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NEWARK PLANT
CHEMICAL DIVISION - COLORS

FINAL REPORT

TITLE: **DURABLE GRAY PIGMENTS - DEVELOPMENT OF "ARGENT GRAY" A-6-D**

Period Covered **1950 - 1953**

CHARGE: **1100-11-272**

FILE: **210**

DATE: **8/17/53**

NJ 5747

ITN-53-19

210

Serial No. KN-53-14

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SUBMITTED BY: R. R. DENSLOW DATE SUBMITTED: 7/21/53

APPROVED BY: B. H. PERKINS 8/17/53 DATE ISSUED: 8/17/53

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INTRODUCTION:

This report covers the work started early in 1950 on gray inorganic pigments leading to the development and standardization of "Argent Gray, A-6-D". The major portion of the study was carried out under Experimental Project #272.

SUMMARY AND CONCLUSIONS:

In a search for new pigments which could be used to formulate metallic gray enamels superior to those in commercial use, a number of inorganic compounds were investigated. A number of promising candidates were found. Of these, the one of most interest to F & F and Sales was the material obtained by the addition of sodium hydroxide/sodium phosphate to a solution of copper sulfate. This product was coded A-6-D and subsequently released to the trade as "Argent Gray".

The process was carried through the Semi-works and plant without noteworthy difficulties and has been accepted by the Production Division.

An active Sales promotional campaign was undertaken and the material is under test by a number of customers.

Evaluation work has given generally favorable results. Recent tests have indicated certain deficiencies, the most serious being gloss retention, in which it is apparently surpassed by other products, viz. Fe_2O_3/Cr_2O_3 and CuO/Cr_2O_3 combinations. Any decision as to future promotional activity must await the results of current customer tests and the completion of numerous exposure series now in progress.

A product/process patent application has been filed on A-6-D.

TABLE OF CONTENTS:

The problem is discussed below under the following headings:

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HISTORICAL DEVELOPMENT - A-6-D

Some years ago (circa 1940-1941), F & F (Parlin) prepared experimental transparent finishes from special black iron oxide pigments. These ferroso-ferric oxide combinations gave attractive metallics including both colors and grays with a high degree of "two-tone" flash. On exposure, however, they were found to be unsatisfactory because of their tendency to go from black to brown, presumably as a result of oxidation of the ferrous iron in the pigment.

A cooperative study (with Parlin) was carried out in 1944 aimed at the development of a pigment which would give color effects similar to those obtained with the ferroso/ferric oxide products but with better resistance to color change on exposure. A partial solution to the problem was obtained by reducing the amount of ferrous iron present by treating the pigment slurry with bichromate. Material prepared in this way was coded as AF-7-P and is claimed, as part of a coating composition, in U.S. Patent 2,466,770. Development studies were carried as far as the Semi-works but subsequently discontinued for two principal reasons: (1) the pigment was similar to F-4-P in respect to color drift of the pulp on storage. (2) it was not a true black but rather a dark brown. Furthermore, indications were that reproducibility of the process left something to be desired.

Among the other black pigments investigated at the same time was cupric oxide, prepared by the addition of sodium hydroxide to cupric sulfate. It showed some promise in Parlin's preliminary tests and was later exposed in lacquer in our Series 6978, also with promising results.

No further work on blacks was carried out until 1950. In March of that year, the technical representatives of F & F indicated that there was a definite need for pigments to make gray finishes (particularly metallics) which would be superior to existing products in color and durability. Conventional pigmentations were said to be unsatisfactory in respect to film chalking and fading, making refinishing difficult. Gloss loss of conventional grays was particularly serious since it was impossible to bring back the original gloss by polishing.

Laboratory work was undertaken covering a general survey of black/gray compounds to select one or more candidates for evaluation in respect to the properties mentioned above.

On the basis of the earlier work, further attention was given to precipitated cupric oxide. This is easily made from copper sulfate and sodium hydroxide. Some variation in color and strength can be obtained by changes in the method of precipitation, thus making it possible to produce grays of different types when aluminum is added. It was found to give self pigment (masstone) enamels of only moderate transparency and, as a result, the metallics showed considerably less

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flash than those made from F-4-P or AF-7-P.

To correct this deficiency, experimental samples were made by coprecipitating copper oxide with other insoluble products having lower opacity but which would be expected to have adequate durability. Products were also made comprising precipitated (black) manganese oxide alone or in combinations. (Work on these latter was dropped later since they usually showed rather severe color change on exposure). Preliminary tests for color were made by small alkyd resin grinds and panels were exposed in the Weatherometer. On the basis of discussion of the results with F & F representatives, small Semi-works batches were made of the following for further tests.

DRY	PULP	COMPOSITION
AA-4-D	AA-5-P	Copper oxide
6	7	Copper oxide/orthophosphate
8	9	Like A-6 but higher in CuO (stronger)
10	11	Iron/Chromium/Copper oxide
12	13	Copper/Manganese oxide
14	15	Copper/Zinc Oxide
16	17	Like A-14 but lower in CuO (lighter)
18	19	Copper oxide/lead sulfate
20	21	Copper/Magnesium oxide

In masstone color, these were all gray or black; with aluminum, they gave attractive finishes, varying from brownish to relatively neutral "metallic grays". These metallics were exposed in S-10192 against metallics from Mapico Black, Lamp Black, "Ti-Pure"/lamp black blends and three commercial automotive refinish enamels. (see exhibits in appendix). The following samples showed better performance than the others: AA-4-D, AA-6-D, AA-8-D, AA-10-D, AA-14-D, AA-16-D. AA-6-D and AA-10-D were considered as the most promising, AA-6-D being preferred by F & F and Sales because of its more neutral (less brown) color. The outstanding properties demonstrated in this series for the better products were:

Color and Gloss retention - unpolished film
Color retention - polished film
Gloss recovery upon polishing back

Three additional Semi-works batches of A-6-D were made and blended (Lot 1421) to supply material for complete evaluation by Sales Service and the preparation of Sales' promotional exhibits.

