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OPTIMIZATION OF BINARY PIGMENT COATINGS

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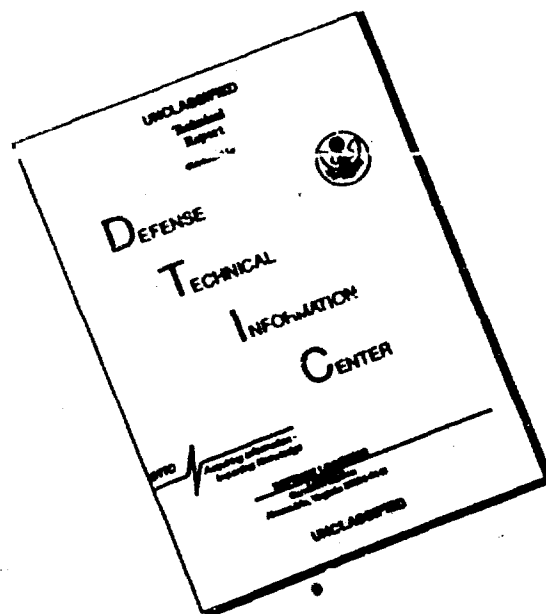
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Coating formulators are often faced with the task of determining pigment and resin concentrations of a coating which will provide the best combination of a number of specified properties. Usually this is performed via a cycle of formulating, testing, and analyzing coating systems. This 'trial and error' approach is inherently inefficient due to the high frequency of cycles required to produce the final composition. In this effort, an approach was devised and utilized to theoretically predict optimum coating compositions using two parameters, the maximum pigment packing factor and the coating critical pigment volume concentration, CPVC. Five binary pigment systems were evaluated in this study: 1. titanium dioxide - silicon dioxide 2. titanium dioxide - antimony oxide 3. titanium dioxide - zinc chromate (Continued on back)				
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4. titanium dioxide - vesiculated polymer beads
5. titanium dioxide - solid polymer beads,

Each pigment system was systematically formulated in a polyurethane resin using a simplex statistical design. After application and cure, the coatings were tested and evaluated for gloss and flexibility to experimentally determine the CPVC of the coating system. The correlation between the theoretical and experimental CPVC's was either good or fair for the five systems studies. These results indicate that theoretically predicting CPVC's along with a statistically designed formulation procedure will yield an optimum coating region for the given pigment system.

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INTRODUCTION

The objective of this project was to theoretically predict optimum coating formulations and to validate these predictions with a statistically designed experimental program. This approach could yield optimum coating formulations in a time efficient manner.

A pigmented organic coating or a paint is a thin layer of composite material which is used as a barrier to protect an underlying substrate from the surrounding environment and to decorate or camouflage the substrate. There are four major types of components in a coating: the pigment system, the resin, the solvents, and the additives. The pigment system can be a single pigment or a combination of pigments that are solid particles which can provide color and opacity, and improve strength, durability, and corrosion resistance of the coating. Pigment types vary greatly in chemical composition, and particle size and shape. They can be spherical, nodular, rectangular, platy, acicular or fibrous (1,2). The resin is a polymeric material which forms a continuous matrix holding the pigment particles and adhering to the substrate. The resin can be a thermoset or thermoplastic polymer and is referred to as the binder. The solvents are volatile materials that allow thorough milling and dispersion of the pigments and application of the coating. The additives are primarily incorporated into the resin for plasticization, adhesion promotion, catalysis, defoaming, and rheology modification.

A paint can consist of only two components or as many as fifteen or more. Determining the optimum concentration of these components is tedious and time consuming. A common method involves an experimental cycle consisting of preparing, applying, curing, testing, and evaluating candidate coating formulations (3). This cycle is repeated until a coating is attained that has the optimum combination of desired properties. Four or more 'trial and error' cycles are usually required to determine the optimum coating. With the amount of time required for one cycle normally being over two weeks, and possibly much longer with long term testing, this entire process is inefficient with respect to time, material, and labor.

By predicting optimum coating concentrations and subsequently analyzing statistically determined formulations, the coating development process time can be significantly reduced. In this effort, two simultaneous approaches were performed: a theoretical approach to predict optimum resin/pigment concentrations and an experimental approach to validate these predictions.

THEORETICAL APPROACH

THEORY

The theoretical method for determining the optimum pigment concentration utilizes two physical coating parameters, the maximum packing factor ($\Phi_{\max}$) of the pigment system and the critical pigment volume concentration (CPVC) of the coating.

Packing Factor: Packing factor, Φ , is the fractional volume of pigment in a unit volume of space or, in other words, the true volume occupied by distinct particles per bulk volume of those particles. It is an indication of the volume packing efficiency of the particles. Empirical equations for calculating packing factors of variable-sized spheres were developed by Lee (4) based on experimental volume measurements of dense random arrangements of mono-sized and binary-sized spheres. The maximum packing factor, $\Phi_{\max}$, of mono-sized spheres was experimentally determined to be 0.640. The packing factors for a wide range of binary-sized spheres (diameter ratios of large to small spheres ranging from 1 to 20) also were experimentally determined and graphically plotted. $\Phi_{\max}$ for binary-sized spheres with a diameter ratio of 20 was determined to be 0.850. This is due to the smaller spheres filling the interstices created by the larger spheres. As this diameter ratio approaches infinity, the packing factor approaches 0.870. Lee's algorithm is presented in Appendix A. By converting Lee's algorithm into a computer program (5) and providing the particle size distribution of the desired pigment(s), the packing factor of various pigment mixtures can be calculated.

Patton illustrated that as pigment concentration increases, a coating property inflection or optimum occurs at the point where the coating becomes resin starved (1). McCullough and Quigley showed that a minimum electrical resistivity inflection occurs at the maximum packing factor filler concentration (6). Janowski and Rossi illustrated that the maximum elastic modulus and strength occur at the maximum packing factor (7). Therefore, since the maximum packing factor of pigments yields the highest potential loading level, the pigment mixture concentrations at the $\Phi_{\max}$ are likely to produce an optimum combination of optical and physical properties.

Pigment Volume Concentration (PVC): Pigment volume concentration is the fractional amount of pigment in the total solid content of a coating. A mathematical representation of PVC is:

$$PVC = \frac{V_{\text{pig}}}{V_{\text{pig}} + V_{\text{res}}} \quad (1)$$

where V_{pig} and V_{res} are the volumes of pigment and resin, respectively. This ratio of pigment to resin has a significant effect on the final coating properties. For example, when a resin is added to a packed bed of pigment particles, the resin initially fills the interstices of the pigment, producing a pigment rich, resin starved coating with air voids and poor strength, flexibility, adhesion, and durability. As the resin concentration is increased, a specific composition is reached where all of the air which initially filled the interstices is displaced by resin. At this point there is just enough resin to wet the pigment particles and fill the interstices. This specific composition is known as the critical pigment volume concentration (CPVC). Further addition of resin forces the pigment particles to be displaced. Patton experimentally showed that a significant effect in the major paint properties occurs at the CPVC (1). These effects are depicted in Figure 1. The porosity graph in Figure 1 illustrates the above concept of adding resin to a bed of pigment particles with a higher density than the resin. CPVC can be represented by the following equation:

$$CPVC = \frac{V_{\text{pig}}}{V_{\text{pig}} + V_{\text{res-min}}} \quad (2)$$

where $V_{\text{res-min}}$ is the minimum volume of resin necessary to wet the pigment and fill the interstices.

CPVC is closely related to a common pigment property, oil absorption. Oil absorption, OA, refers to the amount of linseed oil required to form a paste-like consistency when combined with 100 grams of a pigment sample. A common oil absorption procedure is described in ASTM method D281. The oil absorption value is expressed in grams of linseed oil per 100 grams of pigment. In forming the paste, the linseed oil is absorbed, adsorbed, and interstitially bound to the pigment particles. The amount of absorbed oil is dependent upon the chemical and physical nature of the pigment particles, while the amount of adsorbed oil is determined by the surface chemistry properties of the particles and oil. Note that the adsorbed oil layer changes the particle size and will have an effect on the packing factor. The amount of interstitial oil is directly related to the packing factor in that this volume is equal to $(1-\Phi)$. The OA endpoint signifies the minimum amount of oil necessary to wet the pigment particles and fill the interstitial volume $(1-\Phi)$ between the particles. In effect, this is the same concept as the CPVC except that oil absorption is standardized with linseed oil as the pigment binder and CPVC can be obtained with any binder. The mathematical expression relating CPVC to oil absorption is:

$$CPVC = \frac{1}{1 + OA} \quad (3)$$

where OA in this equation is represented in milliliters of linseed oil per milliliter of pigment. See Appendix B for the derivation of equation (3). Note that for individual pigments, the OA value used in equation (3) is simply that for the pigment in question. However, if pigment mixtures are to be used, which is normally the case, the OA of the specific pigment mixture must be used. Appendix B also describes the computational approach to theoretically predict CPVC's of pigment mixtures. Appendix C is a listing of the computer program used to calculate CPVC. This program calculates the pigment packing factor which is utilized in the determination of CPVC values. The required input data for this program consists of the oil absorption value, particle size distribution, and the volume fraction of each pigment in the pigment system. Utilizing this program, maximum packing factors and critical pigment volume concentrations were determined for the five pigment systems analyzed.

PIGMENT CHARACTERIZATION

Three pigment systems were evaluated in this study:

1. Titanium dioxide (TiO_2) - diatomaceous silicon dioxide (SiO_2)
2. Titanium dioxide - antimony oxide (Sb_2O_3)
3. Titanium dioxide - zinc chromate (ZnCrO_4)

Two other pigment systems were similarly evaluated in an associated study (5):

4. Titanium dioxide - titanium dioxide vesiculated beads (TiVsBd)
5. Titanium dioxide - titanium dioxide solid beads (TiSiBd)

These pigments were chosen because they represent a wide variety of commonly used pigments and fillers. Rutile titanium dioxide is a widely used pigment for formulating white opaque materials. Diatomaceous silica is a commonly used extender pigment which is often mixed with titanium dioxide. Antimony oxide is a common fire-retardant pigment and zinc chromate is a corrosion inhibiting pigment used in primers. Polymer beads are a relatively new class of pigments. They are currently used in trade sales paints; but their use in industrial coatings, including aircraft paints, is increasing. Reference (5) provides a detailed description of the TiVsBd and TiSiBd pigments and concepts for using them in paint formulation.

Scanning electron micrographs (SEM's) of the above pigments are shown in Figures 2 to 7. These SEM's were used to obtain particle size distributions with a computer program which measures and categorizes pigment particle diameters. Particle size distributions for the pigments investigated are illustrated in Figures 8 to 13.

Oil absorption values were determined per ASTM method D281. The linseed oil has an acid value of 1.15 and a density of 0.929 g/ml. The OA values along with other pigment properties are listed in Table I.

Φ AND CPVC PREDICTIONS

Figure 14 is a graph of packing factor versus pigment composition for the five pigment systems investigated. The zinc chromate and antimony oxide systems produce flatter graphs, not varying as much in packing factor than the other three systems. Their packing factors remain around 0.64 to 0.66 which agrees with data obtained by Lee (4) where binary pigment mixtures with a diameter ratio of 1 yield a maximum packing factor of 0.64. This is low relative to the highest possible Φ_{max} of 0.87 discussed previously and is due to the particle size of ZnCrO_4 and Sb_2O_3 being closer to that of TiO_2 . TiVsBd , TiSiBd , and SiO_2 , when

mixed with TiO_2 , can attain Φ_{max} of 0.84, 0.84, and 0.78, respectively. These pigments have particle diameters which are an order of magnitude larger than TiO_2 . In the systems with the larger particles, the titanium dioxide can fit into the interstices of the larger particles, packing with better efficiency and causing a larger Φ . The maximum packing factor of these systems occurs around 30 to 35% TiO_2 . Deviating from this composition, the particles do not pack as efficiently and the packing factor decreases.

A convenient method of analyzing the data of two pigments in a binder system is by using a triangular coordinate graph with the two pigments and the binder as the apexes of this equilateral triangle (on a volume basis). This graph represents all of the possible combinations of the three components and the property effects can be plotted versus composition. Figures 15 to 19 illustrate the theoretical CPVC's and Φ_{max} for the five pigment systems analyzed. The straight line intersecting the CPVC line represents the ratio of pigments which produces the maximum packing factor.

The slope of the CPVC lines in Figures 15 to 19 can be directly attributed to the oil absorption characteristics of the pigment being combined with TiO_2 . SiO_2 and TiVsBd have high oil absorption values and as they are added to TiO_2 in a resin system, the CPVC decreases. On the graphs, this is seen as an increase in resin content at the CPVC. Conversely, Sb_2O_3 , ZnCrO_4 , and TiSiBd have low oil absorption values which causes a higher CPVC. The intersection of the Φ_{max} and CPVC lines is theoretically the highest filler loading possible without creating a resin starved system. A statistically designed testing and analysis program can be based on these predictions.

The CPVC lines in Figures 15 to 19 are slightly concave up. This is due to the more efficient packing of pigment mixtures. As the pigment packs more efficiently and the interstitial volume decreases, less resin is required to reach the critical pigment volume concentration, causing the concave up graph. However, pigment oil absorption has a more significant and over-riding effect on CPVC.

EXPERIMENTAL APPROACH

COATING PREPARATION AND EXPERIMENTAL PROCEDURES

The resin system used in this study was a two component, aliphatic polyurethane (Table II). More specifically, this polyurethane was obtained by reacting hexamethylene diisocyanate, HMDI (Desmodur N-75, Mobay Chemical Company) with a polyester diol (Desmophen 651A-65 and 670-90, Mobay Chemical Company). Polyurethane resins vary in properties. However, this polyurethane resin is known to exhibit good chemical and weather resistance and flexibility characteristics. It meets the physical requirements for Mil-C-83286 which is a specification for an exterior topcoat used on Navy, Marine Corps, and Air Force aircraft.

A statistical distribution of candidate coating formulations was determined by using a simplex screening design (8). This provided seven compositions for each coating system as shown on the triangular coordinate graph in Figure 20. From this model, the specified concentrations of pigments and diol (Component I) of the polyurethane resin were milled in a quart jar, half-filled with 5mm diameter glass beads on a paint shaker, for 30 minutes. Then, the diisocyanate (component II) was added and agitated for one minute. The formulations were allowed to set for 15 minutes so that the resin component reaction could initiate, forming the desired polyurethane.

The formulated coatings were applied to 3 x 6 x 0.02 inch (7.62 x 15.24 x 0.05 cm) aluminum test panels by a conventional air-atomizing spray technique. The aluminum panels were 2024 - O temper, anodized in accordance with Mil-A-8625, Type I. The coatings were allowed to cure for one week at

ambient laboratory conditions prior to testing. They were evaluated for 60° gloss (ASTM D523), Gardner impact flexibility (ASTM D2784), and General Electric impact flexibility (Method 6226 of Federal Test Method Standard 141C).

After the initial results were analyzed, a new set of formulations was statistically determined in the optimum formulation region and the experimental procedures were repeated to locate the optimum formulation for each system.

