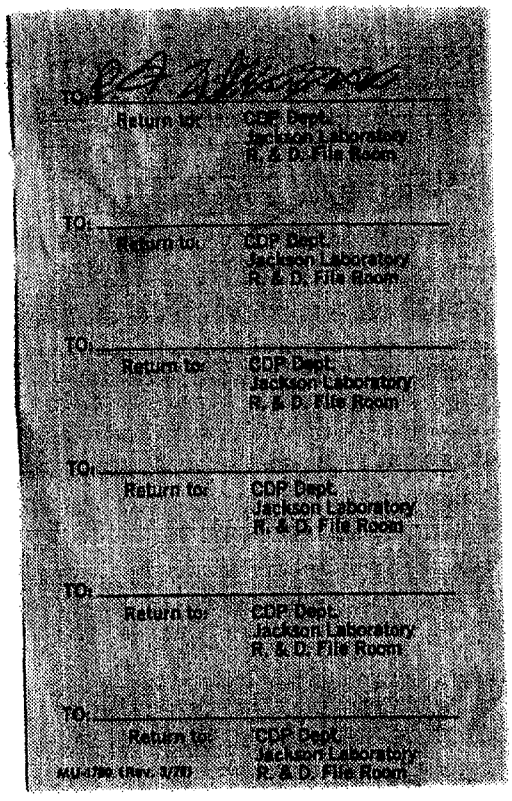
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Final Report

PHYSICAL AND CHEMICAL DATA ON POLYCHLOR CPC TO CHARACTERIZE GREEN GX

NOVEMBER 1, 1953 to AUGUST 1, 1954

CHARGE: A-IC-18

SUBMITTED BY: A. R. HANKE A R Hanke DATE SUBMITTED: 8/23/54

APPROVED BY: B. H. PERKINS B H Perkins 9/7/54 DATE ISSUED: 9/8/54

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I - INTRODUCTION:

The process variation that makes Green GX different from other polychlor CPC's is a two-fold one. First, the chlorination step is carried out in the presence of considerable anhydrous aluminum chloride. Second, the resulting product is treated with ODCB and aqueous caustic. There is no need for a grinding or acid slurry step. It is this last fact that makes the GX process so desirable.

If the product is fundamentally different from polychlor CPC's made in other ways, it may be possible to obtain a product patent. It is the object of this report to record the work done in finding a fundamental difference between Green GX and other polychlor CPC's.

In the course of this work many bits of factual information were obtained and it is the further purpose of this report to record this information for possible use in polychlor CPC technology in general.

II - SUMMARY AND CONCLUSIONS:

1. The following means of characterization were investigated:

   a. Spectrophotometric light transmission measurements in H_2SO_4 solution.
   b. Pigment purity by solvent extraction.
   c. Ultimate analysis for C, H, N, Cl, Cu.
   d. Semi-quantitative spectrographic analysis.
   e. Particle size index.
   f. Specific surface measurements by nitrogen adsorption.
   g. Angular dependence light scattering.
   h. Electron microscopy.
   i. Spectrophotometric light reflectance.
   j. X-ray diffraction.

2. Of all the methods examined, only angular dependence light scattering and X-ray diffraction showed any promise of distinguishing between different types of polychlor CPC. From the limited amount of work done with angular dependence light scattering, it appeared that acid slurried or acid pasted products had a wider particle size distribution than the milled products or Green GX. Hence, one could distinguish between these two groups. One could not, however, distinguish between Green GX and milled products.

3. Green GX can be uniquely characterized with X-ray diffraction. The characterization is based on the observation that certain peaks in the diffraction record that are well developed in solvent milled products are poorly developed in the case of polychlor CPC made with considerable aluminum chloride, i.e., Green GX. Acid pasted or acid slurried products also do not show the development of these characteristic peaks, but their diffraction pattern as a whole is much less crystalline than the pattern of Green GX; hence, GX can be distinguished from these products as well as from milled products. Electron micrographs from Jackson Laboratory of Orchem yield an explanation as to why the X-ray records of Green GX are unique. These micrographs show that Green GX has a unique crystal shape and size distribution.

4. A fair amount of factual information was obtained, such as ultimate chemical analysis, light transmission curves in sulfuric solution, and X-ray diffraction peaks of crystalline impurities in polychlor CPC. These data are recorded in the experimental section of the report.

III - PATENT SITUATION:

This work contributed to the characterizing of Green GX for the purpose of obtaining a product patent. Application for such a patent has been filed under Stryker, Williamson and Gross, Case 2, Serial No. 422,492. However, none of this work herein described is in itself of a patentable nature.

IV - EXPERIMENTAL:

Five samples were obtained from the CPC Group and all of the initial characterization work was done on these five samples. When it became evident that X-ray diffraction could characterize Green GX, the X-ray method was applied to many other samples in the attempt to find an exception to the rules laid down by the X-ray method. No exception was found.