Initial plant manufacture was undertaken in March 1952, three 1450 lb. batches being made. An equal part mix of these was made up as lot 85498 which served as a tentative working standard and to supply samples and fill orders from manufacturers of automotive finishes. Compared with lot 1421, lot 85498 was vs/s light in masstone, very slightly strong (ca 95) and vs/s red in tint. Additional manufacture (3 batches) was carried out in December 1952, without any serious difficulties. A new mix (89671/89672) was made up from this material

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together with some of the earlier manufacture, and set up as the first official standard. It was sl dark, vs intense; vvs weak (101); vs yellow vs 85498 and also close to an equal part mix of the six plant batches. The process was taken over by the Production Division, March 2, 1953.

PATENT SITUATION:

An application (Case 1227-K) for a product and process patent on A-6-D has been filed. The product claims are based essentially on the unexpected amorphous character of the product. This lack of crystallinity is obtained only in a narrow range of composition and by carrying out the reaction under specified conditions. Coating compositions claims have also been made.

The patent concerned with the iron oxide "black" pigment, AF-7-P mentioned earlier in this respect in U.S. 2,466,770 (Hucks and Denslow).

No consideration has been given to the possibility of obtaining protection on the other gray/brown pigments (A-10-D, etc.) mentioned above.

CHEMISTRY OF A-6-D AND MECHANISM OF ITS FORMATION:

A-6-D is prepared by the addition to copper sulfate (1 mol) of a mixed solution of sodium hydroxide (1.5 mols) and disodium phosphate (0.5 mols), the two solutions being at an elevated temperature. It was originally assumed that this would give a mixture of oxide and phosphate according to the following equation which involves 1.0 mol of caustic, leaving an excess of 0.5 mol.

$$CuSO_4 + \begin{cases} 0.5\ NaOH \rightarrow 0.25\ Cu(OH)_2 \rightarrow 0.25\ CuO\ (\text{black}) + 0.25\ H_2O \\ \qquad\qquad\qquad\qquad \text{blue} \\ 0.5\ NaOH + 0.5\ Na_2HPO_4 \rightarrow 0.25\ Cu_3(PO_4)_2\ (\text{blue-weak}) \end{cases}$$

It has been found that the additional 0.5 mol caustic is necessary to obtain the color characteristics of A-6-D. If only one mol is used, the product is not black or gray but bluish green and has been identified as a definite basic salt, ($CuO:P_2O_5$=4:1) corresponding to the mineral libethenite and giving the same characteristic X-ray diffraction pattern as the latter.

The actual composition of A-6-D has been found to approximate the mol ratio $CuO:P_2O_5$=7/1 (6/1-8/1). When formed in this range under specified conditions, it is substantially amorphous, giving only a slight indication of the CuO X-ray lines and no indication of the characteristic libethenite lines. (See appendix).

The particle size is very small. Visual examination of electron micrographs (see appendix) indicates it to average less than 0.1 micron.

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It is believed that the mechanism of the formation of A-6-D involves, as a first step, the precipitation of the basic phosphate (CuO/P_2O_5=4/1) together with some copper oxide/hydroxide. As the alkalinity builds up towards the end of the strike, there is a change in color from bluish/green to a gray which becomes somewhat darker at higher temperatures or upon aging. Presumably, this color change is the result of the reaction of the excess sodium hydroxide on some of the basic phosphate as indicated in the following equations. (For simplicity, the starting material has been assumed to be a normal rather than a basic phosphate).

(a) $Cu_3(PO_4)_2 + 6\ NaOH \rightarrow 3\ \underline{CuO} + 3\ H_2O + 2\ Na_3PO_4$

(b) $Cu_3(PO_4)_2 + 4\ NaOH \rightarrow 3\ \underline{CuO} + H_2O + 2\ Na_2HPO_4$

According to reaction (a), the quantities of ingredients used in A-6-D would yield, as a final product, material of the composition 6 CuO/1 P_2O_5 with a substantial amount of phosphate in the backwater. Reaction (b) would give 8 CuO/1 P_2O_5. As mentioned earlier, analyses of typical samples have given approximately 7 CuO/1 P_2O_5.

The presence of phosphate in the A-6-D filtrate has been shown by the quantitative determination of the ingredient balance in a laboratory experiment, as well as by simple qualitative tests on plant filtrates.

The degree of hydrolysis is determined by the amount of caustic used, the concentration of the reactants, the temperature, and possibly other factors, and is quite critical if an amorphous product of standard analytical composition is to be obtained. With too little caustic, the product is light and greenish, weak and shows to some extent the X-ray lines of the 4/1 basic phosphate. With too much caustic, the product is dark (brown) and strong and there is evidence (X-ray pattern) of the presence of free copper oxide.

Co-precipitation appears essential; attempts to form this product by successive addition of phosphate and sodium hydroxide have, so far, been unsuccessful. Striking at an elevated temperature (rather than striking cold and heating afterwards) also appears to be essential.

COMPOSITION AND YIELD:

A-6-D is apparently not a definite compound but more likely a mixture of amorphous cupric oxide and basic (4/1) cupric phosphate. The copper content, expressed as "% CuO", runs about 66-69% and the phosphate content, as "% P_2O_5", about 16-18%; these figures correspond to a CuO/P_2O_5 mol ratio of 6-8/1. Dark products are higher in copper content than the light ones.

In addition to the above, the products contain a little over 1% (as "SO_3") of sulfate, most of which is in water insoluble form and presumably present as basic copper sulfate. Sodium is also present

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(ca 2-3% as "Na_2O"), probably combined with some of the phosphate as a result of the hydrolytic action of water and/or alkali on the basic phosphate as discussed in the section on the mechanism of formation of the product.

A typical analysis (Lot 85498) is shown below:

Copper as "CuO"	66.4%)	Correspond to mol ratio
Phosphate as "P_2O_5"	16.9)	CuO/P_2O_5=7/1
Sodium as "Na_2O"	3.2	
Sulfate as "SO_3"	1.2	Chiefly "acid soluble".
Water as "H_2O"	6.8	By toluene distillation.
"Combined water" etc	5.5	By difference. The "loss on ignition" was slightly more than 12%.
	100.0	

The goal yield (analytical) given in the standard "B" formula is:

1527# (as is) ex. 3250# N-629.