EXPERIMENTAL RESULTS

The experimental results of the 60 degree gloss, G.E. impact flexibility, and direct and reverse Gardner impact flexibility for all the systems analyzed are provided in Figures 21 to 33. The contour lines were obtained by analyzing the raw data and plotting representative property value contour lines as a function of coating composition represented on the triangular coordinate graph.

Figure 21 to 33 illustrate that gloss and flexibility decrease as pigment concentration increases. Gloss is the specular reflection of light at an angle equal to the angle of incident radiation. As the pigment concentration of a coating increases, the particles begin to fill the matrix and protrude through the surface, scattering the light and reducing gloss. In addition, the gloss results confirm that high oil absorption pigments such as silica and TiVSBd reduce gloss more efficiently than low oil absorption pigments because they require less resin to obtain a pigment rich coating, forcing the pigment to reach the surface and protrude through it.

Impact flexibility decreases as pigment concentration increases due to the inclusion of rigid filler particles into the flexible, tough polyurethane matrix. The pigment particles provide sites for crack initiation and decrease the flexibility properties of the matrix, especially when incorporated at high concentrations (9).

The experimental CPVC of the systems tested were determined using Figures 21 to 33. CPVC was observed as a leveling off of gloss and flexibility, as is illustrated in the gloss graph of Figure 1. Although increased pigment concentration reduces coating gloss and flexibility, the mechanisms which cause gloss reduction and coating cracking are different. Therefore, experimental CPVC's determined by these methods are slightly different. Figures 34 through 38 illustrate the experimental and theoretical CPVC's for the five pigment systems tested. In general, CPVC determined from gloss data is lower than that determined from flexibility data. As predicted by equation (3), the CPVC is inversely proportional to the oil absorption of the pigment system.

DISCUSSION

The objective of this effort was to theoretically predict optimum coating formulations and to validate these predictions with a statistically designed experimental program. By achieving this objective, the effectiveness of predicting CPVC will be demonstrated. Figures 34 to 38 graphically compare theoretical and experimental CPVC's for the five pigment systems analyzed. In general, theoretical CPVC's are in the same coating composition region as the experimental CPVC's. This is especially true for the SiO_2 , Sb_2O_3 , and ZnCrO_4 systems. However, several discrepancies are evident. The polymer bead systems do not agree as well as the inorganic pigment systems. This may be due to the unique nature of these polymer bead pigments and their interaction with the polyurethane matrix.

In most cases, the experimental CPVC's are lower than the theoretical CPVC's. This is especially true for low oil absorption pigment systems ($\text{TiO}_2/\text{Sb}_2\text{O}_3$ and $\text{TiO}_2/\text{TiSiBd}$). This effect is probably due to the large concentration required for low oil absorption pigments. When large pigment concentrations are present, introduction of defects such as pigment clumping and air-voids into the coating is more likely. As stated by Patton (1), 'Latices tend to resist penetration into the innermost voids or fail to adequately wet the pigment surface'. Although the paint contains enough binder to fill the pigment interstices, a resin starved-like system

is obtained. Dobkowski also observed lower theoretical CPVC's than experimental (85° gloss) CPVC's with calcined kaolin pigments (10).

Although the shape of the theoretical CPVC graphs is concave up, several of the experimental curves are concave down. Another reason for discrepancies between predicted and experimental values is the use of the oil absorption value to predict CPVC. Theoretical CPVC is determined with linseed oil while the coatings were formulated with a polyurethane resin. The chemical interaction between pigment and binder has an effect on CPVC; and, therefore, pigments may exhibit different CPVC's in chemically different binder systems. A second contribution may be that, although consistent oil absorption values were obtained for all the pigments analyzed, determining the exact endpoint can vary between operators. Therefore, the oil absorption may be slightly different than the experimentally recognized CPVC.

When developing and formulating a paint, a large number of components are usually required. Obtaining the optimum concentration of these components which will yield the desired properties can be tedious and time consuming. By theoretically predicting the critical pigment volume concentration, the wide range of potential formulation compositions can be significantly reduced. By subsequently using a statistically designed formulation plan in the suspected composition region, the formulator can confidently obtain the optimum pigment/resin formulation in a time efficient manner.

CONCLUSIONS

Critical pigment volume concentrations can be theoretically predicted using pigment oil absorption and packing factor data. These predictions agree well with experimentally determined CPVC's. Using theoretically predicted CPVC's to narrow the potential pigment/binder concentrations, along with a statistically designed formulation analysis, can yield optimum coating formulations in a time efficient manner.

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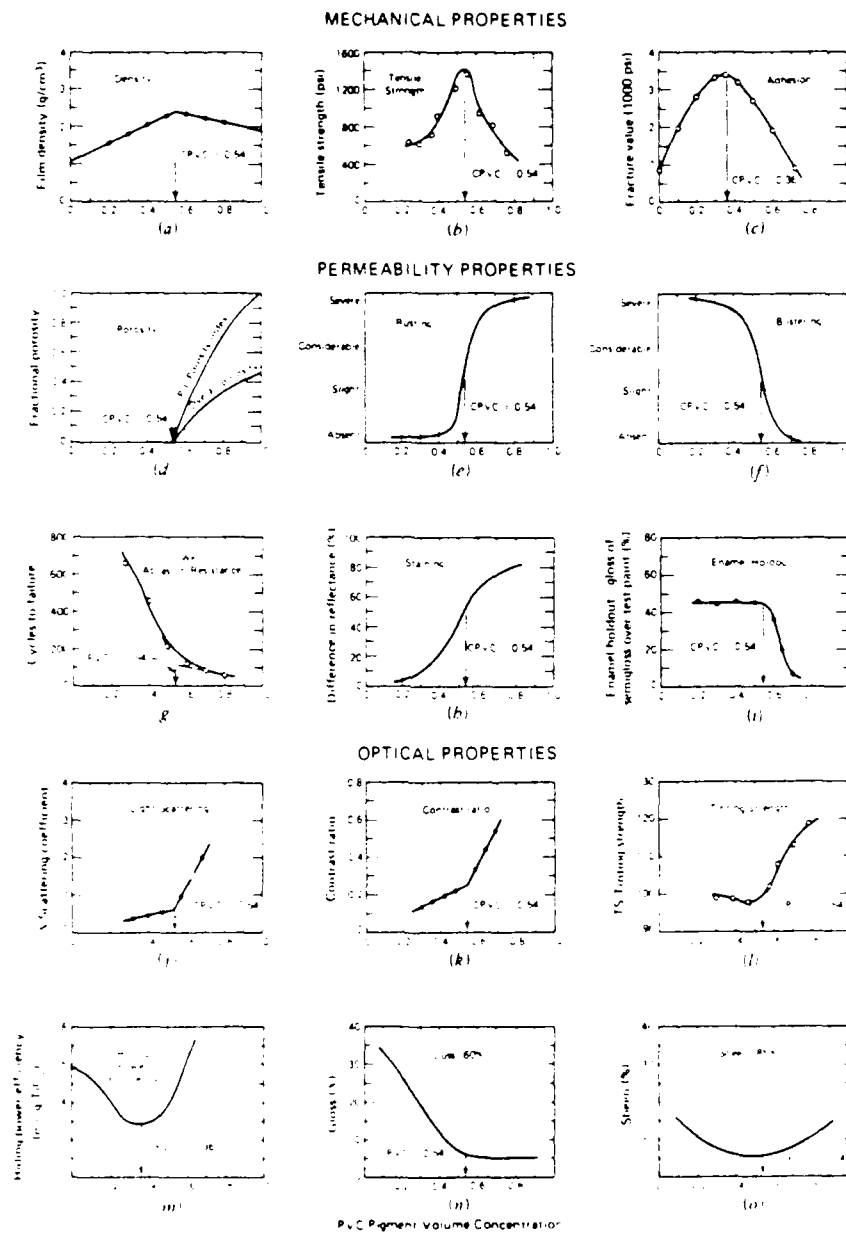


Figure 1: Effect of Pigment Volume Concentration on Coating Properties (1).

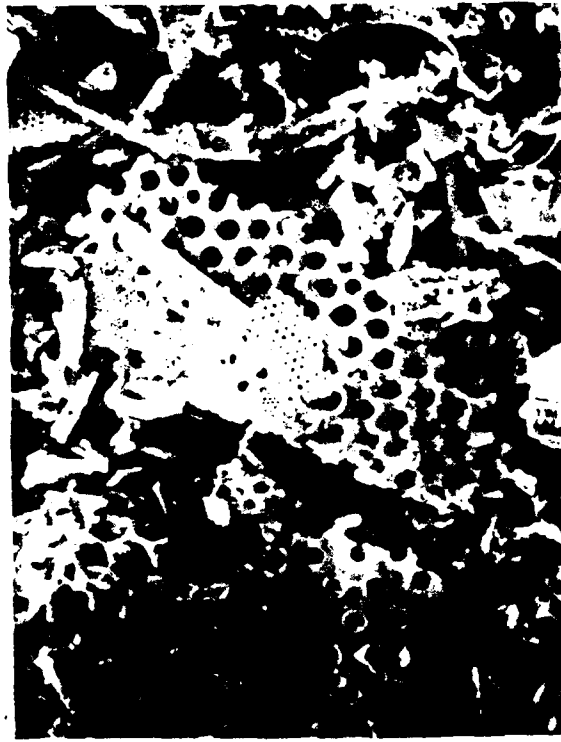


Figure 3: Scanning Electron Micrograph of Diatomaceous SiO₂ at 2,000 X.

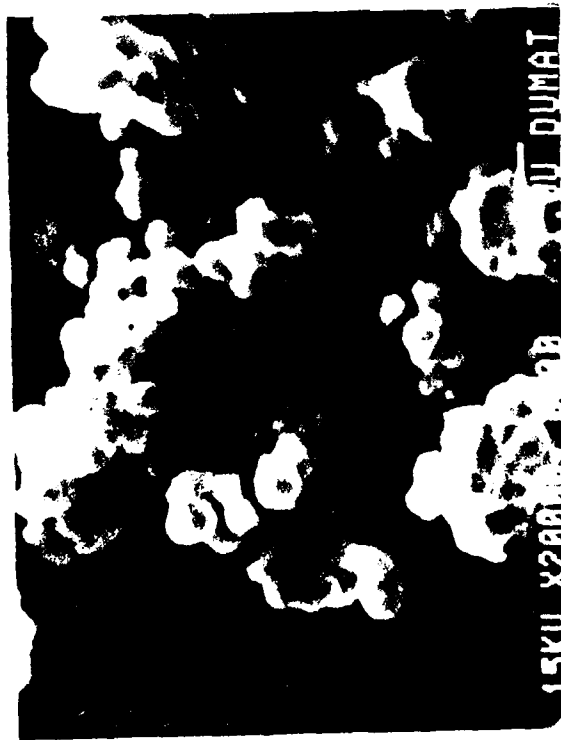


Figure 2: Scanning Electron Micrograph of TiO₂ at 20,000 X.



Figure 5: Scanning Electron Micrograph of ZnCrO_4 at 20,000 X.



Figure 4: Scanning Electron Micrograph of Sb_2O_3 at 20,000 X.



Figure 6: Scanning Electron Micrograph of Titanium Dioxide Vesiculated Beads at 2,000 X. (5)

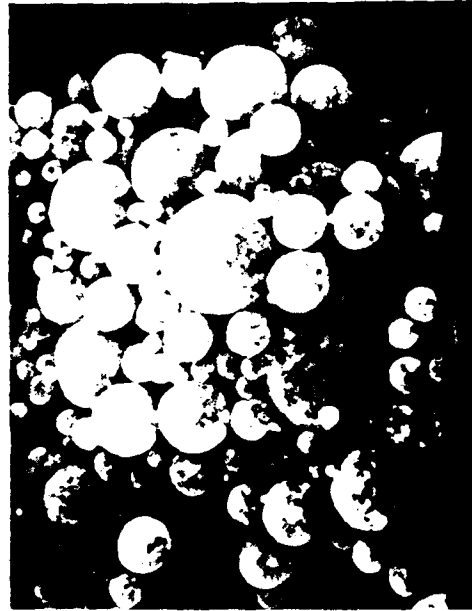
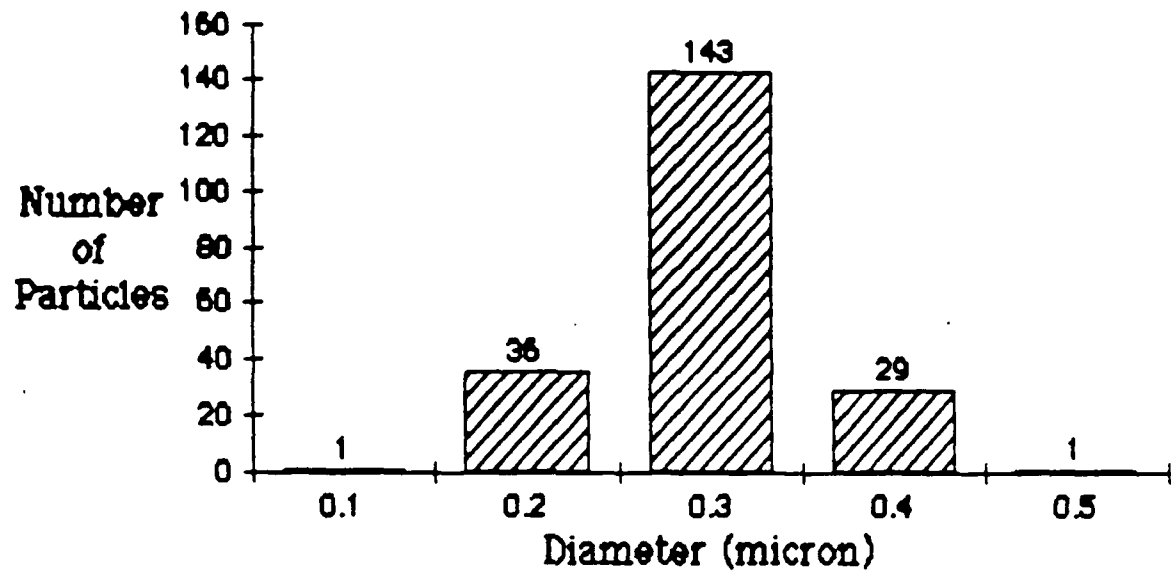


Figure 7: Scanning Electron Micrograph of Titanium Dioxide Solid Beads at 2,000 X. (5)