The five samples were labeled as follows:

1479-7A - Dispersion milled SW-2585.
1479-7B - Typical Green GX SW-3457-2.
1479-7C - Solvent milled N-624 (511).
1479-7D - Acid slurried - an Orchem product.
1479-7E - Acid pasted - an Orchem product.

A. MEASUREMENTS RELATED TO CHEMICAL COMPOSITION

1. Light Transmission Measurements of Sulfuric Acid Solutions of Polychlor CPC.

Polychlor CPC gives a characteristic spectrophotometric light absorption curve in the near infra-red when dissolved in H_2SO_4. Chemical differences, such as extent of chlorination, can be observed by this method. This method can also be used to determine relative purity by observing the ratio of extinction coefficients.

Sulfuric acid solutions were obtained for the five pigments following the method outlined in Tentative Analytical Method 6/13/52. Some difficulty was had by the analytical group in getting complete solution, but the ratio of extinction coefficients is believed to be sufficiently precise to indicate relative purity. The curves all had the shape characteristic of polychlor CPC. A record of such a curve is given in KN-53-23. There is also given curves of lower chlorinated species of CPC as well as curves of polychlor meta-free CPC and polychlor AlPC. Table I shows the extinction coefficients and the relative percent purity estimated from them.

2. Pigment Purity by Solvent Extraction.

Each of the five pigments was subjected to the solvent extraction procedure of the analytical group recorded as Tentative Method 3/27/52. This employs a mixed solvent consisting of hydrochloric acid, acetic acid, 23A ethyl aocohol, and benzene in a volume ratio of 10/40/45/5. This solvent admittedly does not remove all impurities, but it serves as a useful purity index. Table I shows the results of the extraction method and also offers a comparison with the extinction coefficient method.

TABLE I

	*Ext. Coeff.	**Relative % Purity	% Purity by Extraction
1479-7A Dispersion Milled	90	83	89
-7B Green GX	108	100	96
-7C Solvent Milled	99	91	93
-7D Acid Slurried	105	97	97
-7E Acid Pasted	106	98	98

*The extinction coefficient is expressed as optical density per 1 cm light path per gram per liter.

**A relative purity of 98% was assigned to sample 7E to make it identical to the 98% obtained by extraction. This makes for easy comparison of both methods.

3. Ultimate Analysis

Total Cl and total Cu were obtained by our analytical group. Total C, H, and N were obtained from the Microanalytical Laboratory of Carl Tiedke of Teaneck, N.J. The results are shown in Table II.

TABLE II

	% Cl	% Cu	% C	% N	% H
1479-7A Dispersion Milled	44.8	5.01	34.8	9.8	0.7
-7B Green GX	47.1	5.31	35.0	10.1	0.0
-7C Solvent Milled	46.3	5.46	34.9	10.1	0.0
-7D Acid Slurried	47.9	5.47	35.1	10.2	0.0
-7E Acid Pasted	47.8	5.44	34.5	9.2	0.0

NOTE: Only the 7A sample showed evidence of trace amounts of inorganic Cl and Cu. The other samples showed none. The C, N, and H determinations were made on the solvent extracted samples, whereas the Cl and Cu analyses were on the "as is" samples.

4. Spectrographic Analysis

Semi-quantitative analyses were obtained on the "as is" samples by the Chemical Department of the Experimental Station. Table III shows the results.

TABLE III

	Elements Found
1479-7A Dispersion Milled	Major - Cu Minor - Pb, Fe, Si Trace - Al, Ba, Mg, Ca, Mn, Mo. Na
-7B Green GX	Major - Cu Minor - Ca Trace - Mg, Fe, Al, Si, Mo, Na, Mn
-7C Solvent Milled	Major - Cu, Sb Minor - Fe, Al, Ca Trace - Zn, Pb, Si, Mo, Mg, Na, Ni, Mn
-7D Acid Slurried	Major - Cu Minor - Fe, Al Trace - Ca, Pb, Si, Mg, Mn, Mo, Na
-7E Acid Pasted	Major - Cu Minor - Al, Si, Ca Trace - Fe, Mg, Mn, Mo, Na

5. X-ray Diffraction to Detect Crystalline Impurities

During the chemical examination of sample 1479-7A, dispersion milled polychlor CPC, it became evident that it was the least pure of the set of five samples examined. The X-ray record showed some lines thought to be due to these impurities. To test this point, the X-ray record was re-run on the solvent extracted sample. It was observed that some lines disappeared or became much less prominent. These lines were considered as due to crystalline impurities and a realization of this will aid in interpreting X-ray patterns of polychlor CPC. It is not meant to imply that these peaks represent all of the crystalline impurities. There may be others. This is simply a start in classifying the crystalline impurities. Table IV shows the 2θ values for these peaks.

TABLE IV

IMPURITY PEAKS IN POLYCHLOR CPC

2θ (CuKα)	Intensity
20.9	Medium
27.7	"
29.7	"
32.4	weak
42.6	"
43.7	medium
44.6	weak

B - MEASUREMENT OF PHYSICAL PROPERTIES RELATED TO PARTICLE SIZE

1. *Particle Size Index

The five enamels were dispersed in toluene and the particle size index determined according to the method outlined in KN-51-43. The results are given in Table V.