The specific gravity of A-6-D is 3.7.

VARIABLE STUDIES:

The discussion of the mechanism by which A-6-D is formed will serve to indicate in a general way the effects of formulation and process variables on the final product. Any specific information is based on laboratory work; Semi-works and plant batches were made on substantially identical formulas.

The most important variable influencing composition and color is the amount of caustic used. A slight decrease (eg. 5#/mol $CuSO_4$) gives a lighter, greener and weaker product; a similar increase gives a darker, browner and stronger product. (In general, light products are greenish, dark ones are brown). This change is associated with the ratio of $CuO:P_2O_5$ in the product and is also reflected in the yield which decreases as the percentage of copper is increased.

Increased strike concentration gives lighter, weaker material.

Decreasing the strike temperature gives lighter, weaker material. If the temperature is increased above the standard figure (180°) there is a slight increase in depth. However, the difference between 180° and 200° samples is less than that between samples made at 160° and 180°. Development after the strike has considerably less effect than the actual strike temperature. For example, it is not possible by striking at a low temperature and heating subsequently to a boil to equal material struck hot.

Long stirring or prolonged washing tends to give slightly

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darker products, presumably as the result of increasing the amount of copper oxide in the pigment.

Increasing the strike time appears to have a similar effect to that of long stirring. A very rapid (dump) strike was unsatisfactory; apparently the conversion of basic phosphate to oxide was incomplete under these conditions.

Adding sodium phosphate and sodium hydroxide successively did not give the same results as the standard coprecipitation procedure. Indications were that the final product was a simple mixture of phosphate and oxide such as is obtained by slurrying these two substances together.

In most of the work, the standard amount of phosphate was used. Attempts to make the product with substantially lower amounts were not successful. However, the few experiments in which the phosphate was varied do not justify positive conclusions on this point. Trisodium phosphate can be substituted for disodium phosphate provided that a suitable adjustment in the amount of sodium hydroxide is made. Pyrophosphate gives a product quite different from A-6-D.

Experiments to compare direct and reverse precipitation gave inconsistent results. The standard procedure in which caustic/phosphate is added to copper sulfate is believed to be preferable.

Some further discussion of variables will be found in the formula notes on the "B" process.

EVALUATION AND SALES PROMOTIONAL WORK

A-6-D has been exposed in the following series. Unless otherwise noted, the vehicle was of the baking alkyd type used for automotive finishes:

S-10192 Gray metallics on miscellaneous products, including the first Semi-works batch of A-6-D (lot 1270), together with suitable controls. It showed excellent performance (12 mons. Florida). This series is discussed in the "Historical" section of this report.

S-10271 Includes lot 1270 (see above) as well as lot 1421 which was used in the preparation of Sales promotional exhibits, Masstones and Metallics. Performance good but gloss retention slightly worse than that shown in S-10192.

S-10264 This does not include A-6-D but compares copper oxide (A-4-D) samples made respectively by direct and reverse precipitation. These are quite different from each other in color but not markedly different in performance on exposure.

S-10289 Film fortifier study covering Cr_2O_3 and Fe_2O_3/CrO_3 and other products. A-6-D included as a control. Masstones.

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S-10290 Metallics corresponding to 10289.

S-10304 Sales Service Series. Pigment volume and aluminum ladders. Also includes tints.

S-10305 Sales Service Series. Metallics of blends of A-6-D with other durable pigments.

S-10313 Sales Service Series. Masstone and Metallics in 5 vehicle systems. % of aluminum varied.

S-10325 Evaluation of AGI-734-D. A-6-D included as a control.

S-10329 A-6-D included as a control in evaluation of CuO/Cr_2O_3 pigment (cf S-10258).

S-10339 Evaluation of experimental samples of various CuO/P_2O_5 ratios.

S-10342 Comparison of A-6-D and other products for color and film fortifying properties in Green and Yellow metallics.

S-10365 Sales Service Series. Evaluation of A-6-D in "Duco" and Rinshed Mason lacquer vehicle.

S-10392 Evaluation of official plant standard (89671/2).

S-10346 Comparison of A-6-D and other products in blue trim paints.

S-10397 Comparison with other gray pigments, viz. Fe_2O_3/Cr_2O_3 combinations and CuO/Cr_2O_3 (cf 10329). A first grade "Dulux" automotive gray metallic is included in this series.

A general discussion and summary of the studies carried out in the Sales Service Laboratory on the behavior of A-6-D in paint is presented in SSR 13-7 (A. MacDonald, 6/12/52). This covers hiding power measurements, grinding properties in alkyd and lacquer, viscosity and reactivity studies, tests in alkyd and lacquers for color stability (including sensitivity to water and citric acid), baking behavior, bleeding behavior and a rough cost comparison with "Rough Gold" in blue and green metallics. Reference should be made to this report for further details.

The results of standardization tests on Lot 89671/2 are presented in a letter, W. H. Avery - F. E. Bolton, 5/1/53.

A preliminary evaluation of A-6-D in house paint was made in S-11066 (White Pigments, Sales Service). Additional work is planned (see letter, Southard-Madson, 5/11/53). Indications were obtained that A-6-D may have some value as a mildew inhibitor.

A sample of A-6-D is under test to determine its value in anti-fouling paint (Pettit Paint Co).

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A sample was submitted to Grasselli for evaluations as a pesticide.

The Glidden Co. reported that A-6-D improved flow and reduced flooding in a dipping enamel. However, difficulties were encountered with film hardening. (see below.)

In general, the customer response to Sales promotion of A-6-D has been favorable. Possible deficiencbs are indicated below.

Grinding(Texture) - In our own work A-6-D has been found to disperse without difficulty. Some adverse comments have been received from the trade. These do not appear serious since further work has shown such complaints to be unjustified. Laboratory work has indicated that the texture can be improved somewhat by means of certain treating agents. Details have not been worked out.