Figure 8: Particle Size Distribution of TiO_2

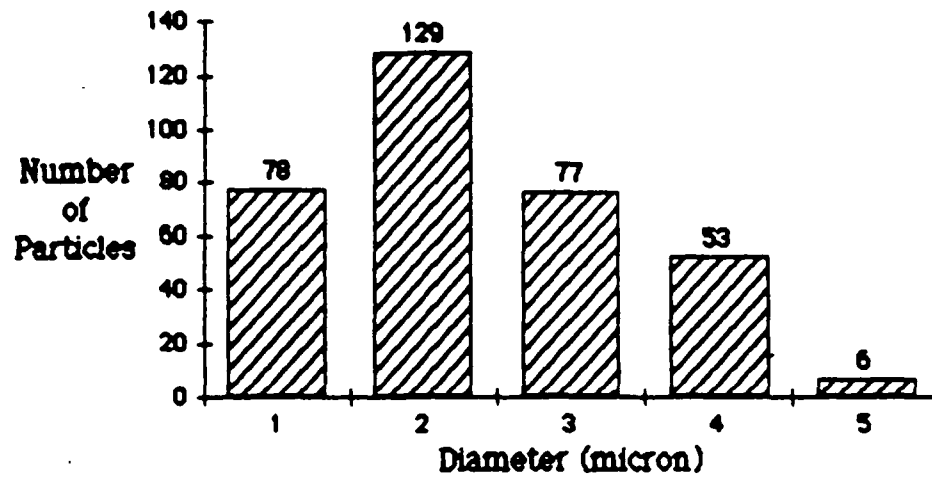


Figure 9: Particle Size Distribution of SiO₂

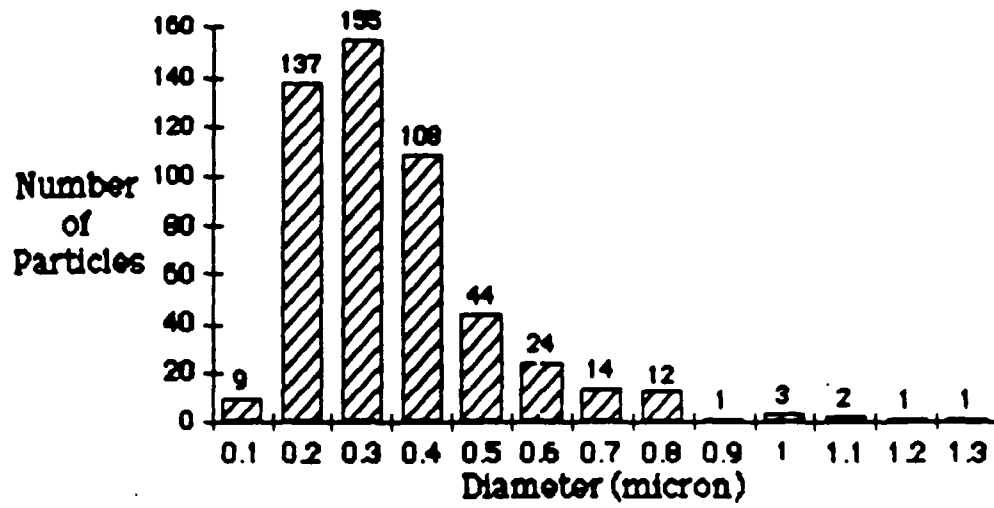


Figure 10: Particle Size Distribution of Sb_2O_3

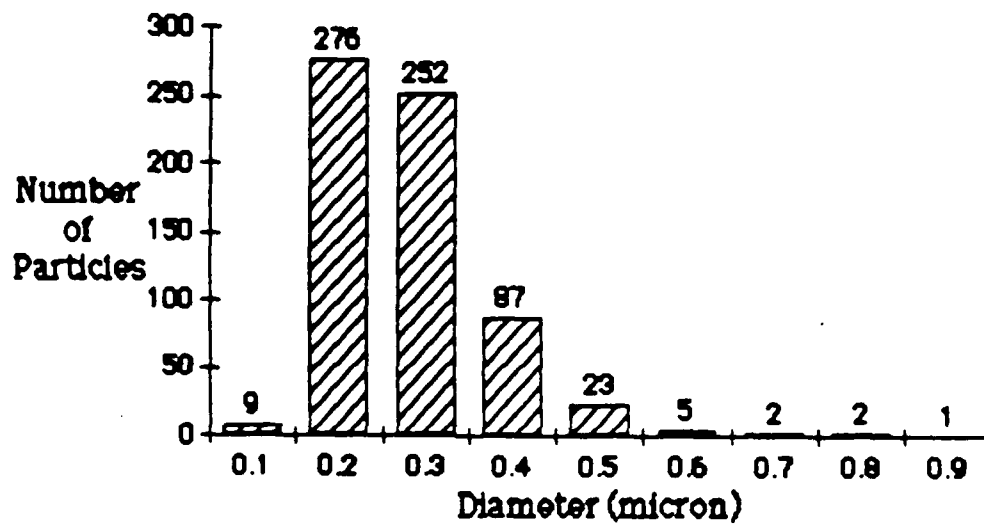


Figure 11: Particle Size Distribution of ZnCrO_4

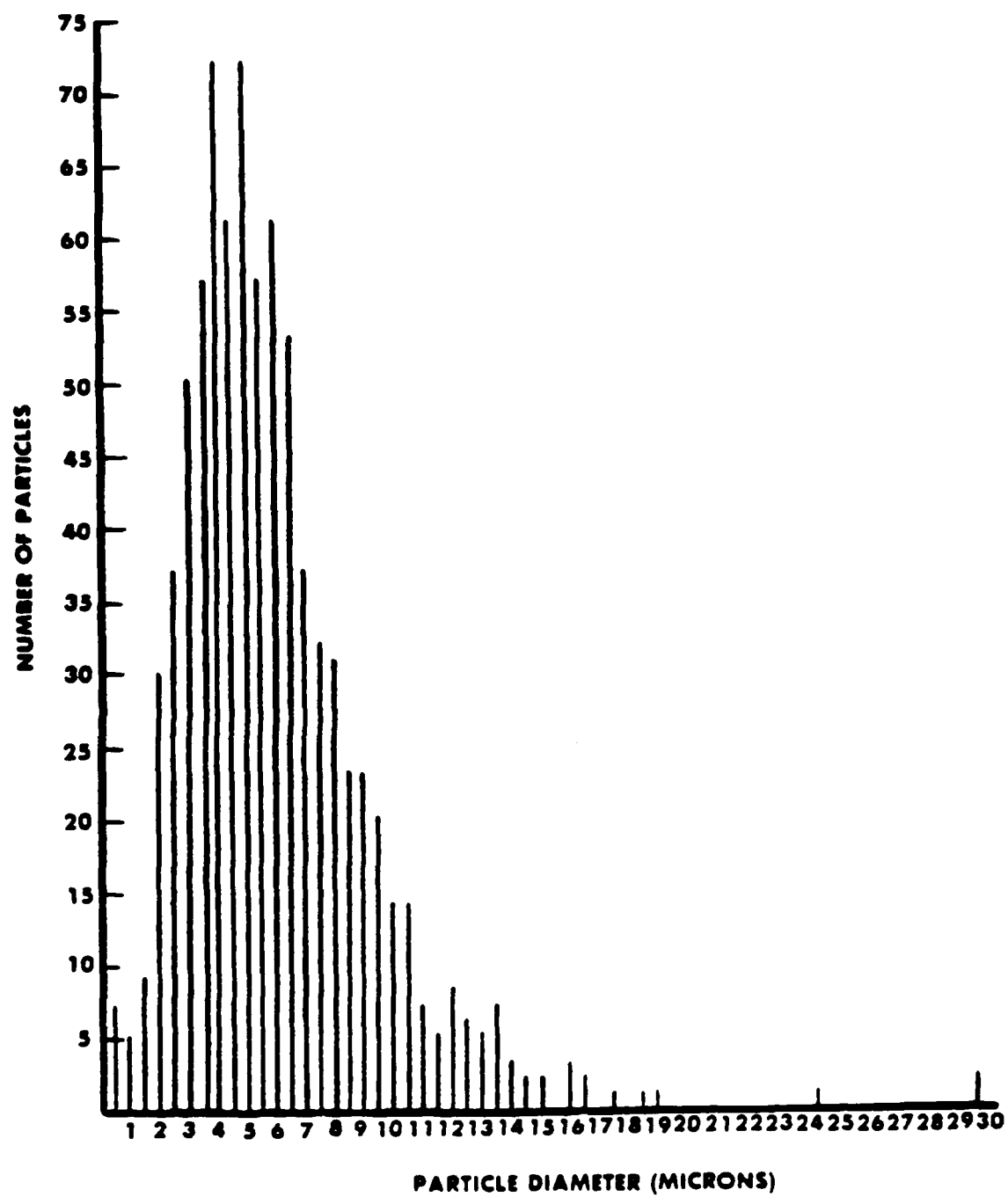


Figure 12: Particle Size Distribution of Titanium Dioxide Vesiculated Beads (5).

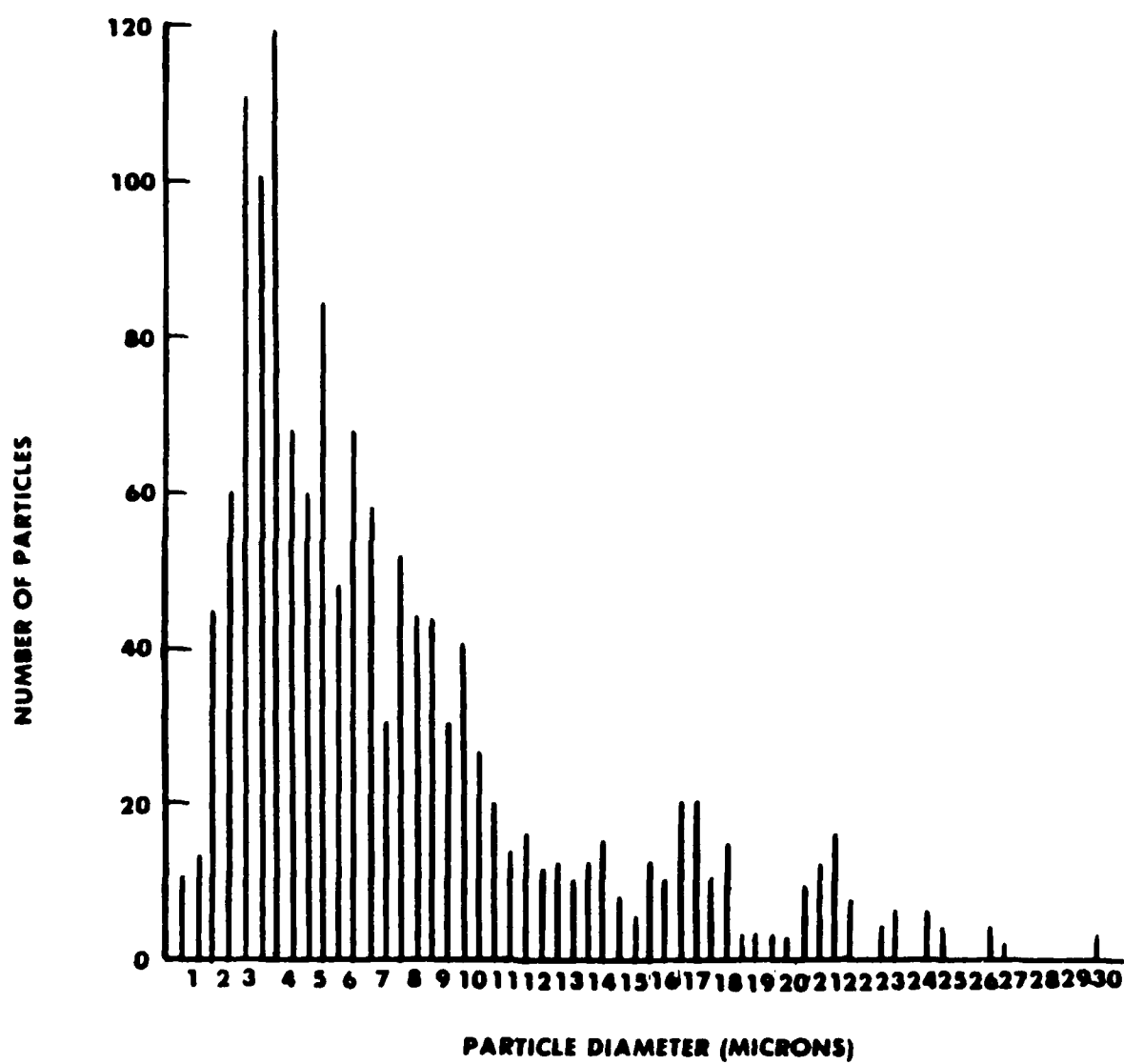


Figure 13: Particle Size Distribution of Titanium Dioxide Solid Beads (5).