2. Specific Surface Measurements

These were obtained by the Chemical Department using the standardized nitrogen adsorption technique. Results were had on both the "as is" pigments and the pigments after a non-aqueous extraction. The extractant was a mixture of hydrochloric acid, glacial acetic acid, 23A ethyl alcohol, and benzene in a volume ratio of 10/40/45/5. The results are given in Table V.

*This is a measurement derived from a spectrophotometer light transmission curve of a pigment dispersion. It is 100 times the ratio of minimum optical density to maximum optical density. See KN-51-43 and KN-52-2 for a complete description.

3. Angular Dependence Light Scattering

These were obtained by the Chemical Department on three different types of dispersed systems. These were (1) the masstone enamels dispersed in toluene with a small amount of triethanolamine oleate to aid in the dispersion. (2) Sprayed panels of the masstone enamels. In this case, measurements were made on the reflected light. (3) Pourouts of the masstone enamels on "Plastacele". In this case, measurements were made on the transmitted light. The most meaningful results were had using method (1) in which the enamels were dispersed in toluene. The other two methods, involving measurements on a dry medium appeared to be too greatly influenced by surface irregularities which have nothing to do with the characterization of any of the pigments. Figure I shows the angular dependence light scattering data plotted in a manner developed by the Chemical Department. See letters ARH to BLP, 11/13/53 and BLP to ARH, 1/19/54.

TABLE V

	Before Extraction			After Extraction	
	Specific Surface sq.meter/gr.	*Average Particle Size mμ	Particle Size Index	Specific Surface sq.meter/gr.	Average Particle Size
1479-7A Disp.Milled	82.4	34.7	6.1	53.8	53.1
-7B Green GD	46.7	61.2	6.4	66.6	42.9
-7C Solv.Milled	75.4	37.9	6.5	71.7	39.9
-7D Acid Slurried	80.1	35.7	6.7	88.5	32.3
-7E Acid Pasted	73.1	39.1	7.8	69.7	41.0

*This assumes a specific gravity of 2.1 and is calculated from the specific surface using the formula -

$$\text{Average Particle Size} = \frac{6 \times 100}{\text{Sp.Surface} \times \text{Sp.Gravity}}$$

4. Electron Microscopy

The five samples were examined with our RCA Console Model EMC. They were examined using both the dry pigment and alkyd enamels made from the pigment. It became evident that the size of the ultimate particle was too small to be well resolved by our microscope. All pigments were highly aggregated and no good pictures were obtained showing the true size and shape of the crystallites. The size and shape of the aggregates did not offer anything distinctive and since Jackson Laboratory with their better microscope was also obtaining electron micrographs of polychlor CPC, no further work was done in this field.

5. Light Reflectance Measurements of Alkyd Enamel Extensions

The degree of dispersion (effective particle size) can influence the shape of a spectrophotometric reflectance curve. The change in curve shape has been studied for CPC Blue and briefly for polychlor CPC. This was reported in KN-50-15.

Normal alkyd grinds of the five pigment samples were obtained. 10/90 extensions were prepared and sprayed on aluminum panels. Reflectance curves were obtained in normal manner using the G.E. spectrophotometer. In most cases, Green GX is associated with yellowness of extension and an intensity that exceeds that of the other types of polychlor CPC. This is illustrated by the curves. Figure 2 compares 1479-7B, Green GX, with 1479-7E, acid pasted polychlor CPC. This serves to illustrate the curve shift which accounts for the yellower hue of the Green GX. This will be discussed in greater detail in the discussion section.

C. MEASUREMENTS RELATED TO CRYSTAL STRUCTURE

1. X-ray Diffraction

Since this method looked promising from the start, the five original samples were supplemented, bringing the total number of samples to 26. Typical diffraction records are shown in Figure 3. The details for obtaining and interpreting the diffraction records are given in Appendix I. The results are shown in Table VI. Most of these results were obtained using the method supplied in Appendix I. In some cases, the details were modified to scan at the rate of 1°/min. with a chart speed of 1/2 inch/min. and the time constant set at 4 sec. For a few curves the "multiplier" was set at 0.8 instead of 0.6. This was done to get the single strong peak at 27.6° on the paper. None of these variations made any difference in the interpretation or conclusions drawn from the records. The data of Table VI gives more information than is required to apply the method outlined in Appendix I. This is done to show that the trend of moderately high crystallinity with a decrease in peak heighth of certain peaks which characterizes Green GX, extends to peaks other than the ones chosen. The choice is dictated by ease of peak measurement and magnitude of the effect. In other words, Green GX can also be characterized by measurements of peaks other than the ones chosen.

