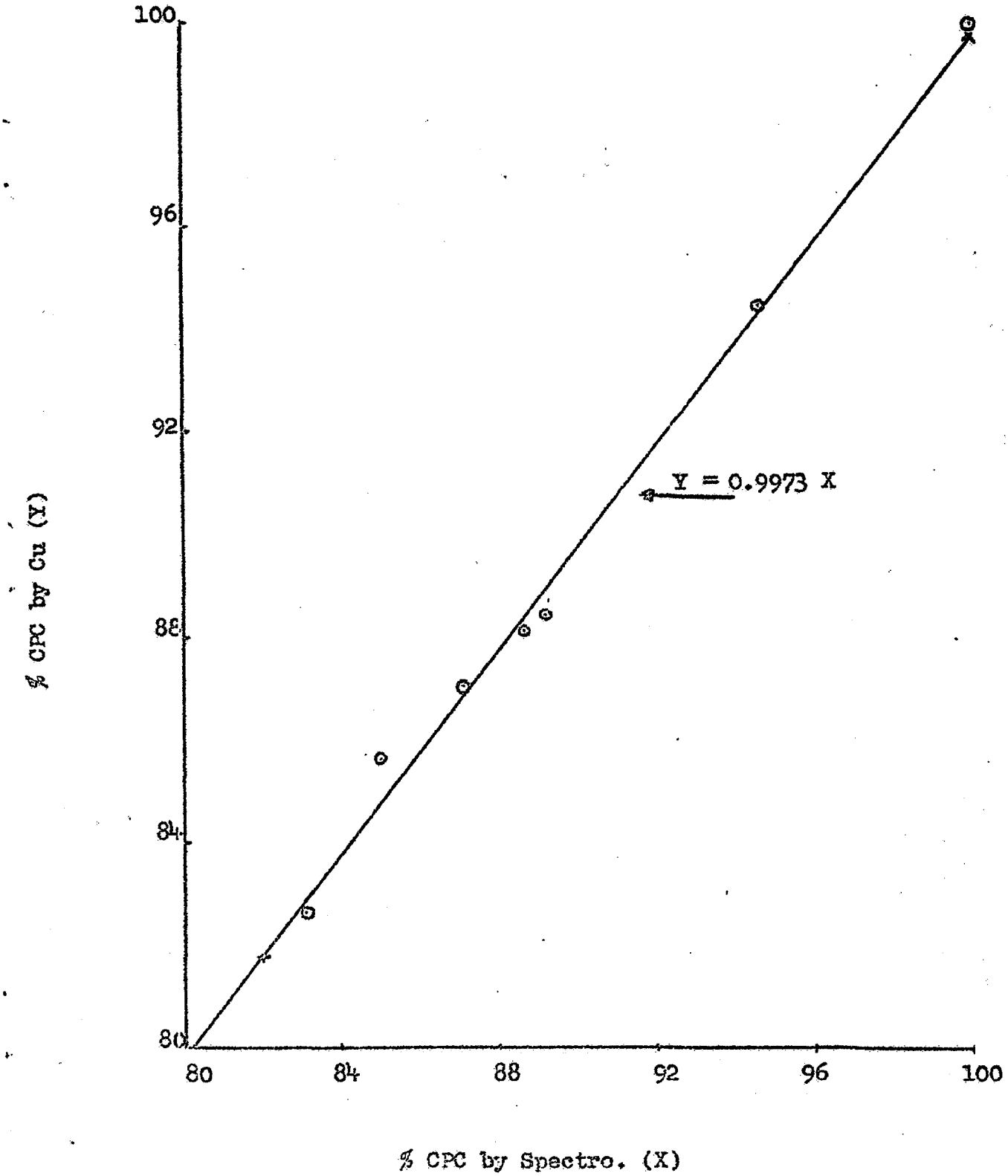


Figure 2. Correlation of Methods



APPENDIX I

I. SUBJECT: Copper Phthalocyanine (Blue)

II. DETERMINATION: Percent CPC by Copper Analysis.

III. REFERENCE:
KCR #159
N.B. 1427-10, 61 & 62

IV. PRINCIPLE:
Non CPC Copper is removed from the sample by acid pasting leaving the organically bound copper behind in the residue. The copper remaining is defined as CPC Copper. This copper is analysed using the standard iodometric method KCR #159.