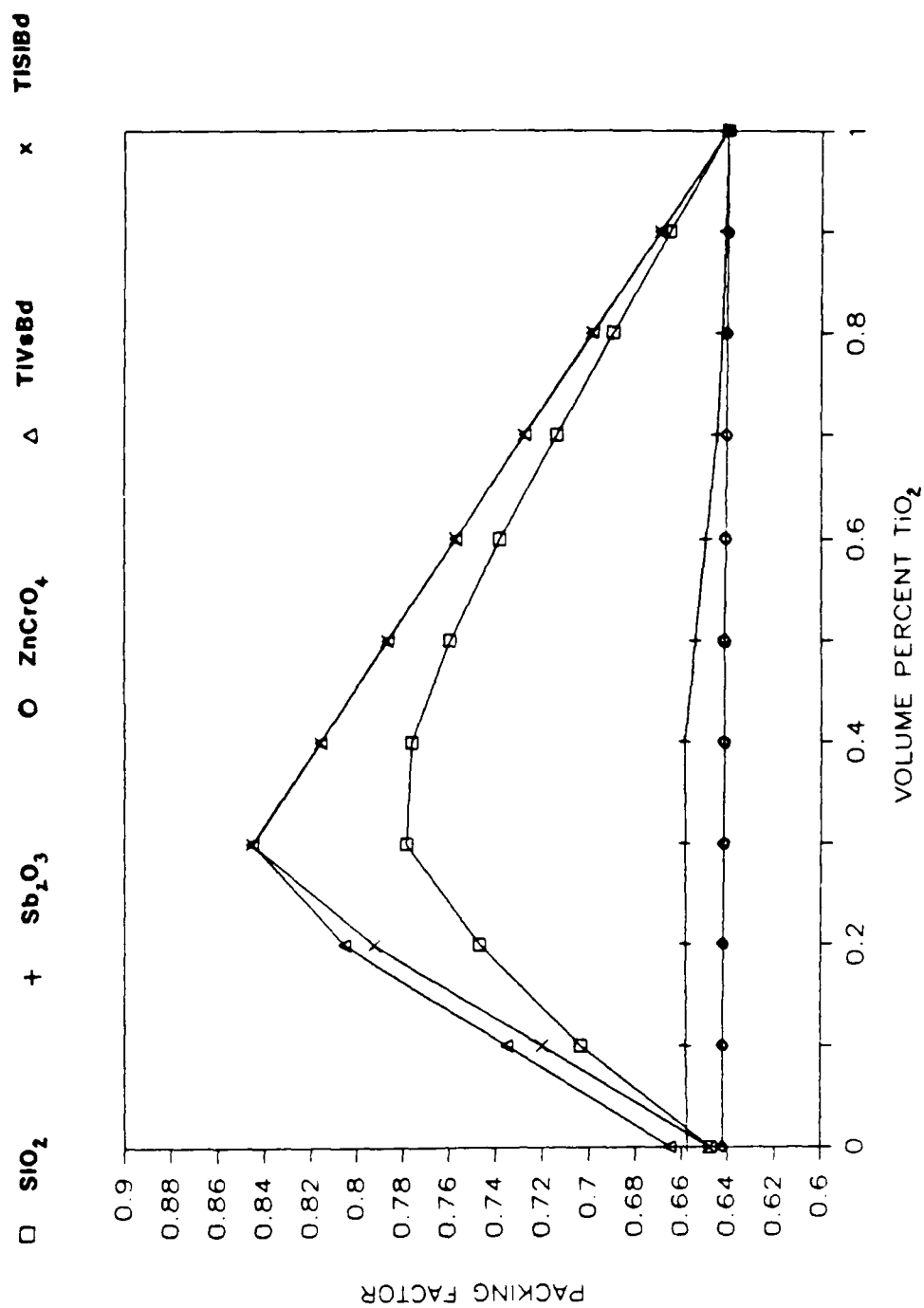


Figure 14: Packing Factor Versus Pigment Composition

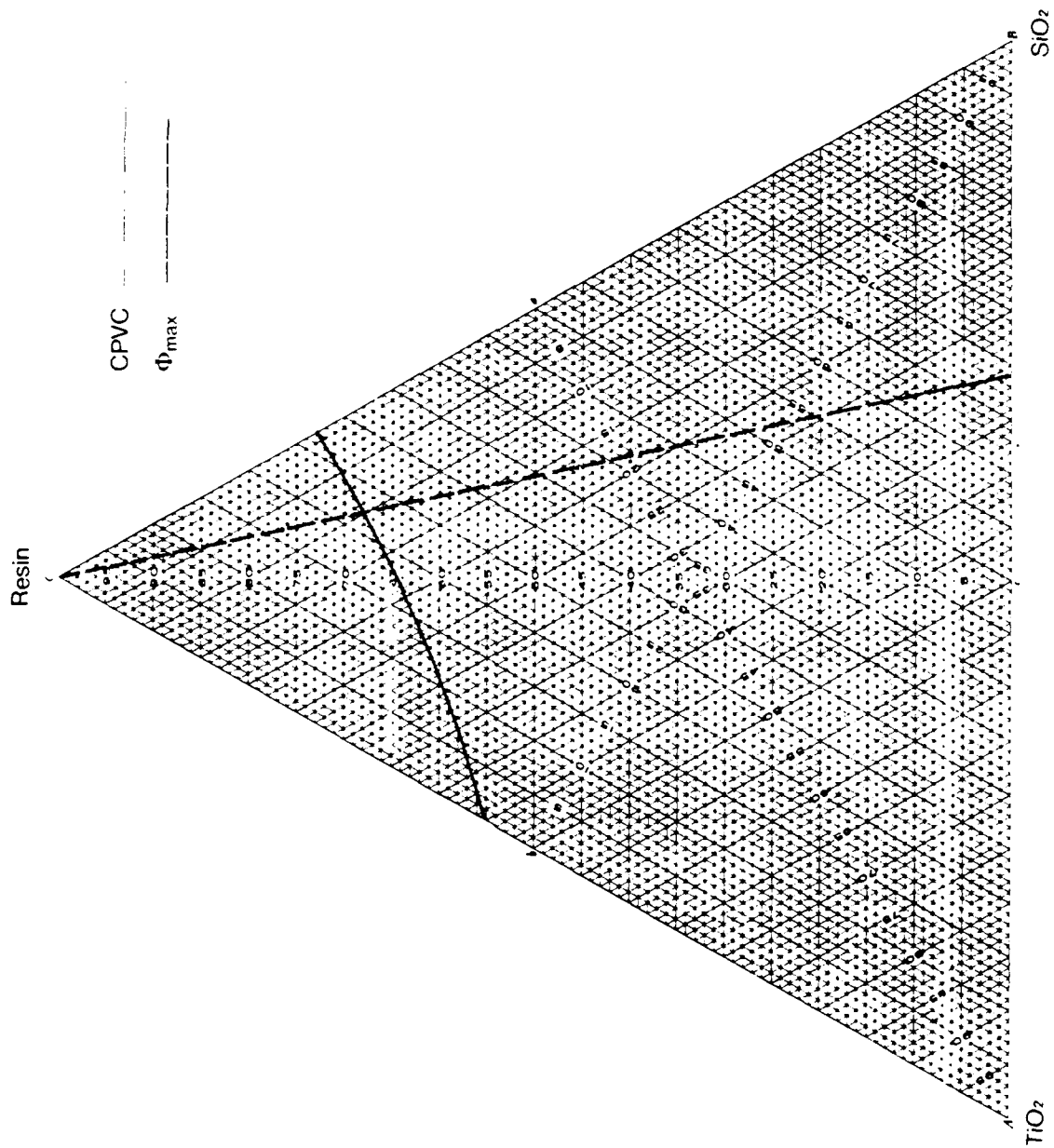


Figure 15: Theoretical $\Phi_{\max}$ and CPVC for TiO₂/SiO₂/Resin

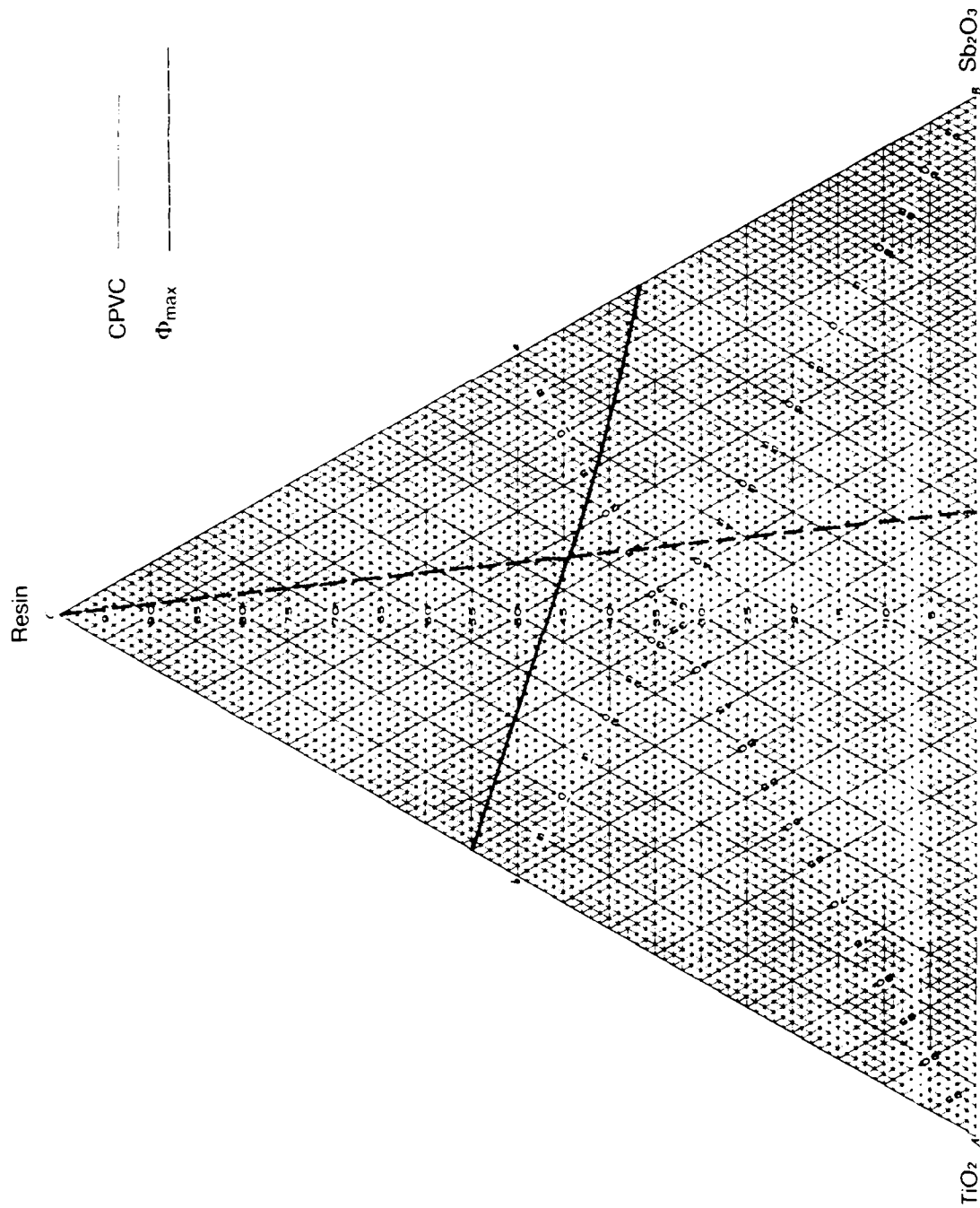


Figure 16: Theoretical $\Phi_{\max}$ and CPVC for TiO₂/Sb₂O₃/Resin

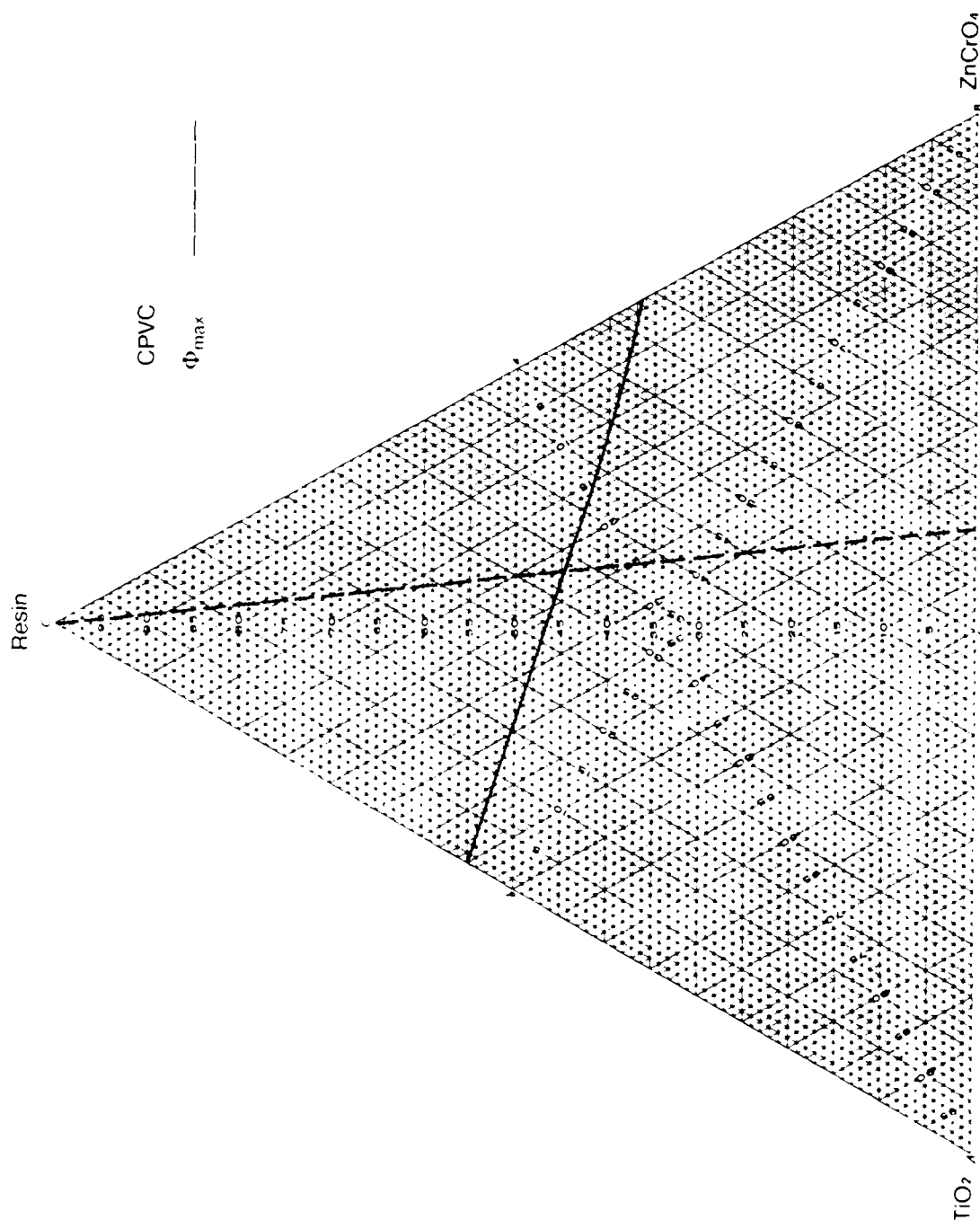


Figure 17: Theoretical Φ_{max} and CPVC for TiO₂/ZnCrO₄/Resin

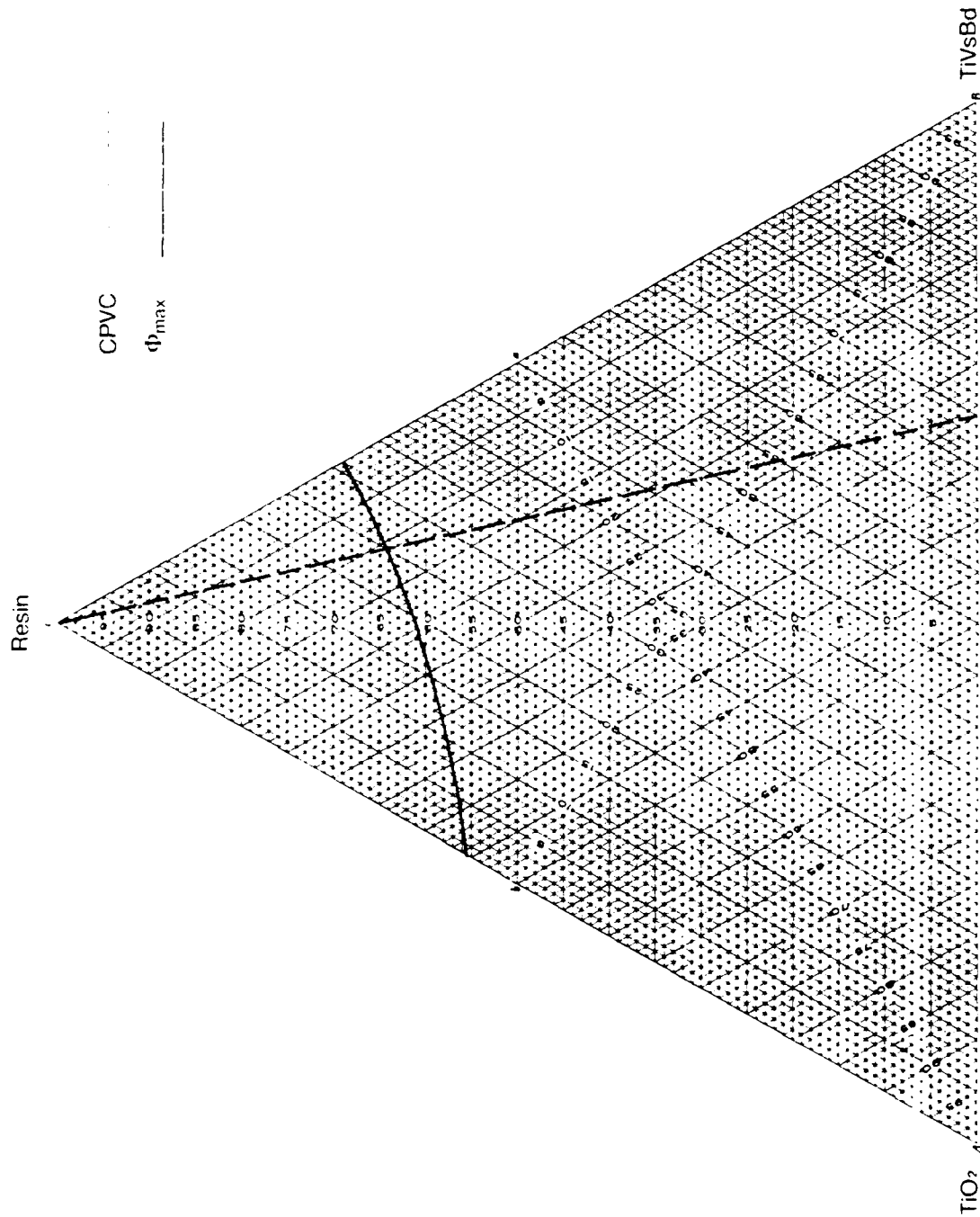


Figure 18: Theoretical $\Phi_{\max}$ and CPVC for $\text{TiO}_2/\text{TiVsBd}/\text{Resin}$