TABLE VI

No.	Sample	Peak Intensity Relative to 26.7° peak (%) at Bragg Angles			Peak Width at half Maximum Intensity in Degrees at Bragg Angles	
		28.6°	15.6°	6.1°	15.6°	6.1°
1	GX-1479-7B	27	12	29	0.4	0.35
2	GX-7043-123-2	32	17	33	0.3	0.40
3	GX SW-3457-1	25	11	24	0.3	0.4
4	GX Orchem lot 2	31	15	33	0.4	0.4
5	GX Orchem lot 1	32	21	40	0.4	0.25
6	GT-751-D SW-3457-1 (Green 70)	26	14	29	0.4	0.35
7	Green 25 1454-25	25	20	35	0.25	0.35
8	Green 25 1454-29A	33	14	37	0.35	0.3
9	GT-722-D, SD-2609 (Solv.Mill SCl_x)	72	28	57	0.3	0.3
10	Solvent Milled 1479-C	51	15	42	0.4	0.3
11	Blue Shade GX-1454-27E(Less $AlCl_3$)	47	23	57	0.35	0.35
12	GX Solvent Milled 1452-30	26	12	24	0.45	0.55
13	Solvent Milled Orchem Eutectic 1479-24C	23	10	25	0.5	0.6
14	Solv.Milled GX crude 1479-24D	32	9	34	0.8	0.6
15	Solv.Milled High $AlCl_3$ ODCB treat. crude lot G-19. 1479-24B	30	13	30	0.4	0.55
16	Dispersion Milled 1479-7A	18	8	20	1.1	0.85
17	Acid-slurried 1479-7D Orchem G-8	7	3	12	0.8	0.7
18	Acid-slurried 7043-123-6	9	4	16	0.75	0.5
19	Acid-slurried 7043-123-4	7	3	17	1.0	0.7
20	Acid-pasted 1479-7E	-	5	18	0.6	0.9
21	Acid-pasted 7043-123-3	-	-	19	-	0.9
22	Acid-pasted 7043-123-5	-	-	18	-	0.9
23	C-54423 Heliogen Grn.(Gen.Dyestuff)	8	2	20	0.7	0.8
24	GT-674-D, SD-121 Solv.Milled	49	16	38	0.25	0.45
25	C-51030 "Monastral Fast Grn.(I.C.I.)	6	2	19	0.8	0.75
26	C-54822 Heliogen Green GA new.	7	4	16	0.8	0.55

V - DISCUSSION

A. Chemical Analyses

From the point of view of showing something unique about Green GX, chemical analyses offered nothing. The initial thoughts that Green GX may be unusually pure or that it contained more than the agerage amount of chlorine in its molecule, or contained some copper-free chlorinated species, or that it was devoid of any trace of lower chlorinated species, whereas the usual polychlor CPC would always have some lower chlorinated species, all were disproved by the data. The accumulated chemical data, although it did not aid in characterizing Green GX, should prove to be useful factual information regarding the chemical nature of polychlor CPC. Relative pigment purity is shown in Table I. It should be recognized that a systematic error exists in all these purity figures which means that the products are actually less pure than this table indicates. The results of ultimate analyses is shown in Table II, and spectrographic analyses are given in Table III. X-ray diffraction peaks of at least some of the crystalline impurities are given in Table IV.

B. Particle Size and Size Distribution

Since a yellow hue and a light masstone seemed to be associated with the Green GX material, and since changes in particle size can alter these properties, particle size appeared to be a likely distinguishing characteristic of Green GX. Following this lead was disappointing because specific surface measurements (See Table V) showed nothing unique about Green GX and actually showed that the measurements were unreliable because of the effect of impurities on the pigment surface. Also, it can be seen from Table V that the particle size index which is a function of an average particle size showed nothing unique about Green GX. This left particle size distribution as something that might set Green GX apart. Angular dependence light scattering appeared promising. As can be seen from Figure 1, the curves of acid-pasted and acid-slurried material are different from those of Green GX or the milled samples. An interpretation of these curves would have the Green GX and solvent milled samples (B & C) almost identical with practically no large material above 10 μ diameter, and very little between 0.6 and 10 μ diameter. Most of the material is in the 0.4 μ diameter range. The dispersion milled sample (A) would also be placed in this class, but it does have a slightly broader distribution in the 0.2 μ to 2 μ diameter range. The acid-slurried and acid-pasted samples (D & E) are in a class by themselves. They have a much wider distribution extending from 0.2 to 8 μ diameter. It would seem that sulfuric acid treated products could be distinguished from milled or GX products.

Another contributing bit of evidence is spectrophotometric reflection curves of alkyd enamel TiO2 extensions. Figure 2 shows the difference between GX and acid-pasted polychlor CPC. As previously stated, Green GX appears to be associated with yellowness of hue. This yellowness can be described by the shape of the spectrophotometric curve. An increase in yellowness can be brought about by any of three different changes in curve shape.

(1) A raising of the right hand portion of the curve.
(2) A lowering of the left hand portion of the curve.
(3) A shift of the maximum reflectance to the right.