V. STATUS:
This method was developed to give an evaluation of CPC content by copper analysis. This method compared favorably with CPC content by a spectrophotometric determination. Over fifteen samples have been run with consistently good results.

It is recognized that after acid pasting the sample may still contain some ionic copper and also some organically bound copper not of a CPC nature. It is also recognized that there may be a small amount of CPC decomposition. However, it is felt that the magnitude of the errors so introduced is low enough as to not seriously effect the accuracy of the method.

VI. REAGENTS:

Sulfuric Acid	conc.	C.P.
Fuming Nitric Acid		C.P.
Perchloric Acid 70-72%		C.P.
Hydrochloric Acid	conc.	C.P.
Acetic Acid (glacial)		C.P.
Ammonium Hydroxide		C.P.
Sodium Fluoride		C.P.
Potassium Iodide		C.P.
Ammonium or Potassium Thiocyanate		C.P.
Sodium Thiosulfate, standard solution	±	0.1N
Starch solution: 1% (freshly prepared)		
Potassium Dichromate		C.P.

VII. SPECIAL EQUIPMENT:
Pyrex glassware, Inter-Joint T 24/40 (may be obtained from Scientific Glass Co. of Bloomfield, New Jersey as listed below in their catalog #42).

#11262 Male connection, medium length, outside tube diameter 12 mm.

#8196 Distilling Head

#9136 Flat bottom flask (250 ml.)

VIII. PROCEDURE:

Place a one gram (1.0000) sample in a dry 50 ml. beaker. Add ±5 ml. of concentrated sulfuric acid and acid paste the sample thoroughly. (Note 1). If the sample contains considerable solvent soluble material such as a rosinate it may be desirable to remove this in the acid pasting flooding stage. (Note 2). Bring the sample to room temperature and flood into ±200 ml. of water at room temperature or below. Mix thoroughly and filter through a #40 Whatman paper. Wash with dilute (1%) sulfuric acid. Drain the sample somewhat and transfer sample and filter paper to a 250 ml. inter-joint flat bottom flask. (Note 3). Add ±4 ml. sulfuric acid (conc.), 20 ml. fuming nitric acid, and 3 ml. perchloric acid (70-72%). With distilling head in place heat to copious fumes of SO_3. Allow to cool and then wash down both head and flask with water. Add a few ml. of conc. hydrochloric acid and with head in place take volume down to almost dryness. Allow to cool and wash down head and flask with water, final volume ±40 ml. Heat to boiling with the head replaced by an inter-joint male connection acting as an air condenser and splash saver. Allow to evaporate till volume is ±20 ml. Cool and add concentrated ammonium hydroxide to just alkaline. Add 6 ml. of glacial acetic acid. Cool and add 1 gram of sodium fluoride and stir until most of it has dissolved. Disregard any small amount that may remain out of solution. Add 0.8 g. of potassium iodide previously dissolved in a little water. Titrate the freed iodine immediately with the standard solution of sodium thiosulfate. (Prepared and standardized as per KCR #159). Near the end point add 1 ml. of the freshly prepared starch solution and 0.8 g. of ammonium or potassium thiocyanate. Continue the titration to the disappearance of the blue starch-iodine color.

IX. CALCULATION:

$$\frac{\text{ml. of } Na_2S_2O_3 \times \text{Normality} \times 0.06357 \times 100}{\text{Sample Weight}} = \% \text{ Cu}$$

% Cu X 9.44* = % LB/CPC by copper analysis.

*9.44 is the current constant accepted for the LB type CPC of M.W.=600.0

% Cu X *9.065 = % Cl free CPC

*9.065 is the current constant accepted for the chlorine free type CPC M.W. = 576.