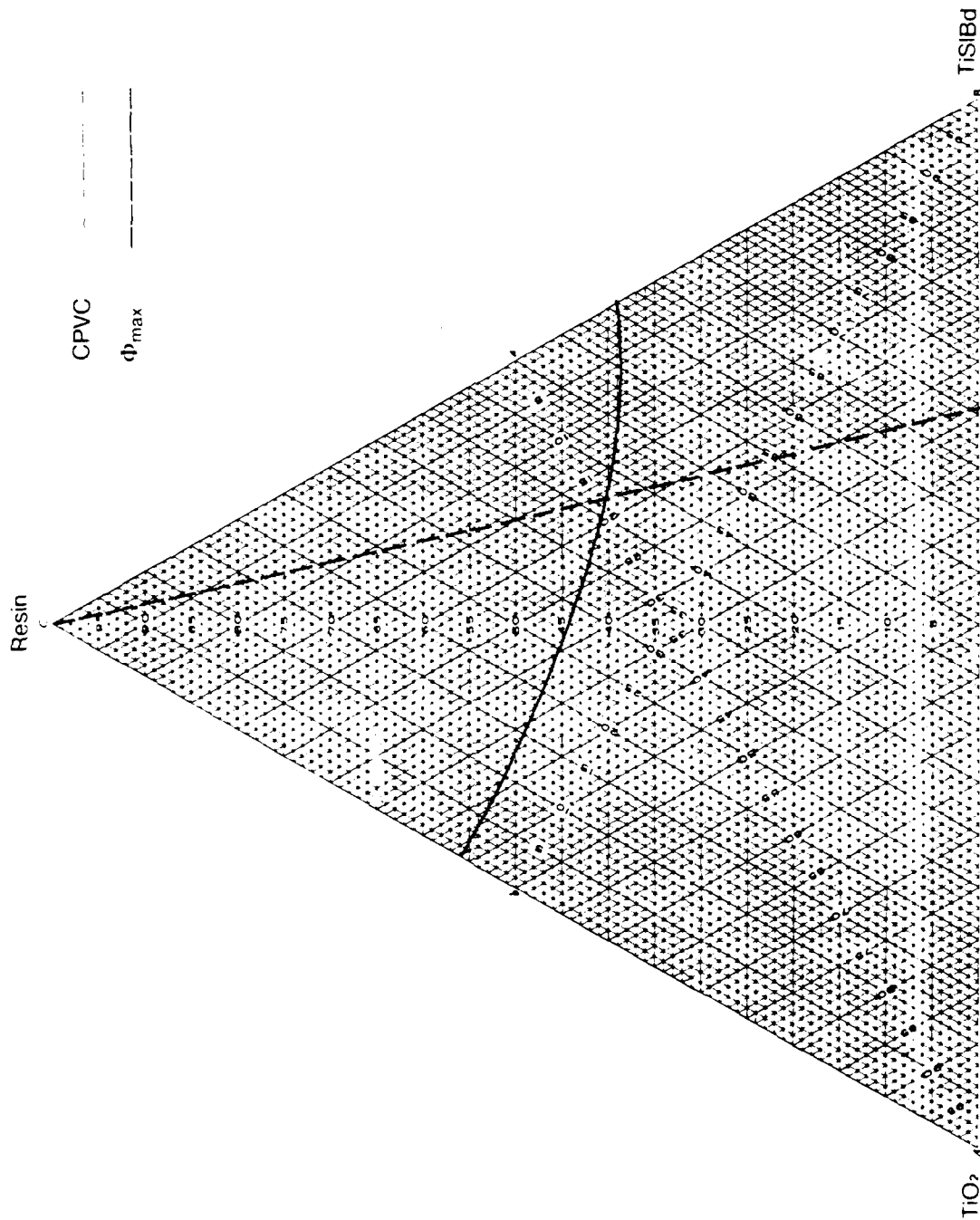


Figure 19: Theoretical $\Phi_{\max}$ and CPVC for TiO₂/TiSiBd/Resin

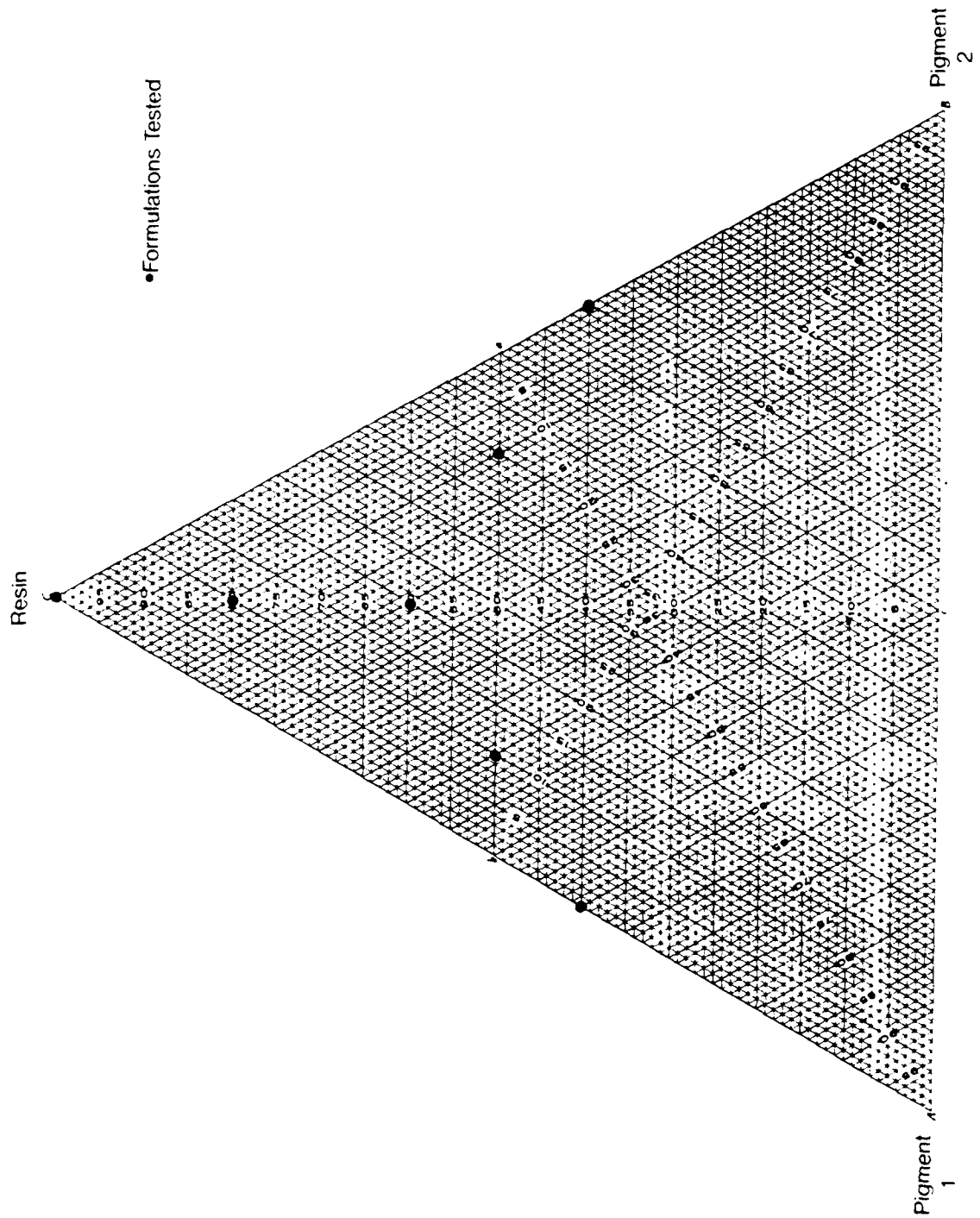


Figure 20: Simplex Screening Design Model

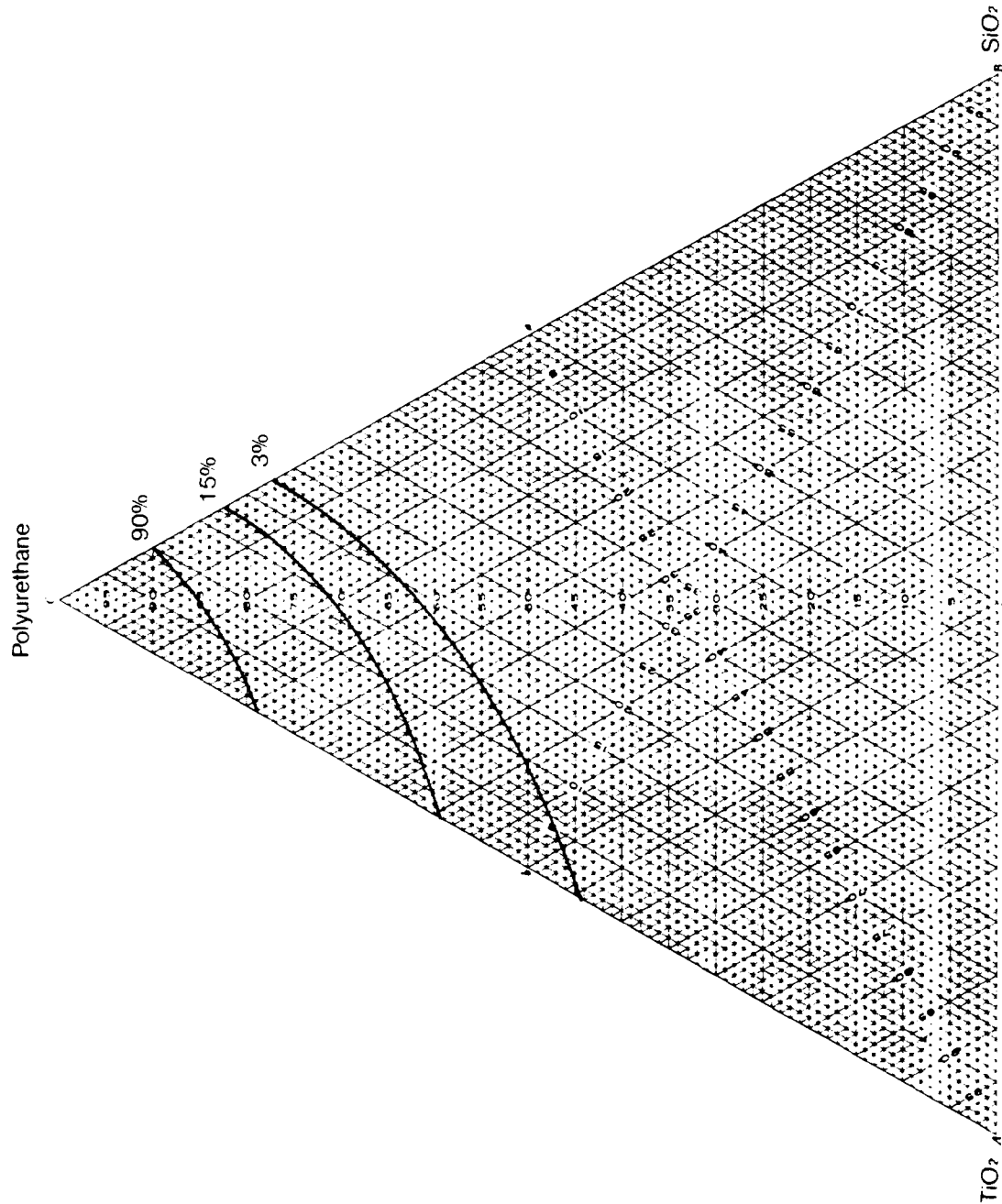


Figure 21: 60° Gloss for $\text{TiO}_2/\text{SiO}_2/\text{Polyurethane}$

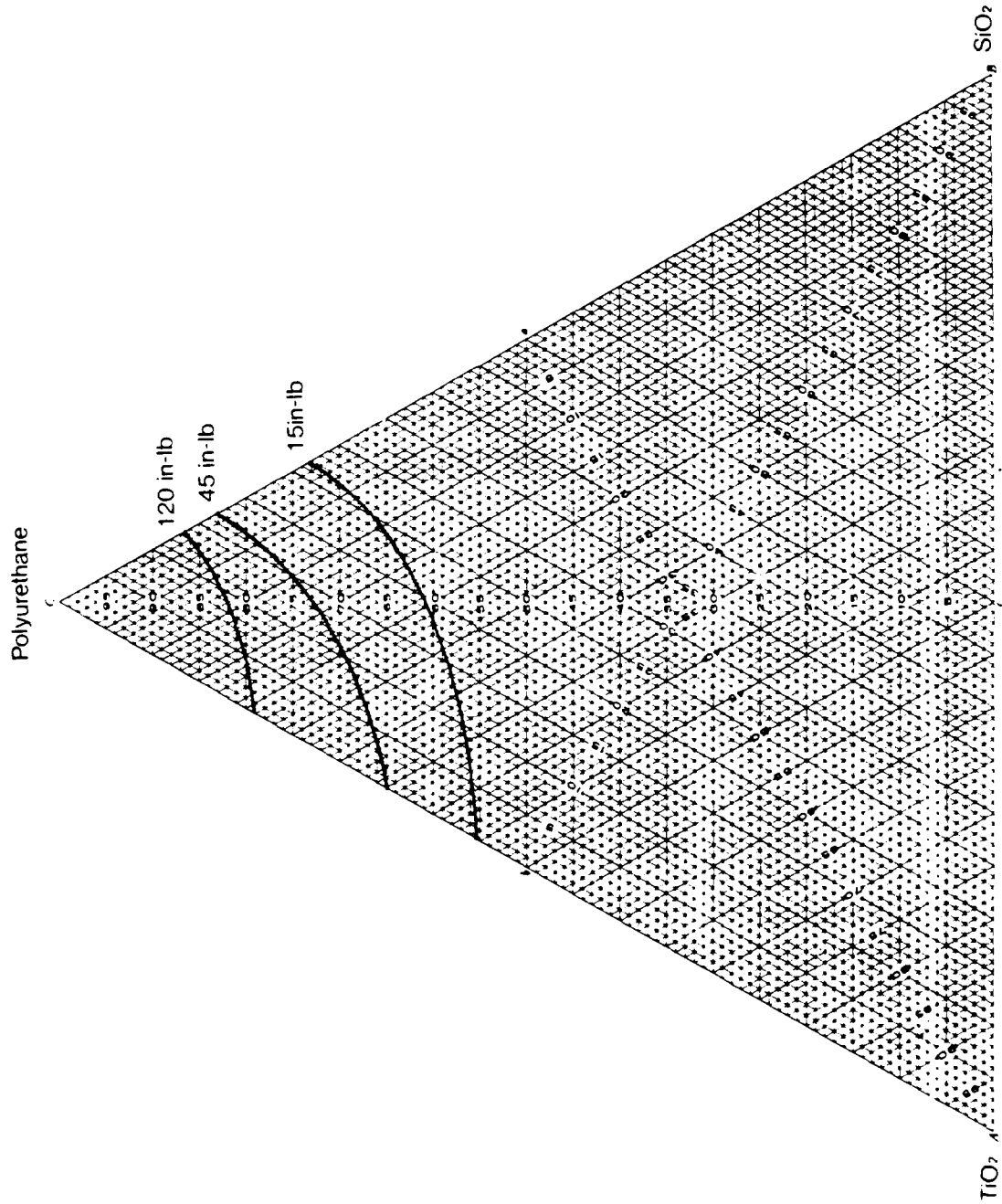


Figure 22: Direct Impact Flexibility for TiO₂/SiO₂/Polyurethane