From our experience with the change in curve shape with CPC flocculation or crystal growth (KN-50-15, KN-50-18, KN-51-40) we can say that an increase in particle size, in the size range of large crystals or flocs, will raise the right hand portion of the curve. This would make a polychlor CPC appear yellower but duller. This is not the kind of change experienced here. Hence, a change in particle size in this size range is not indicated. The Green GX curve is sharper and shifted towards the yellow. This would make the color yellower and more intense. A greater intensity would be expected from a more mono disperse system and this is what angular dependence light scattering shows.

Our electron micrographs do not show any marked difference in degree of dispersion or particle size distribution. However, the electron micrographs make it evident that angular dependence light scattering deals with an aggregate quite a bit larger than the ultimate crystallite. No good explanation is offered why the electron micrographs do not reflect the size distribution indicated by angular dependence light scattering. This size distribution should be well within the range of resolution of our electron microscope.

Jackson Laboratory (Memo of T. E. Beukelman, 2/1/54) with their higher resolving microscope did observe the crystallites of polychlor CPC. Green GX is distinctive in that "The individual particles of Green GX appear to be rectangular parallelopipeds. The long dimension lies between 0.05 μ and 0.2 μ and is two to three times the width and five to ten times the thickness. The particle size distribution is very narrow and there appears to be no small irregular fragments."

It is interesting to note that electron micrographs, angular dependence light scattering, and reflectance curves, all class Green GX as more monodisperse than the other types. The size range of the electron microscope classification is smaller than that of angular dependence light scattering. It would appear that the mono disperse nature of the crystals of Green GX influences the aggregation in such a manner that monodisperse aggregates are formed.

Acid-pasted polychlor CPC appears to have a smaller crystallite size with a broader distribution of crystal sizes, but also forms larger aggregates giving a broader aggregate size distribution as well. The particles that fix the tinctorial properties in light reflectance curves are very likely aggregates because electron micrographs of alkyd enamels show aggregates. So, as with angular dependence light scattering, it is the aggregates that we are measuring as a monodisperse system when making light reflectance measurements of a Green GX alkyd extension.

It has been stated (Ref.) that acicular particles will tend to produce a yellower hue in a green than spherical particles. Green GX has a more acicular crystal than the other forms, but this explanation for the yellow hue suffers from the fact that in a typical alkyd the pigment particles are not dispersed down to its ultimate crystallite size. It is known that crystals of chlorine-free CPC are dichroic and it is reasonable to suppose that polychlor CPC is also dichroic. Altering the shape of the crystal can then be expected to change the hue because light transmission in certain directions through the crystal will be favored over other directions. Probably a combination of both of these factors are operating to give Green GX its yellow hue. It appears fairly well established, however, that the intensity in Green GX is due to the monodisperse nature of the pigment and not due to any unusual purity or degree of chlorination.

C. Crystal Characteristics by X-ray Diffraction

X-ray diffraction examines differences in the crystalline nature of the product. Crystallite size, crystal phase, and crystal shape can affect the X-ray diffraction patterns. The X-ray diffraction pattern of polychlor CPC is very complex. There are many diffraction peaks, but few very large ones. Figure 3 illustrates how different the records can be for different types of polychlor CPC. The difference lies mainly in the development of the peaks and not in the creation or disappearance of peaks. This means that there are no phase changes, but that we are dealing with a change that can affect the relative intensities and the width of the peaks. A change in crystallite shape and size can do just that. The broader the peak, the smaller the size in the crystal direction responsible for that peak.

The fact that Orchem's electron micrographs do show a characteristic crystal shape and size for Green GX, lends support to the postulate that the characteristic X-ray record of Green GX is indeed due to this change in shape and size. It but remains to put a quantitative interpretation on the differences observed in Figure 3 between Green GX and the other types. This has been done and a detailed description of the method is given in Appendix I. Table VI shows the results of the application of this method to 26 samples of polychlor CPC. All Green GX samples in the table

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REF. Maresh and Kienle, Observations on Optical Properties of Pigmented Films. Official Digest of the Federation of Paint and Varnish Production Clubs 26, 5, (1954).

meet the requirements described in Appendix I, with the possible exception of No. 11. This, however, since it was made with less $AlCl_3$ cannot rightly be called a Green GX because one of the necessary requirements of Green GX is that it be made with a considerable excess $AlCl_3$. It is the presence of this excess that inhibits the development of the 28.6° peak.

This work is recorded in Notebooks 1476-25 and 1496-3-9-14.

MPH

APPENDIX I

CHARACTERIZING GREEN GX USING X-RAY DIFFRACTION

PRINCIPLE:

1. X-ray diffraction records show that Green GX material is of a fairly high degree of crystallinity. This distinguishes Green GX from all acid-pasted or acid-slurried products which are much less crystalline, but does not distinguish it from normal solvent milled green which also is fairly crystalline.