NOTE 1. Care must be taken at this point to insure a smooth uniform paste. Use of a straight flat-end stirring rod, will facilitate attainment of this condition.

NOTE 2. Acid paste as usual and then flood into a solvent mixture composed of 1:2:3:4 Benzene, Glacial Acetic Acid, Water, and Alcohol (23-A found suitable). Wash with the mixture and after draining well proceed as in determination beginning with the transfer of sample and filter paper to a 250 ml. inter-joint flat bottom flask. A BP-173-D sample run qualitatively by this procedure showed good dispersion and filterability.

NOTE 3. The transfer is facilitated by wrapping the filter paper in onion skin paper and adding it all to the flask. The sulfuric acid rapidly oxidizes the paper.

APPENDIX II

I. Subject: Copper Phthalocyanine

II. Determination: CPC Content by spectrophotometric method.

III. References: NB 1397-4, 1397-9

IV. Principle: CPC content is determined spectrophotometrically in sulfuric acid solution. CPC green has strong absorption peaks near 815 and 855 mμ, and CPC blues have peaks near 700 and 790 mμ. The extinction coefficient at the wavelength of maximum absorption is compared with that of a known standard.

V. Status: This method was developed because of the need for a reliable method for the determination of CPC content in toner and lakes. The method has been used successfully on five samples of polychlor CPC and nine samples of CPC blue toners and lakes to date, with good results. For this work, the Beckman Model DU spectrophotometer was used. The method is not suitable if oxidizing agents or substances yielding oxidizing agents such as nitrocellulose are present.

VI. Special Equipment: Spectrophotometer equipped with one centimeter light path cells and operable within the wavelength range of 650 mμ to 900 mμ.

VII. Procedure: Somewhat different procedures are used for CPC blue and green, so they will be described separately.

A. Procedure for Polychlor CPC: Transfer 0.0500 g of finely powdered pigment into a 150 ml beaker. (Note 1.) Add a few drops of conc. sulfuric acid to wet the sample, and disperse well with a stirring rod. Gradually stir in more sulfuric acid to a volume of about 60-70 ml. Carefully transfer to a 250 ml volumetric flask. Wash out beaker with additional portions of sulfuric acid until flask is filled to the mark. (Note 2.) Place the flask in a steam bath with the bottom of the flask resting on the bottom of the bath, and the concentric rings placed around the neck of the flask. (Note 3.) After 2 hours, remove the flask from the steam bath and cool to room temperature. (Notes 4 and 5.) Transfer a 5 ml aliquot to a 100 ml volumetric flask, allowing to drain for 5 minutes, and fill to mark with sulfuric acid. (Note6.) Mix well and fill a 1 cm cell for the spectrophotometer. Determine % T at the two minima found around 815 and 855 mμ, using conc. sulfuric acid as a blank, and convert to optical density (optical density=-log T). (Note 8.) Calculate % CPC in terms of a known standard.

B. Procedure for CPC Blues: Transfer 0.0600 g of finely powdered pigment into a 250 ml beaker. (Note 1.) Add a few drops of conc. sulfuric acid to wet the sample, and disperse well with a stirring rod. Gradually stir in more sulfuric acid to a volume of about 100 ml. Carefully transfer to a 500 ml volumetric flask. Wash out beaker with additional portions of sulfuric acid until flask is filled to the mark. Mix well. (Note 4 .) Transfer a 5 ml aliquot to a 250 ml volumetric flask, allowing to drain for 5 minutes, and fill to mark with sulfuric acid. (Note 7.) Mix well and fill a 1 cm cell for the spectrophotometer. Determine % T at the two minima near 700 and 790 mμ, using

conc. sulfuric acid as a blank, and convert to optical density (optical density = -log T). (Note 9.). Calculate % CPC in terms of a known standard.