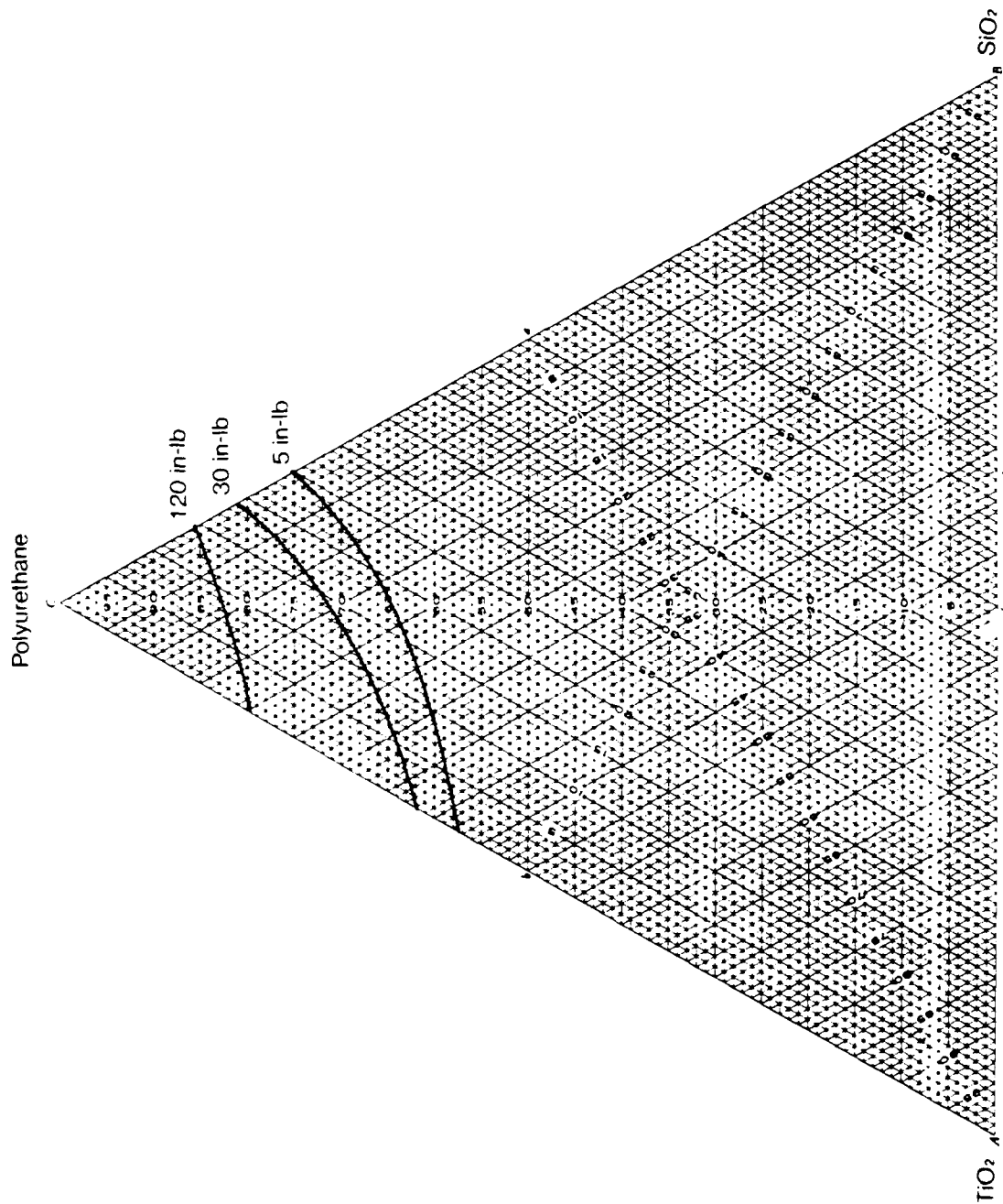


Figure 23: Reverse Impact Flexibility for $\text{TiO}_2/\text{SiO}_2/\text{Polyurethane}$

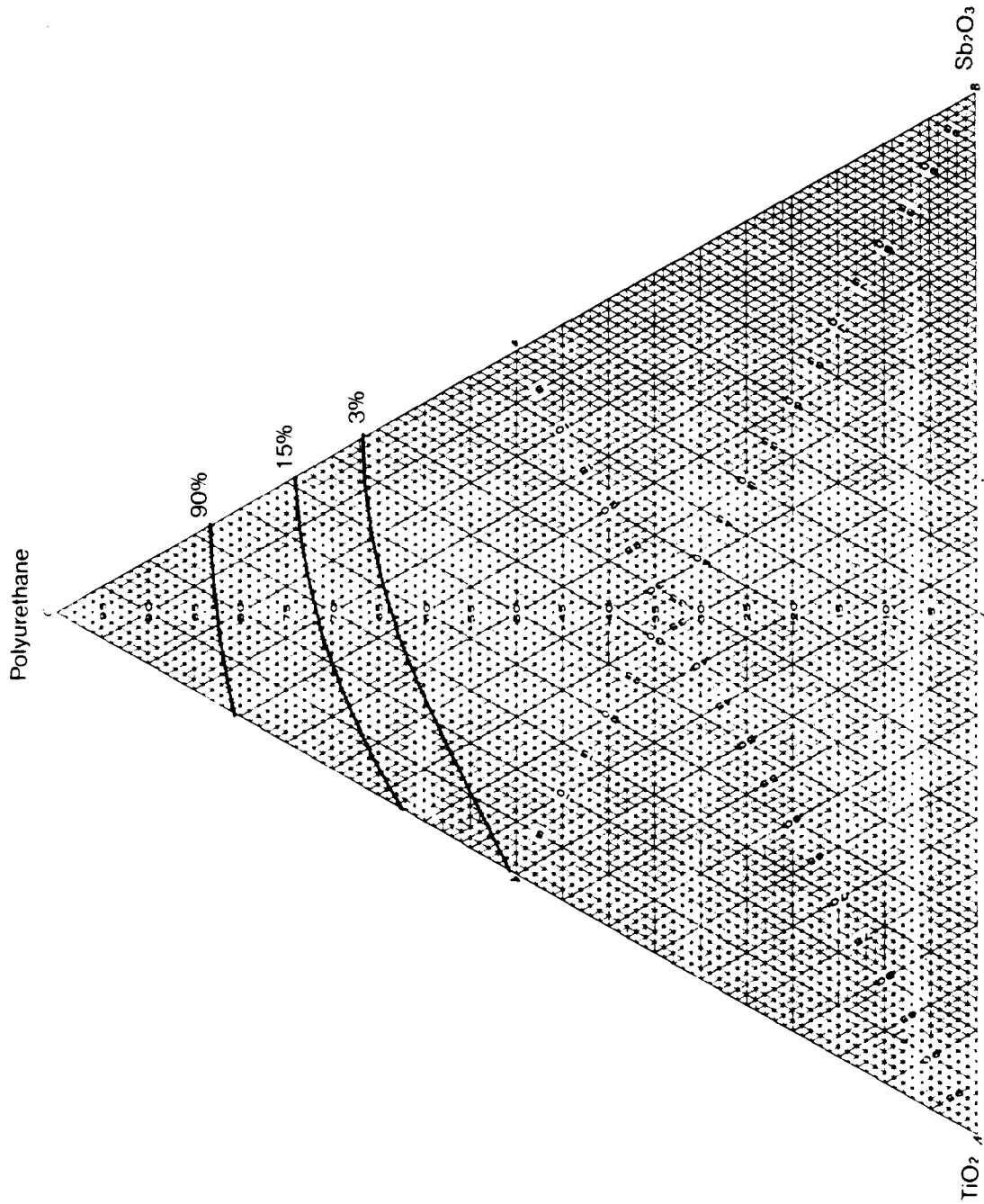


Figure 24: 60° Gloss for $\text{TiO}_2/\text{Sb}_2\text{O}_3/\text{Polyurethane}$

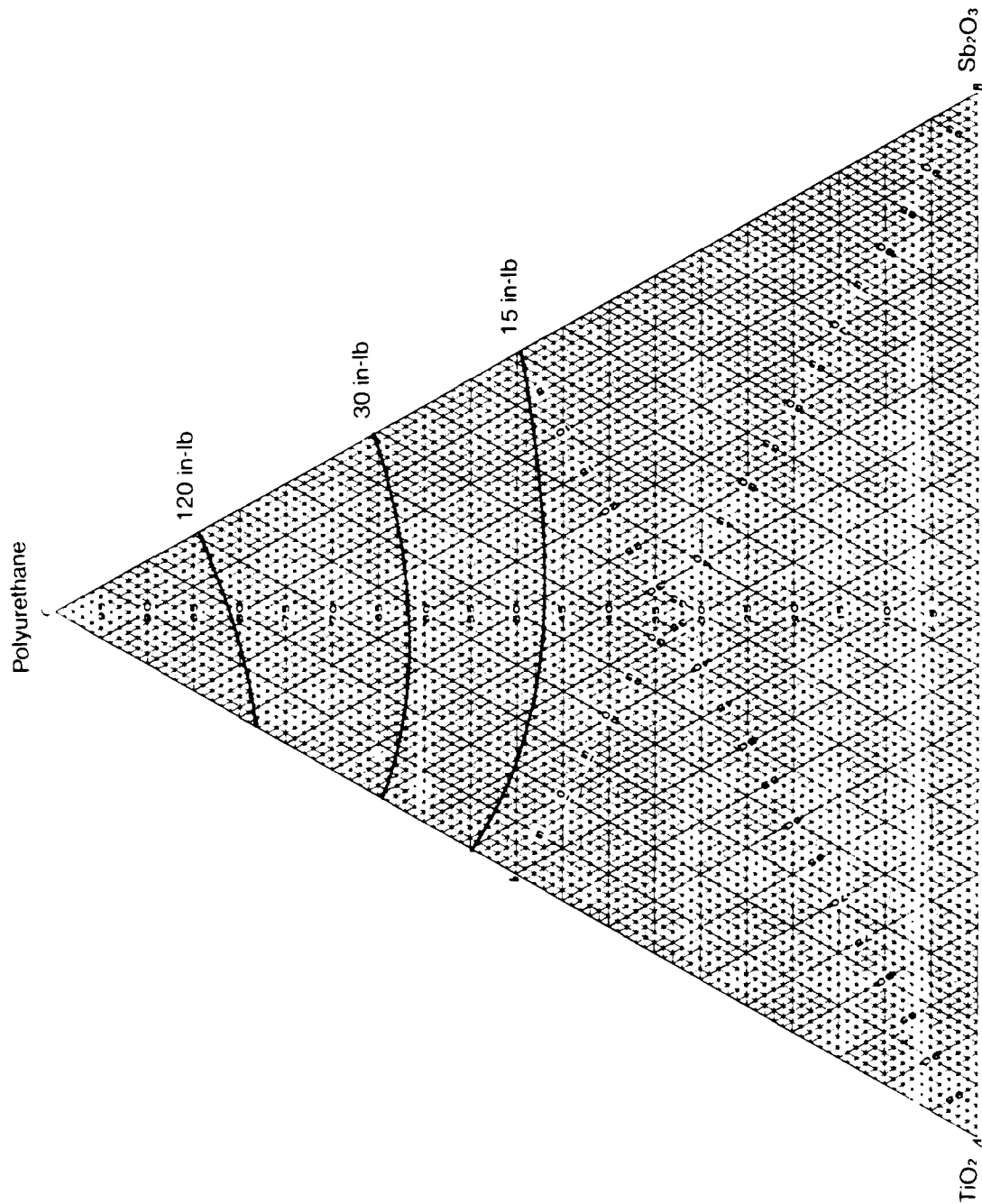


Figure 25: Direct Impact Flexibility for $\text{TiO}_2/\text{Sb}_2\text{O}_3$ /Polyurethane