Dusting and Toxicity - F & F reported that workers handling large amounts of A-6-D complained that it had an irritating effect. This has not been observed at Newark in our own work. Tests by the Haskell Laboratory confirmed the fact that undue exposure might result in serious irritation and it is planned to label the product accordingly. The precautions normally followed with irritating materials, that is the use of gloves and respirators should minimize this hazard.

Reactivity - Viscosity stability has been satisfactory in most vehicles. Occasional difficulties have been reported, however, in specific systems.

Film Hardness - Difficulties have been reported on drying properties by three customers - Rinshed Mason, Herbert Schorndorfer and Glidden. Studies in the Paint Laboratory (SSR 13-12) suggest that this may be due to copper in the product acting as a polymerisation inhibitor, rather than simply retarding oxidation of the film. As in the case of reactivity, the effect appears to be rather specific, varying with the vehicle system used. A change in oil length of the alkyd served to solve Rinshed Mason's problem. Attempts to correct the difficulty by changes in drier have, so far, been unsuccessful.

Gloss Retention - Indications have been obtained in recent exposure series that the gloss retention of A-6-D enamel films is not as good as had been believed from the results of early tests (S-10192). More data are necessary before positive conclusions can be drawn. Exposure series 10397 should give more definite information and suggest possible alternatives if A-6-D fails to be generally accepted.

IMPROVED PRODUCTS OF THE A-6-D TYPE

While it is too early to make a reliable forecast of the future of A-6-D, some consideration has been given to other products of similar color which have been made in this laboratory and which might be superior in one or more respects. Two of these deserve

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special mention, namely (1) combinations of hydrated iron and chromic oxides and (2) cupric/chromic oxide combinations. Preliminary results (S-10258, 10329, 10290) have indicated that these types which are similar in color to A-6-D may be superior in durability. Additional exposure tests are in progress which should make a definite conclusion possible.

While A-6-D was originally developed primarily for use in the production of grays, it has been found possible to use it in the production of novel colors; part of its value in such usage may lie in film fortifying properties. Whether it can compete with other pigments (F-4-P, F-13-D, A-33-D, etc.) in this field is still open to questions.

The $Fe_2O_3/Cr_2O_3/CuO$ pigment A-10-D showed excellent performance in S-10192 and other series. It lacks the relatively neutral color of A-6-D but might find application where the exact color of the latter is not essential.

ARGENT GRAY PULP A-7-P

Argent Gray when filtered gives a pulp of ca 30% solids content; this is coded as A-7-P.

Laboratory (spatula) flushouts indicate it to give results which are substantially identical with those obtained with the dry product. Flushed enamels were made by F & F in their early evaluation studies. Apparently no advantage was obtained by this technique since their later work was based entirely on the dry product.

An attempt was made to flush the pulp into alkyd resin in our small Semi-works W & P mixer. The experiment was unsuccessful in that a complete transfer was not obtained and the enamel was unsatisfactory. Due to the pressure of other items on the program, no further work was done to work out more suitable conditions.

A-7-P has been flushed into pyroxylic lacquer (2 roll procedure) without difficulty. No detailed comparison has been made with corresponding products made from A-6-D.

PROGRAM:

This can be considered as a final report on this problem, covering the work done in the Chemical Division under Research Project #272 as well as the work carried out to date by the Sales Service Laboratory to evaluate A-6-D.

No further work on A-6-D is planned aside from what may be done in an incidental way in the development and evaluation of similar products such as those mentioned under the section "Improved Products of the A-6-D Type".

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REFERENCES:

Preliminary Expts. on "Gray" pigments (Preparation & Evaluation)
(NB 1220-37, 46, 50, 60, 65, 69
(NB 1372-11, 13, 15, 16, 19, 22, 23, 24, 29, 31, 35, 36
37, 47, 50, 52, 56, 60
(NB 1389-15, 17, 20
The first preparation of A-6-D is 1220-50C

Experiments on Precipitated Cupric Oxide
NB 1220-26, 30, 49, 56

Experiments on Cu/Cr Oxide
NB 1389-39, 45, 64, 74, 75, 76
Expos. Series 10258, 10329, 10397

Semi-works and Plant Development - A-6-D
DSN 53-7
1372-42; 1389-58, 59, 62, 63, 73; 1441-69

Standardization
DSN 53-7

Laboratory Studies (Miscellaneous) - A-6-D
1389-71, 72, 80, 81, 82, 84, 85, 86, 87, 88
1441-1, 2, 3, 5, 7, 8, 9, 10, 13, 15, 18, 19, 21, 22, 24, 26, 31, 40

Analytical Data - A-6-D
1441-6, 9, 40, 69

Patent Studies (including X-ray diffraction studies) A-6-D
1389-77; 1441-4, 12, 16, 20, 71, 72, 73
See also correspondence in patent file

Texture - A-6-D
1441-70; 1466-1, 4

Flushing - A-6-D
1441-45

Evaluation and Application Studies
Exposure Work: Relevant series are listed earlier in this report
DSN 53-7
SSR 13-7