VIII. Establishing a Standard: The ideal reference standard would be a sample of 100% CPC. The extinction coefficient of a solution of such a sample could then be calculated from equation (1) of section IX. In actual practice it is often more convenient to determine the extinction coefficient of some sample set up as a standard and express the results on a percentage basis relative to that standard. In any event, an extinction coefficient must be established for a suitable standard using the same or equivalent glassware, cells and spectrophotometer as that used for the unknowns.

For this method, the attempt was made to purify a typical LB type and a typical Cl-free type by acid-pasting. The percent purity was determined by a copper analysis and the extinction coefficient expressed on a 100% purity basis. For the polychlor CPC this was not done. AGT-722-D, SW-2609 was used as the reference standard. The "k" values obtained under these conditions are given below with "k" expressed in units of light path in centimeters and concentrations in g/l.

	k
Cl-free CPC	409 at 790 mμ
LB-CPC	373 at 795 mμ
AGT-722-D, SW-2609	100 at 855 mμ

For those purposes where these "k" values are suitable, they may be used directly in equation (3) of section IX in calculating % CPC. For reference purposes, all reports on percent purity using this method should state the nature of the material used as the standard and the extinction coefficient used in the calculation of % CPC.

IX. Calculations:

Let k = extinction coefficient of solution of reference standard CPC
d = optical density of solution of reference standard CPC
c = concentration (g/l) of solution of reference standard CPC
k' = extinction coefficient of solution of CPC sample
d' = optical density of solution of CPC sample
c' = concentration (g/l) of solution of CPC sample

(1) $k = \frac{d}{c}$

(2) $k' = \frac{d'}{c'}$

(3) $\%CPC = \frac{k'}{k} \times 100$

Notes:

1. All glassware should be free of sulfuric acid-soluble contamination. It is advisable to clean all glassware with sulfuric acid-dichromate

cleaning solution, rinse well with water, rinse with alcohol, and dry with suction tube.

Sample is much easier to work into sulfuric acid when it is finely divided. Do not use a brush in transferring the sample from the weighing scoop - a spatula is more desirable.

2. At this point, the CPC will not be completely dissolved, so it is necessary to use heat to effect complete solution.

3. Because of the thermal expansion of sulfuric acid, it is necessary to provide flasks with sufficient space above the mark to allow for the increase in volume on heating. Allow about 15 ml in a 250 ml flask.

4. The time required for complete solution is a function of the initial particle size and composition of the sample. Completeness of solution may be observed by holding the flask in front of a strong collimated light beam.

Blue lakes containing dextrin and other similar materials should be allowed to stand in sulfuric acid for about 2 hours at room temperatures to effect complete solution. However, the solution should be run the same day as it will darken with age.

5. The solution should be at the mark on the flask as when originally filled. This eliminates the necessity of checking the temperature with a thermometer.

6. This gives a pigment concentration of 0.0100 g/l, which, in the case of a toner, has a minimum of about 10% T at 855 mμ. Lakes will require proportionately higher concentrations, e.g., for a 50% lake, the pigment concentration should be doubled.

7. This gives a pigment concentration of 0.0024 g/l, which, in the case of a toner, has a minimum of about 14% T at 790-795 mμ. For lakes, see Note 6.

8. For routine work, it is only necessary to determine the optical density at one of the two minima, For this method, the minimum around 855 mμ has been used in the calculations. The optical density of both minima are fairly close. Obtaining data for both minima has the advantage of identifying the pigment as truly being a polychlor CPC. Any odd variation in the ratio of the optical densities of these minima can be construed as suggesting some oddity about the sample which should then serve as a warning against the application of this method.

9. For routine work, it is only necessary to determine the optical density at the 790-795 mμ minimum. Obtaining data for both minima has the advantage of aiding in the identification of the pigment as being a CPC blue. Any odd variation in the ratio of optical densities of these minima can be construed as suggesting the presence of a light

absorbing impurity in the sample. If such is the case, this method either should not be used or standard methods of dealing with binary systems can be used to calculate the %CPC in the presence of the light absorbing impurity. The observed ratio for purified Cl-free CPC is 6.82 and for LB-CPC is 6.58.