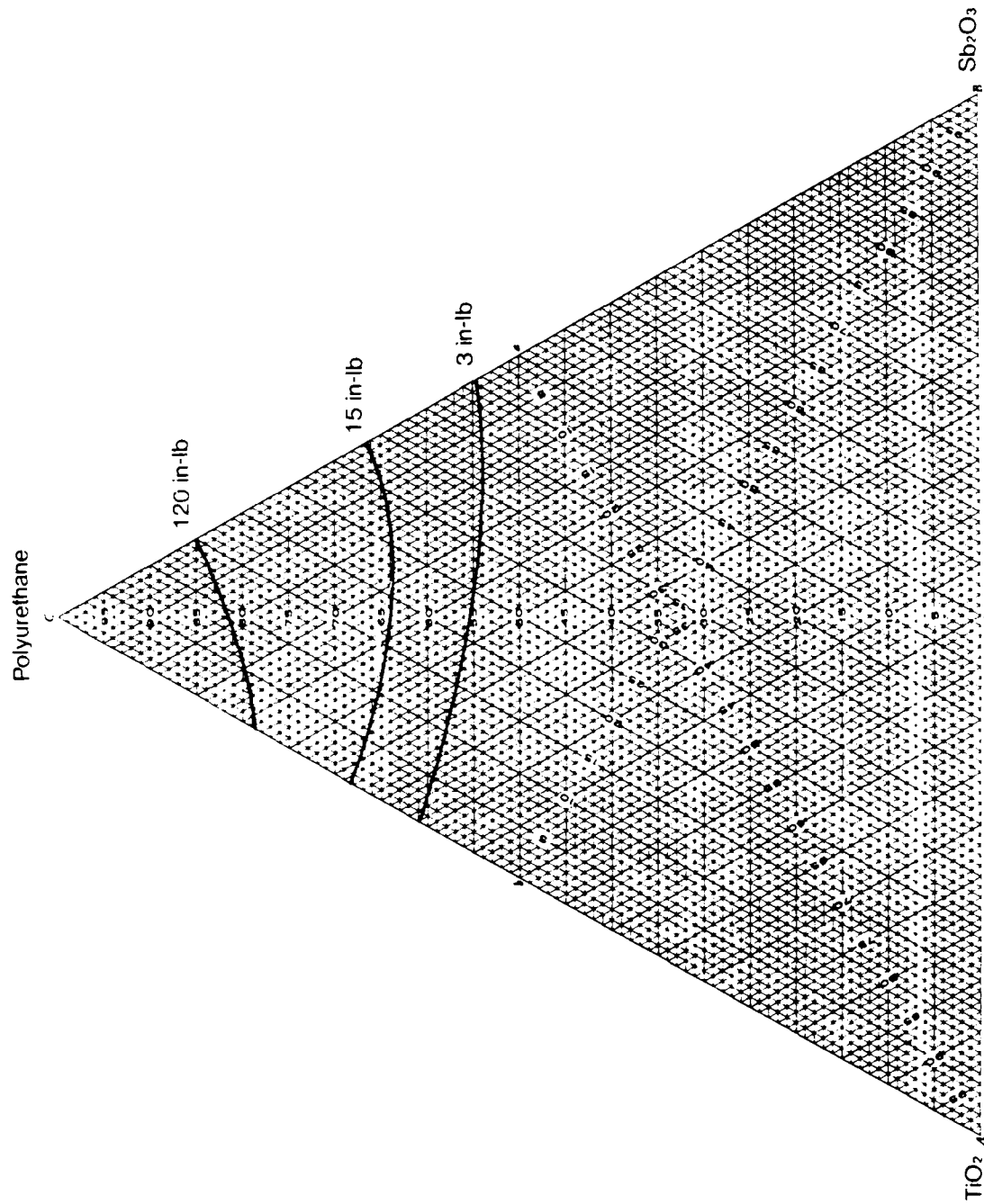


Figure 26: Reverse Impact Flexibility for $\text{TiO}_2/\text{Sb}_2\text{O}_3/\text{Polyurethane}$

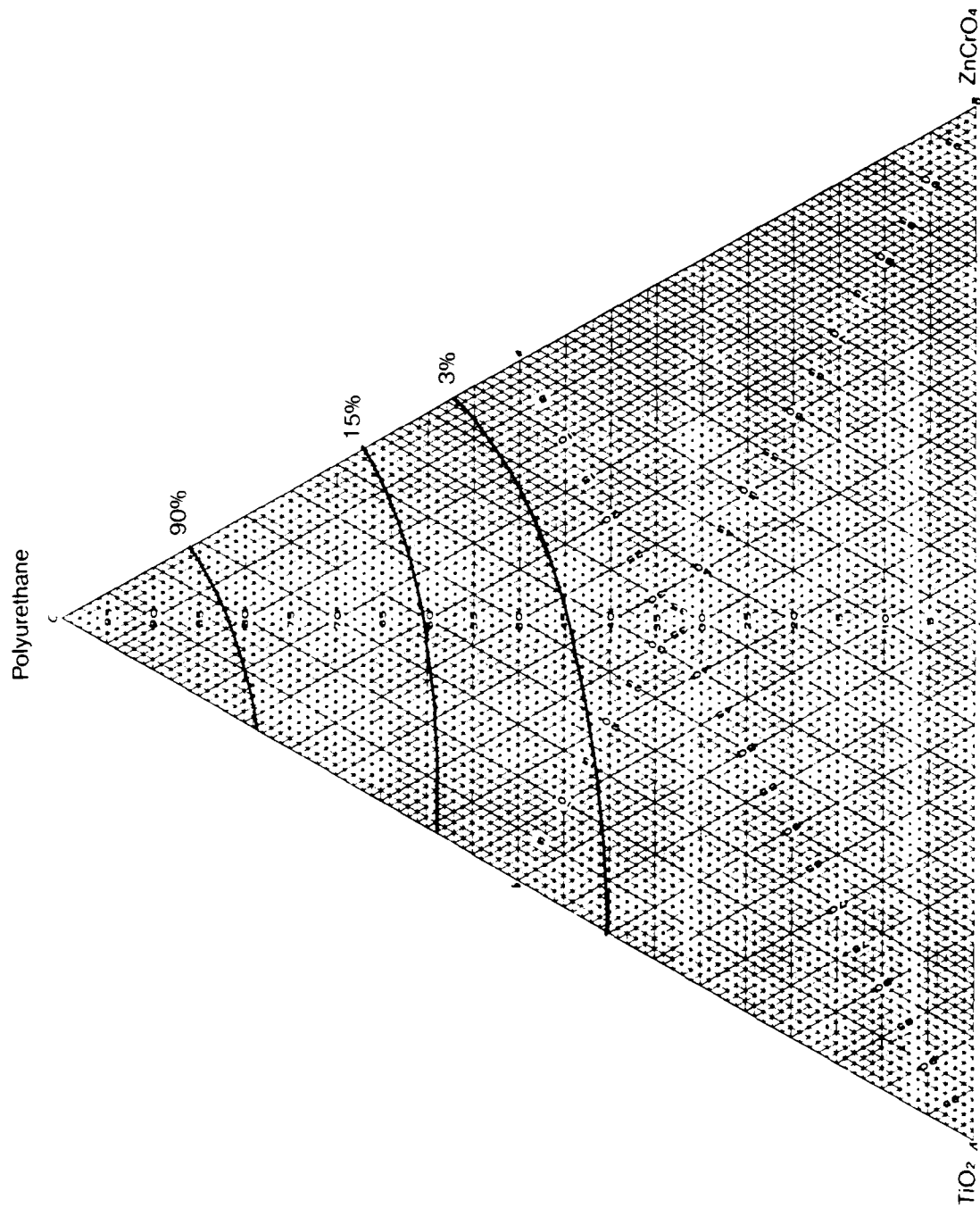


Figure 27: 60° Gloss for $\text{TiO}_2/\text{ZnCrO}_4/\text{Polyurethane}$

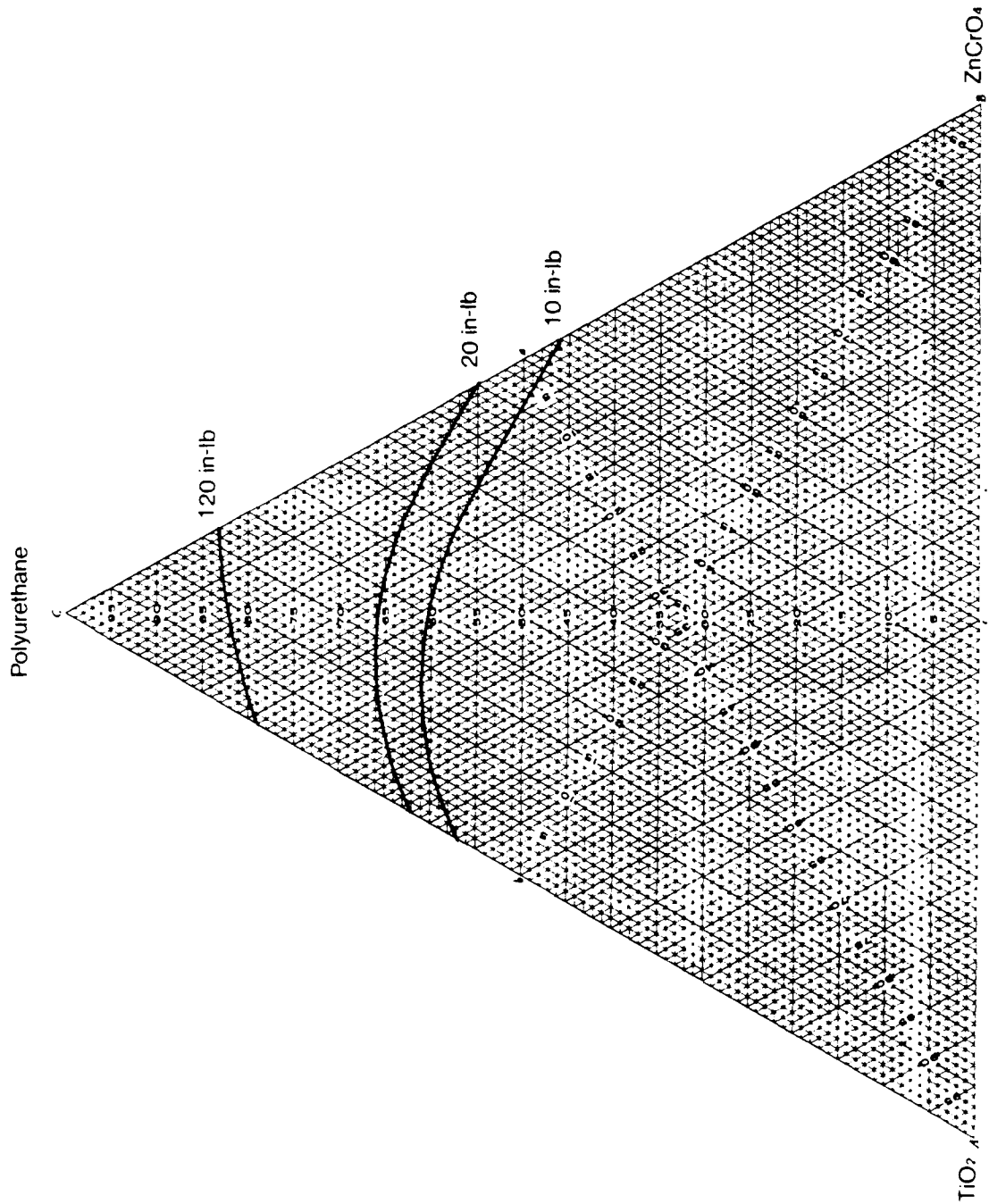


Figure 28: Direct Impact Flexibility for TiO₂/ZnCrO₄/Polyurethane

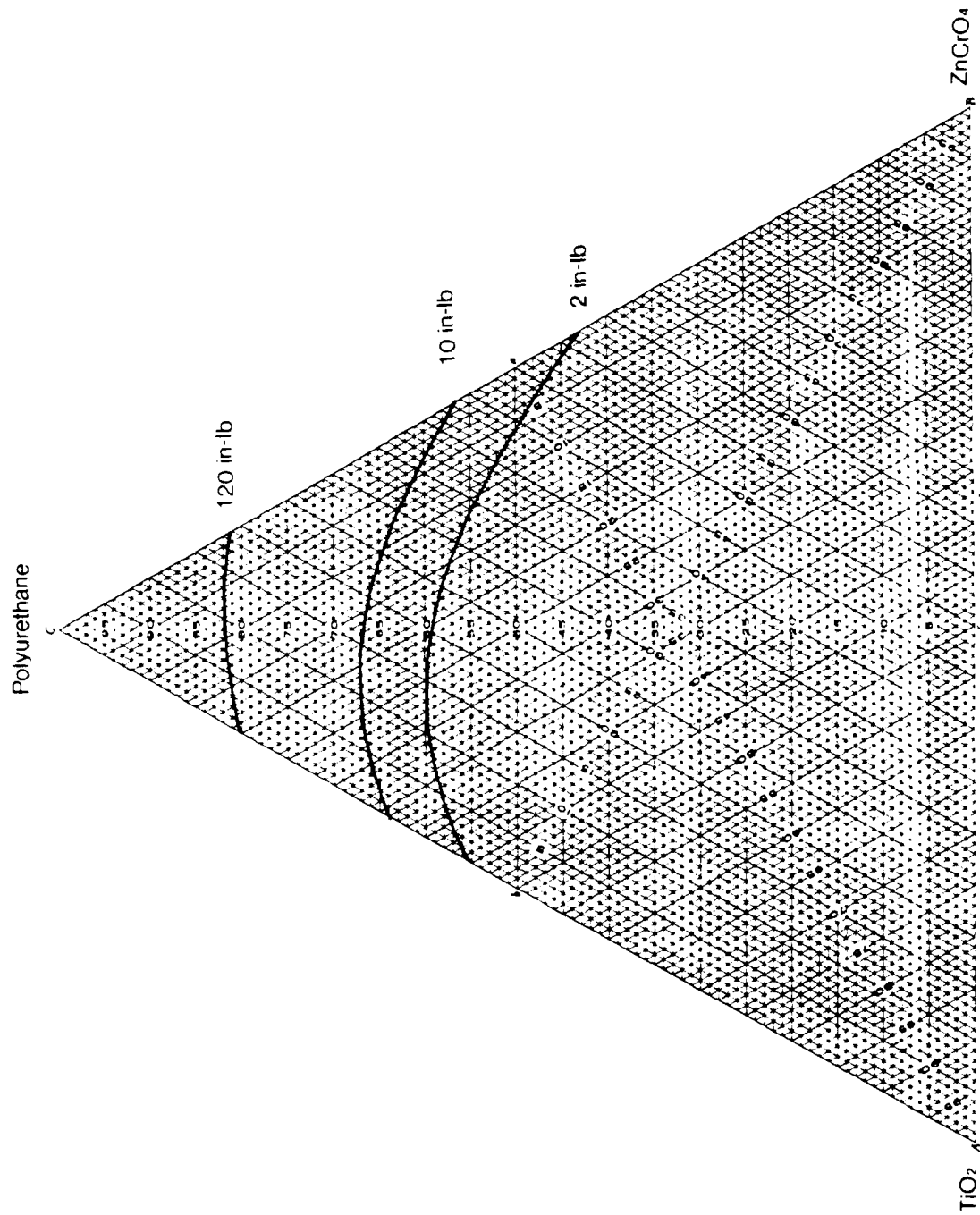


Figure 29: Reverse Impact Flexibility for $\text{TiO}_2/\text{ZnCrO}_4/\text{Polyurethane}$