2. X-ray diffraction can be used, however, to distinguish between finished pigment prepared by chlorinating in the presence of considerable aluminum chloride and a normal solvent milled product. The difference resides in the relative intensities of some of the diffraction peaks. If the intensity of all peaks is measured relative to the strong 26.7° peak, the peaks at 28.4°, 26.0° and 25.1° are weaker than in a normal solvent milled green. It appears that the presence of considerable aluminum chloride during chlorination tends to inhibit the subsequent development during solvent treatment of the interplaner spacings that are responsible for these peaks.

3. The solvent milling of a polychlor CPC chlorinated in the presence of considerable aluminum chloride results in a product with a lower degree of crystallinity than an ordinary solvent milled product or a Green GX. The high aluminum chloride solvent milled product can, therefore, be distinguished from the Green GX product even though both were derived by chlorination in the presence of considerable aluminum chloride by observing that Green GX has a higher degree of crystallinity.

DETAILED METHOD FOR CHARACTERIZING GREEN GX

Use a Philips X-ray diffraction unit equipped with their high angle diffractometer and a Brown Potentiometer Recorder. Use CuK_α radiation at 35 kilovolts and 18 milliamperes passed through a nickel filter. Set the rate of scan of the diffractometer at 1/4 degree per minute and set the chart speed at 1/8 inch per minute. Use a slit system consisting of 1° divergence slits and 0.003" receiving slit. Under these conditions, a peak at maximum sharpness at around $2\theta = 7°$, has a width at half maximum intensity of about 0.18°. Set the rate meter scale factor at 4. Set the multiplier at 0.6. Set the time constant at 8 sec. Under these conditions, a chart scale reading of 100 from instrument zero means a Geiger tube count of 120 counts per sec.

To characterize Green GX proceed as follows:

1. Obtain an X-ray diffraction record using the equipment as described above.

2. Draw a base line through the points of intersection of the curve and Bragg Angles of 30° to 21° to 19° to 16.4° to 14.5° to 11° to 8° to 7.3° to 5.0°. The base line will not be parallel to the Bragg Angle axis but should give a fair representation of the background in the vicinity of the peaks to be measured. If there is any impurity peak at one of these points, it should be ignored and the base line position estimated at some nearby point away from any peak.

3. Determine peak intensities by measuring peak height from the base-line at 26.7°, 28.5°, and 6.1°. Express the peak intensities relative to the intensity at 26.7°. Call the intensity of that peak 100%.

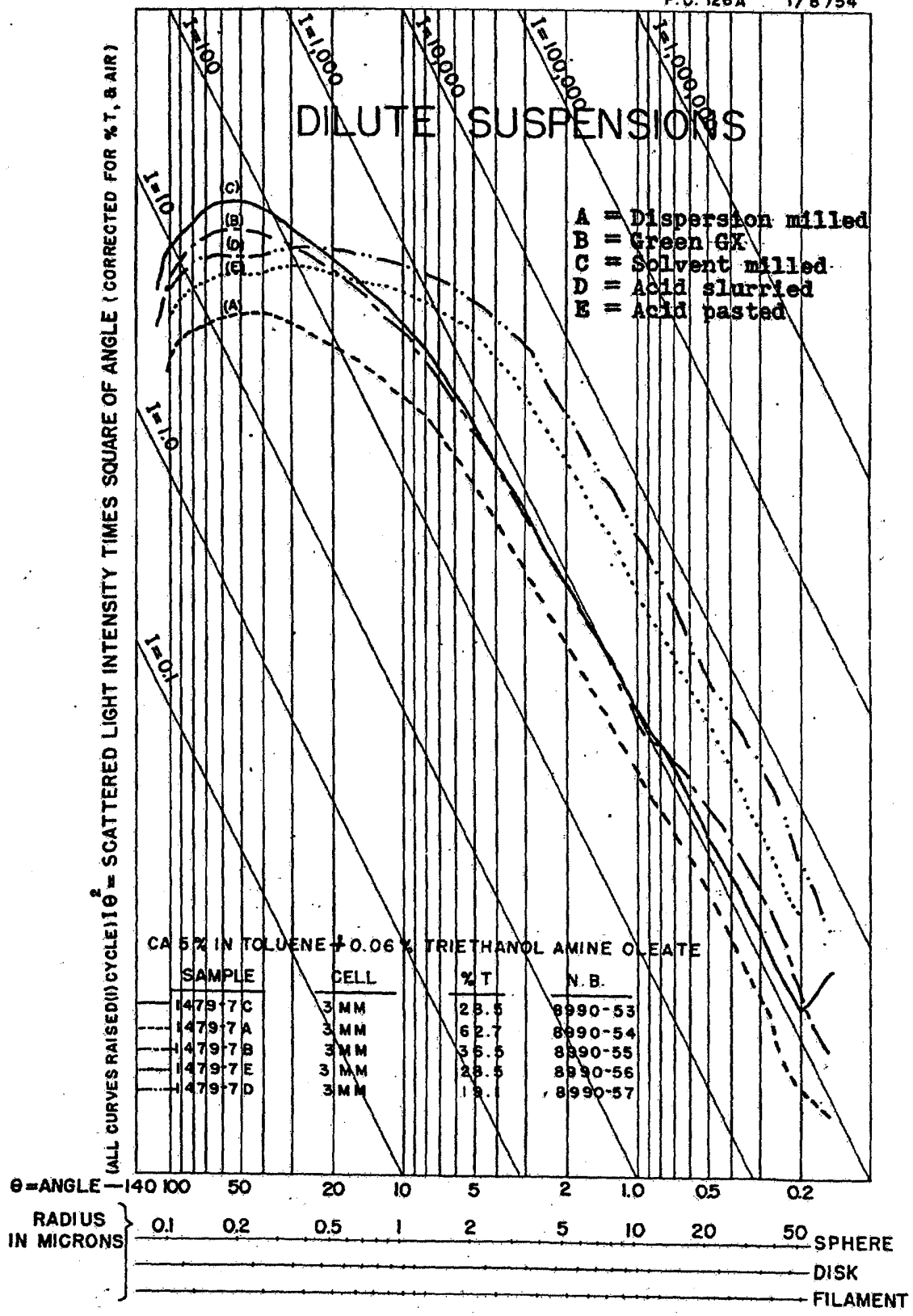
4. Measure peak width at half maximum intensity for the peak at 6.1°. Determine the width by drawing a line parallel to the base-line through the point of half maximum intensity. Measure the length of the horizontal projection of this line and express the results to the nearest 0.05°. This method of measurement is used to compensate for the sloping background, but admittedly it is a refinement that does not change the actual measurement a great deal.