Toxicity
Correspondence with Haskell Laboratory (1953)
See also DSN-53-7

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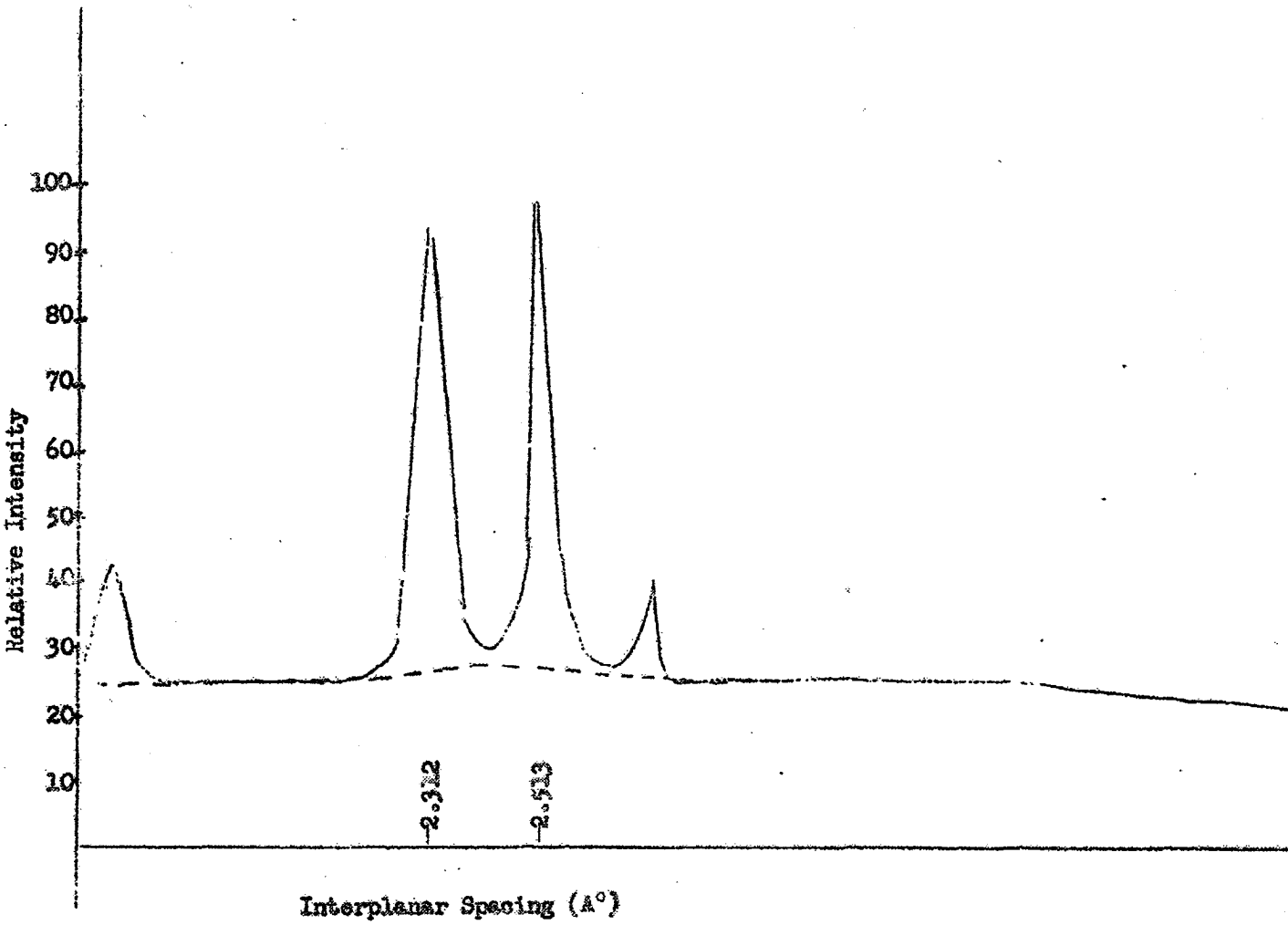

Figure 1 X-ray diffraction Curve of Cupric Oxide

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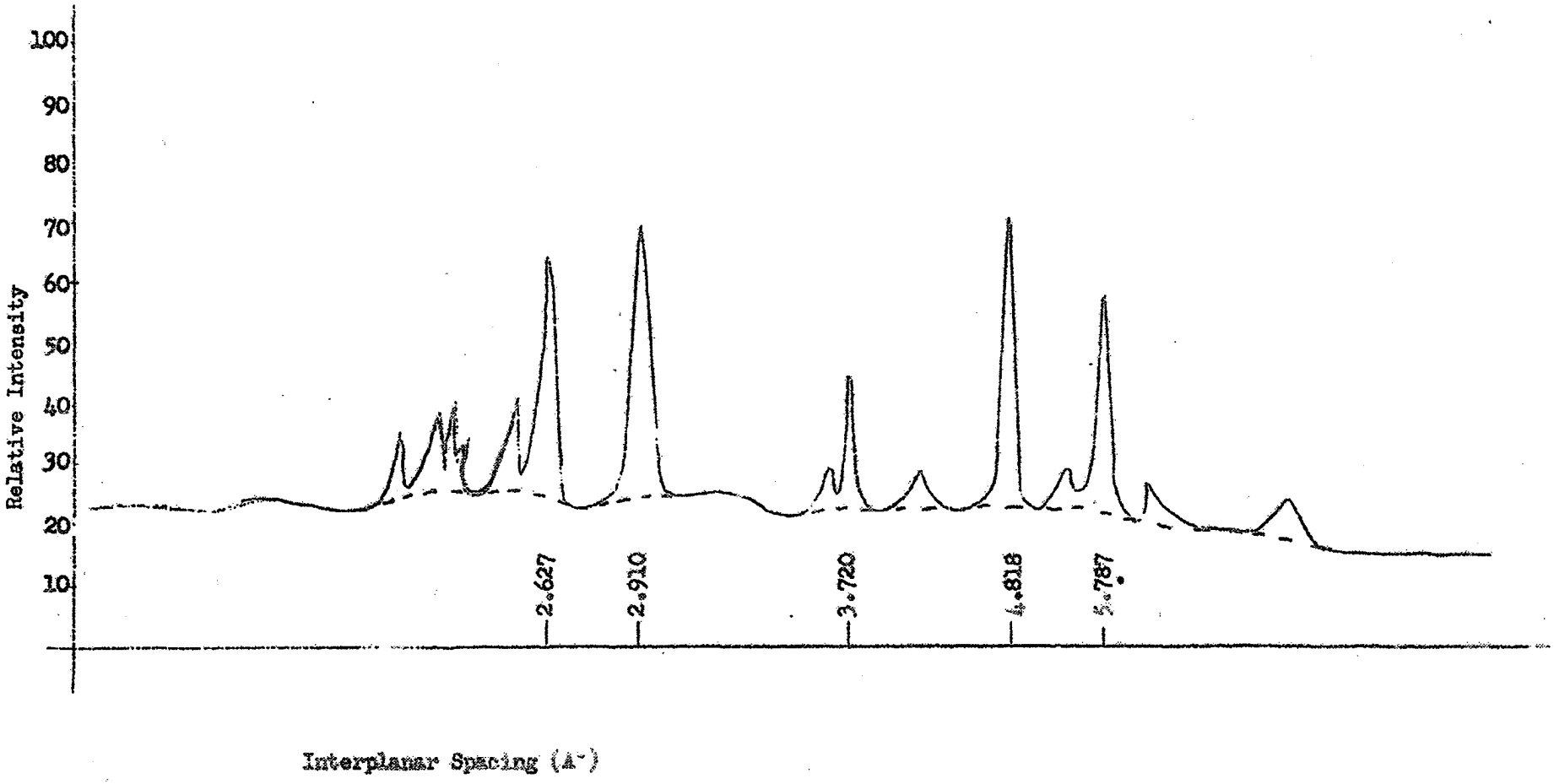

Figure 2 **X-ray diffraction Curve of Basic Copper Phosphate (CuO/P_2O_5=4/1)**

Solid line (———): Basic Copper Phosphate (Libethenite type)

Broken line (– – –): Background

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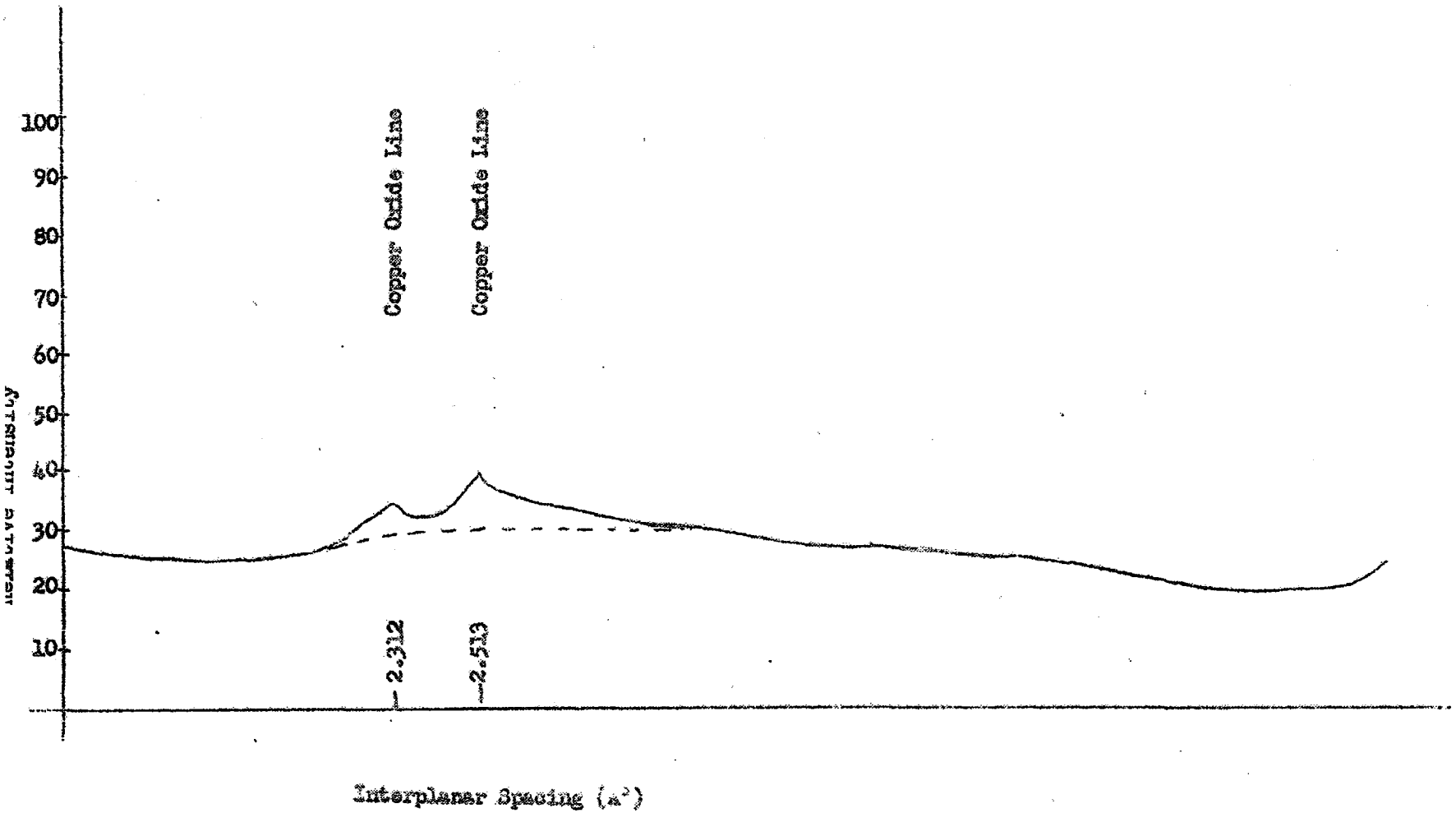