Patterns derived from Green GX will have a line width at 6.1° of less than 0.5° and a relative peak intensity at 28.6° of less than 40% but more than 18%. Solvent milled material made with a normal chlorination technique will have a relative intensity at 28.6° of more than 40%. Solvent milled material made with considerable aluminum chloride present during chlorination, and all acid-pasted and acid-slurried products will have a line width at 6.1° of 0.5° or more.

FIG. 1

ANGULAR DEPENDENCE LIGHT SCATTERING OF POLYCHLOR CPC ENAMELS

NO. 390
A.R. HANKE
P.C. 126A 1/8/54



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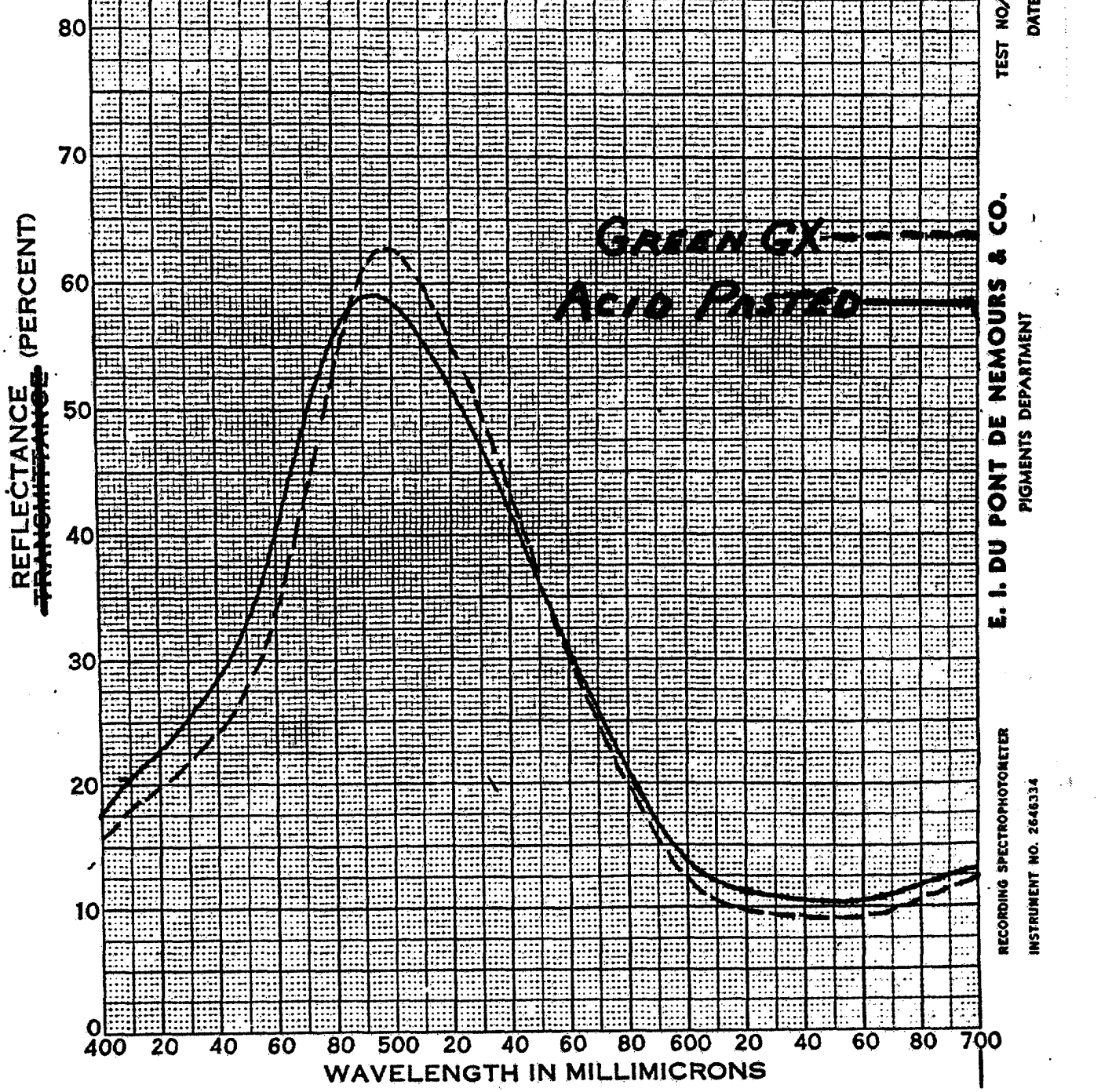
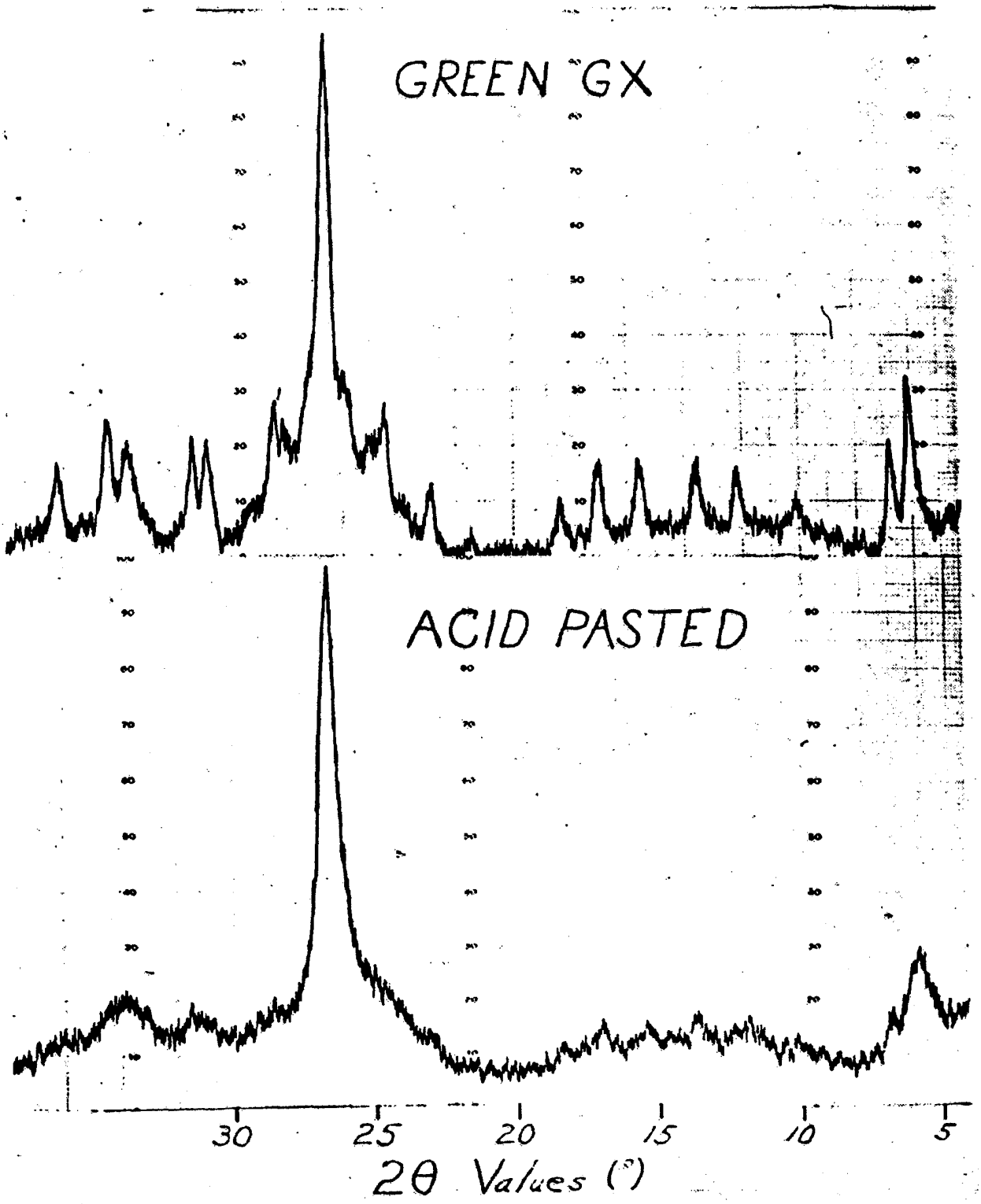


FIG. 3



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