Figure 3 X-ray diffraction Curve of A-6-D

Solid Line: (———): Amorphous Product

Broken Line: (- - - -): Background

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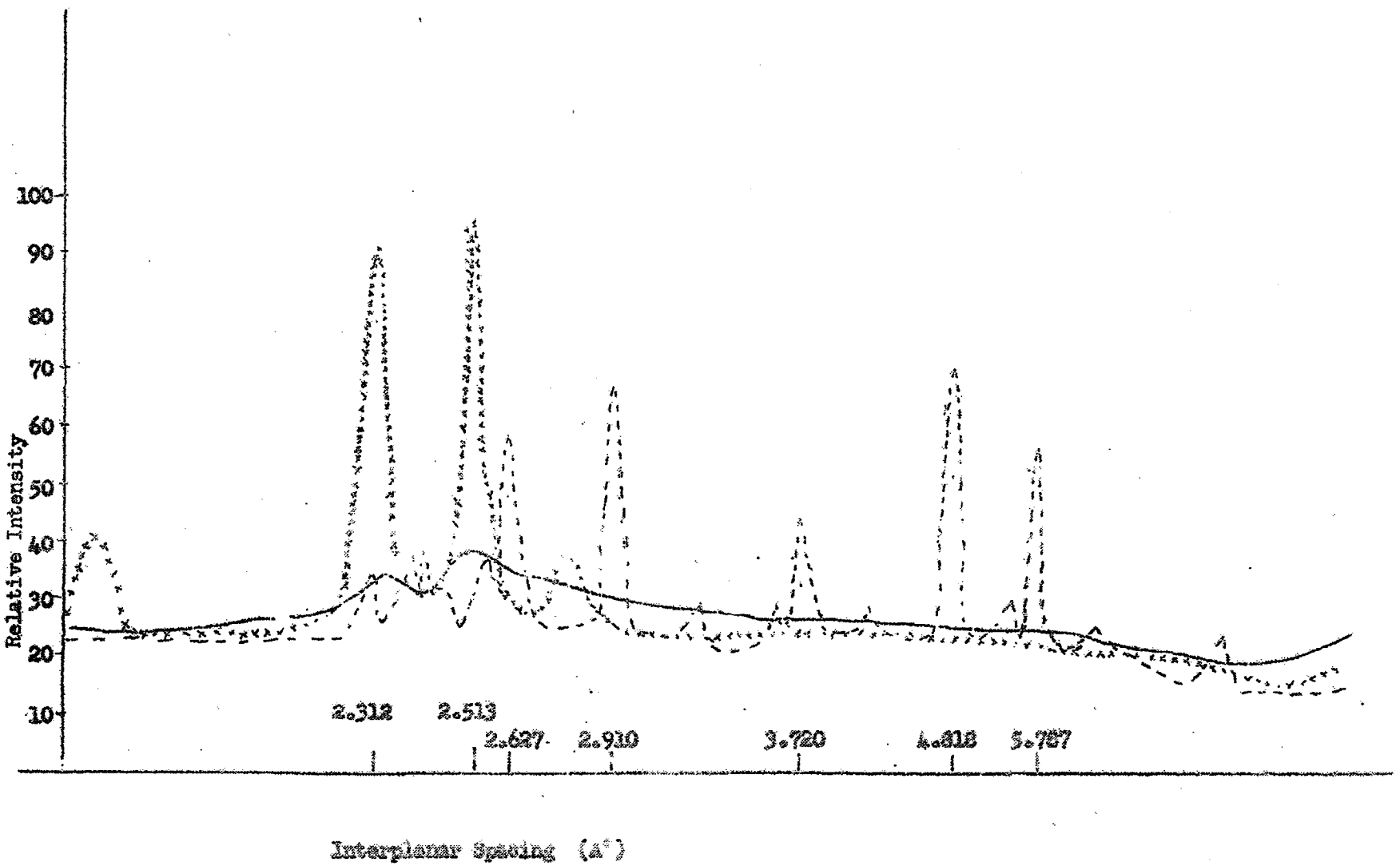

Figure 4 **Superimposed X-ray Diffraction Curves of Copper Oxide, Basic Copper Phosphate and A-6-D.**

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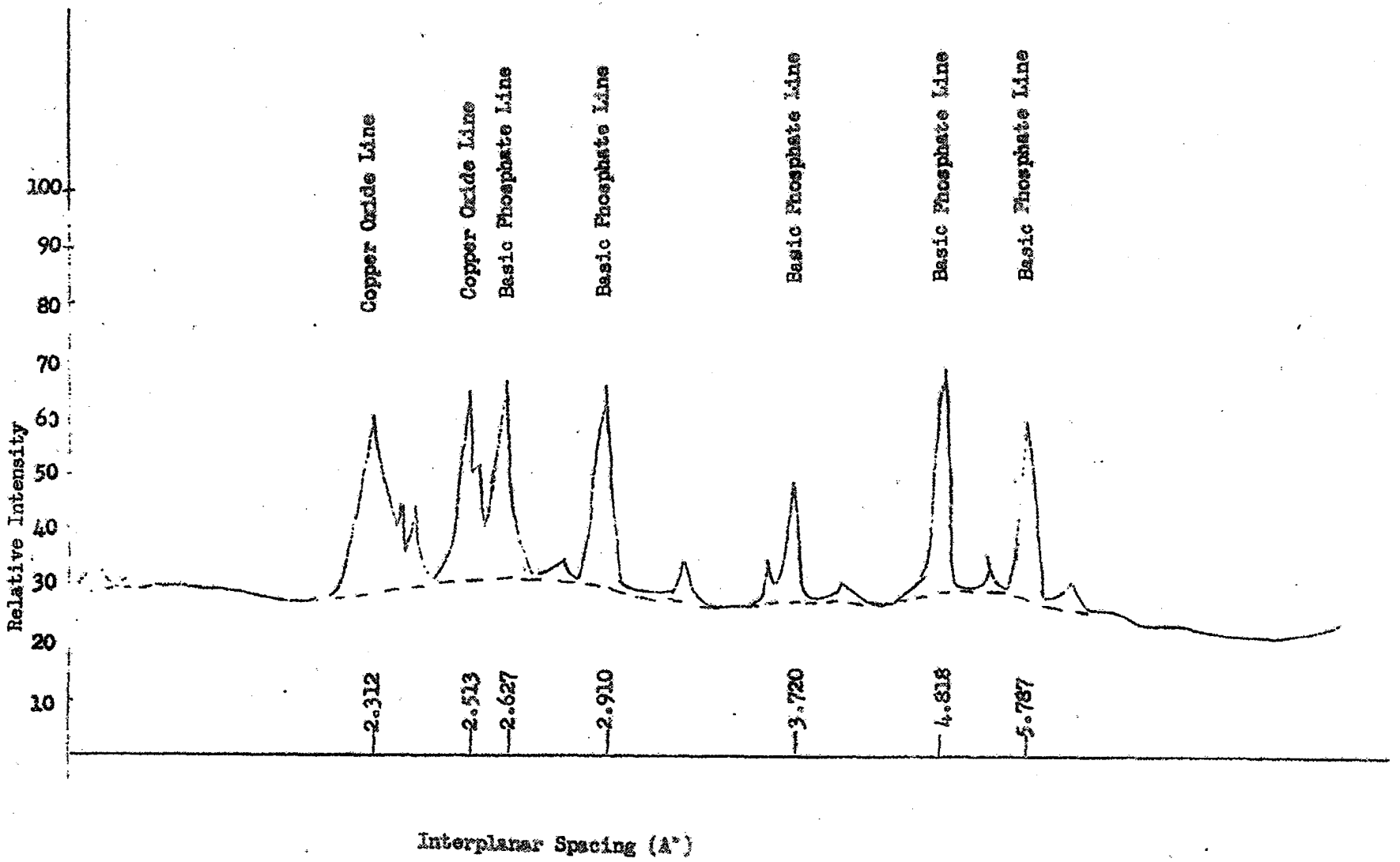

Solid Line(———): Physical Mixture of Cupric Oxide and Basic Copper Phosphate (Libethenite type)

Broken Line(— — — —): Background

Figure 5 **X-ray diffraction Curve of a Physical Mixture (slurries mixed and dried) of Copper Oxide and Basic Copper Phosphate.**

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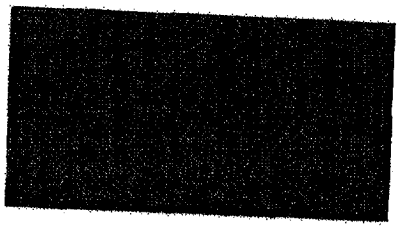
GRAY METALLICS - S-10192

Pigment Volume: 7% (Syntex 26/Resimene 881) } Chips 1-13
Pigment/Aluminum: 4/1 by volume }



(3) A-6-D

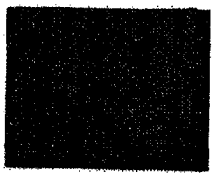
(1) A-4-D

(2) A-8-D

(4) A-10-D

(5)A-12-D

(6)A-14-D



(7)A-16-D

(8)A-20-D

(9)A-18-D

(10) Mapico Black

(11) Lamp Black

(12)R610/Lamp Black (70/30)

(13)R610/Lamp Black (85/15)

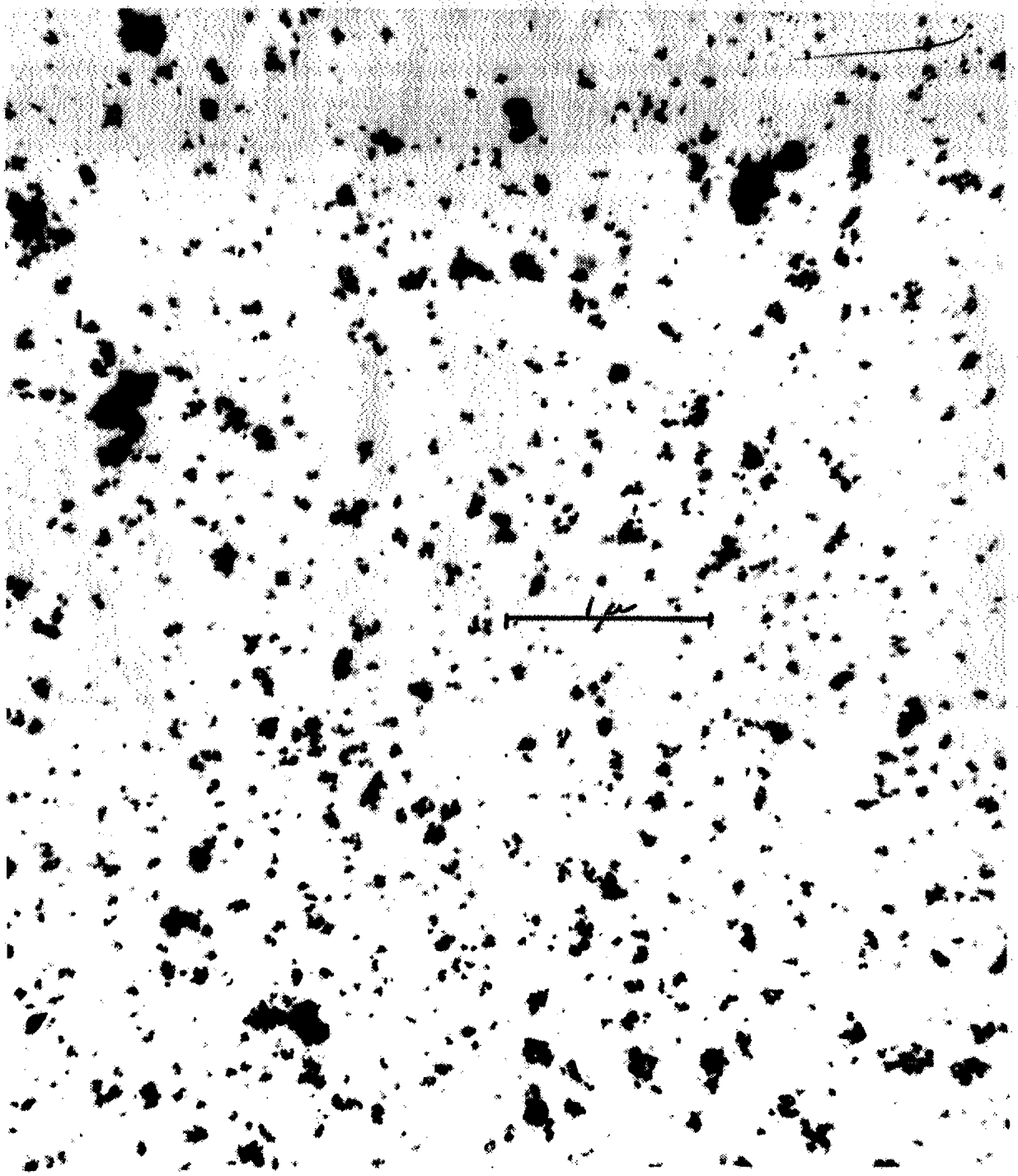
(14)Lima Tan
'49 Lincoln

(15) Summist Gray
'50 Buick

(16) Gunmetal
'50 Chrysler

DUP050069330

"ARGENT GRAY" A-6-D
Magnification 35000X



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