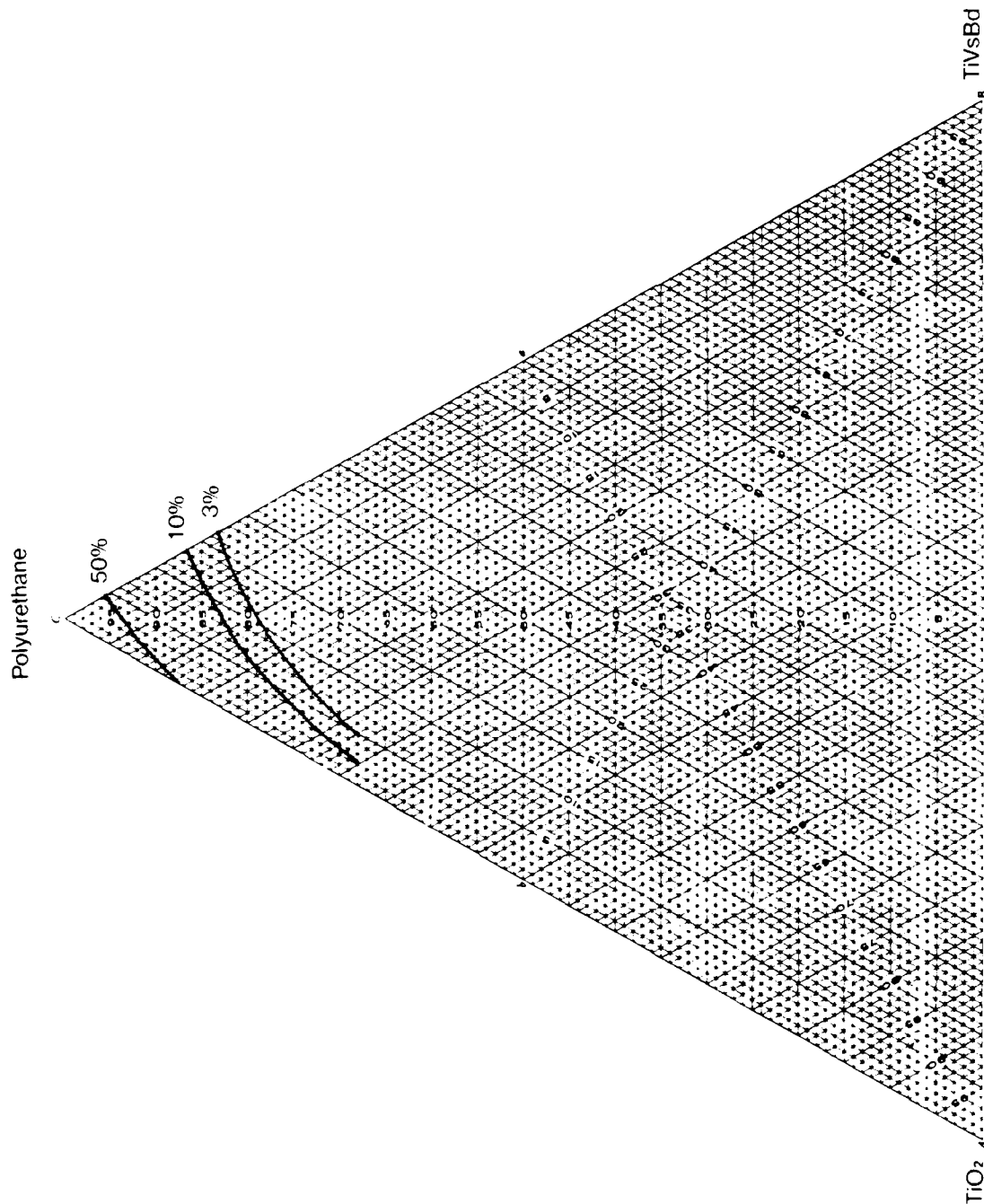


Figure 30: 60" Gloss for TiO_2 /TiVsBd/Polyurethane(5).

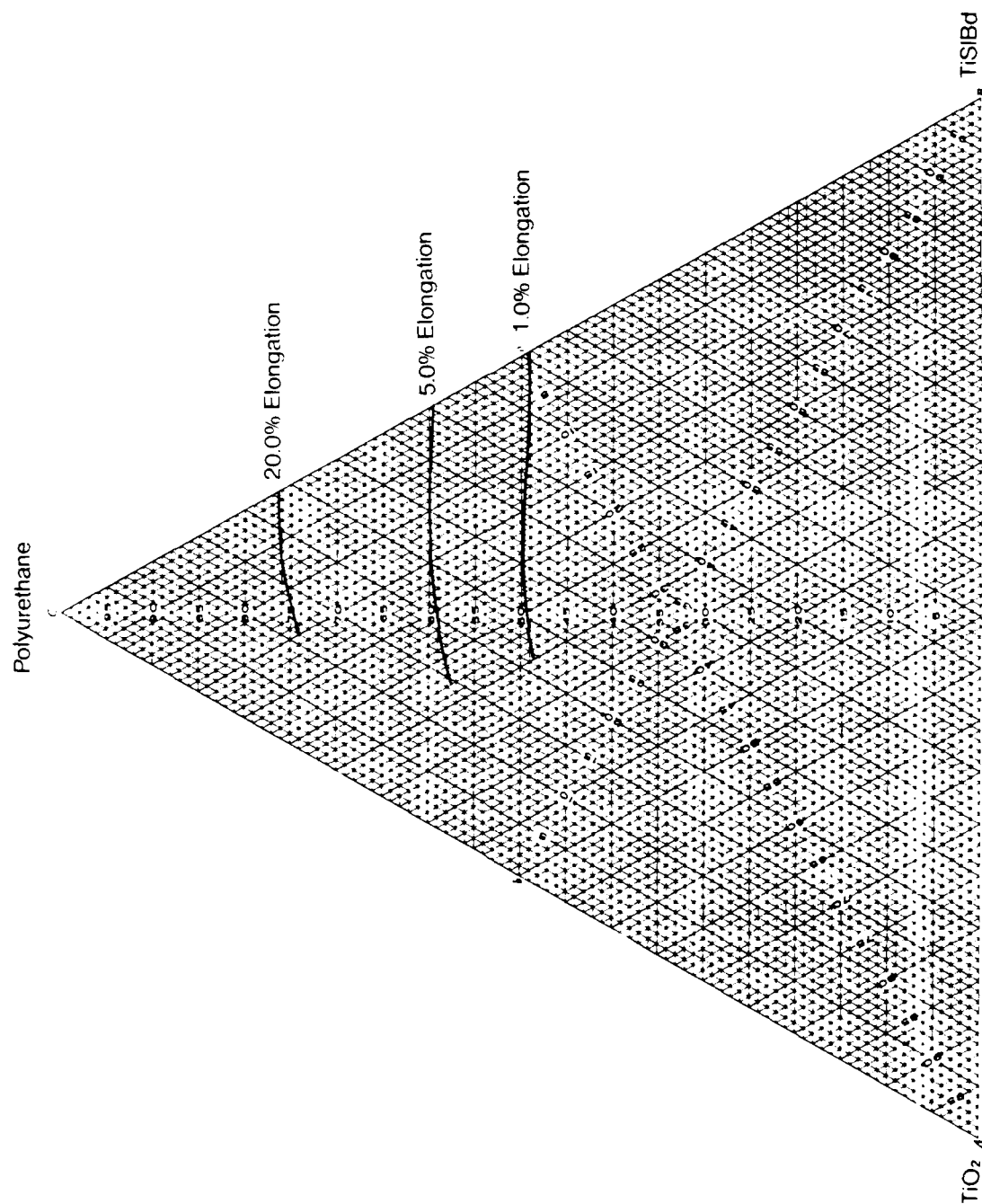


Figure 33: Impact Flexibility for TiO_2 /TiSiBd/Polyurethane (5).

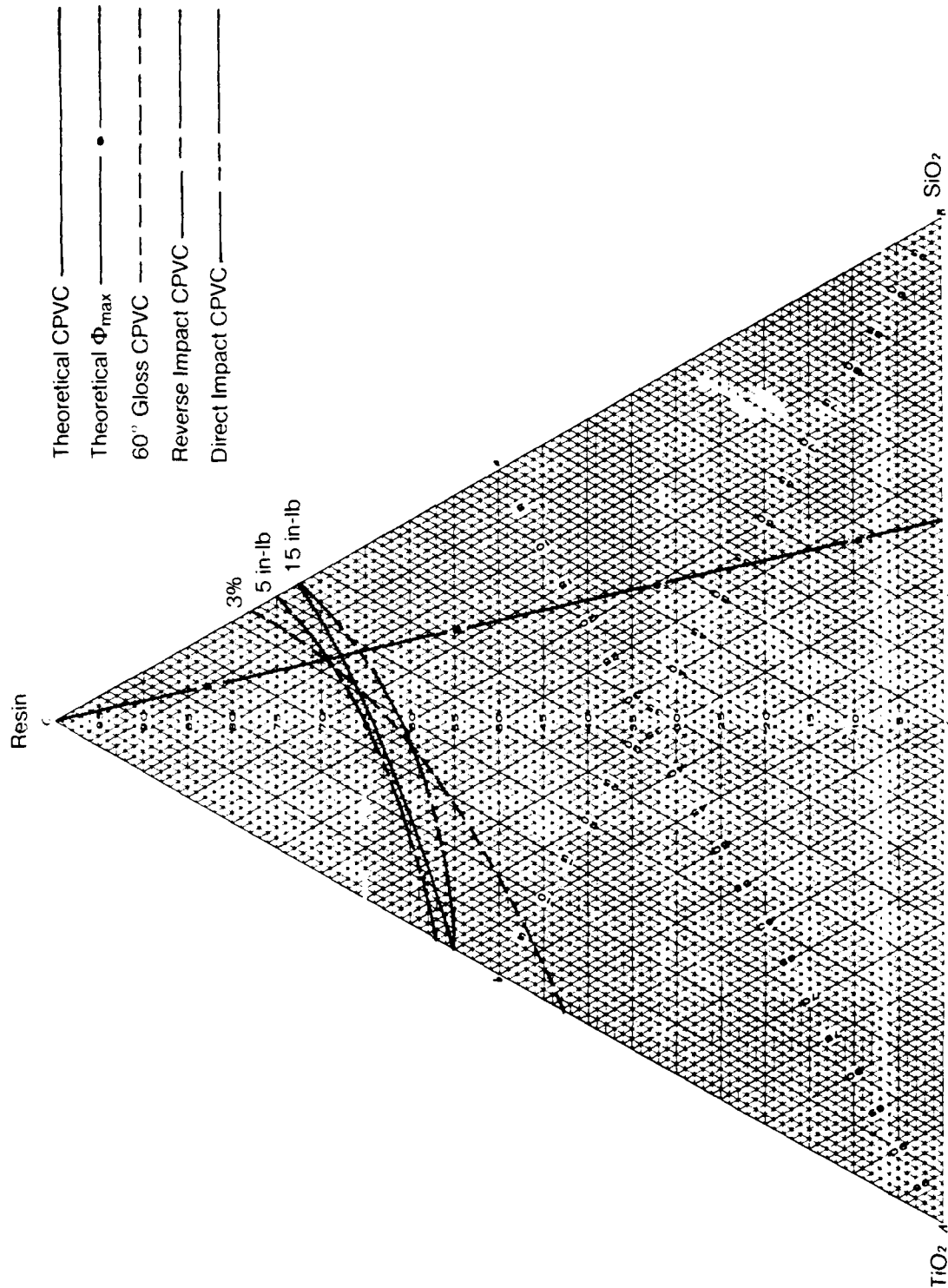


Figure 34: Experimental and Theoretical CPVC's for TiO₂/SiO₂

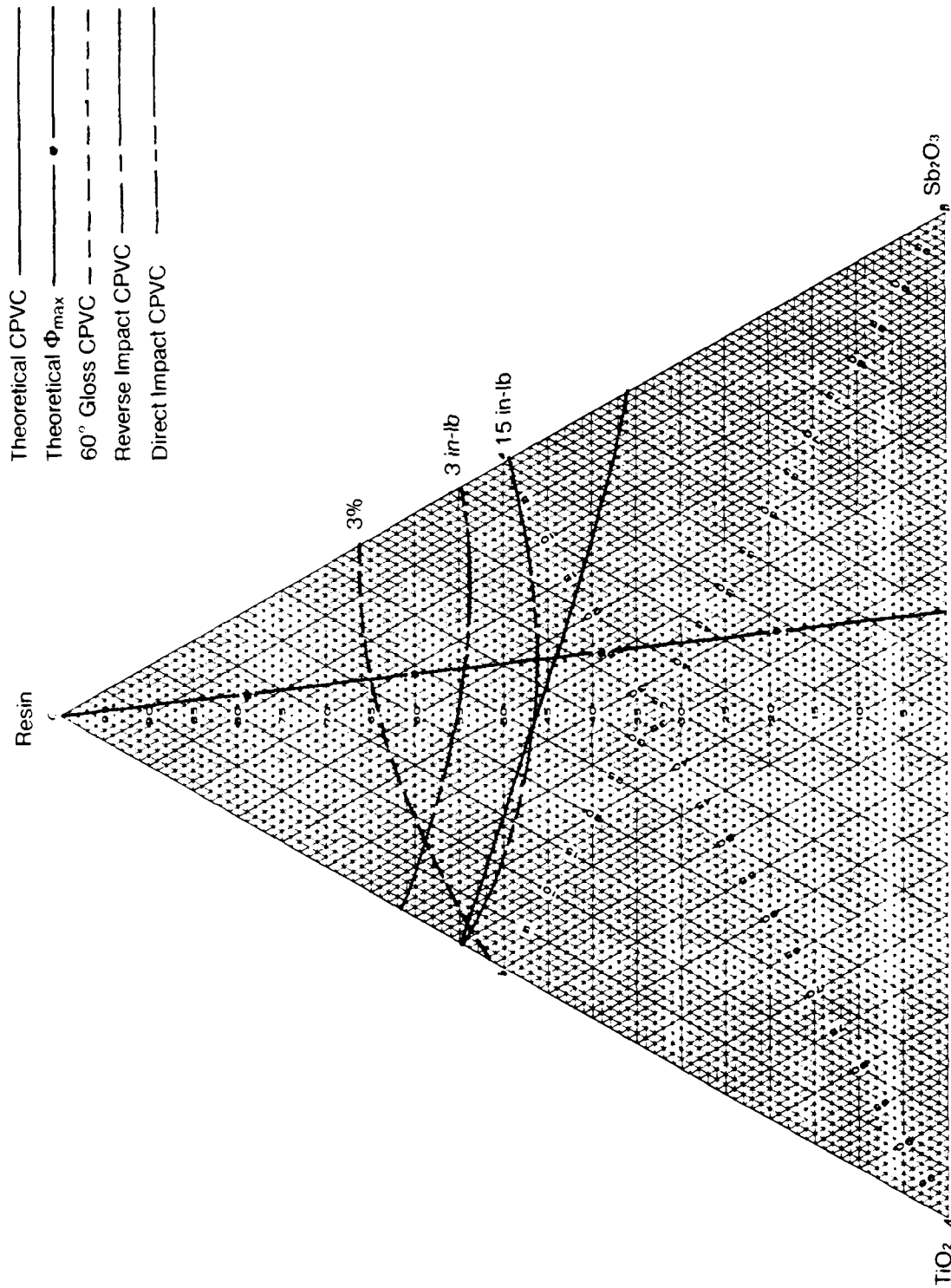


Figure 35: Experimental and Theoretical CPVC's for TiO₂/Sb₂O₃

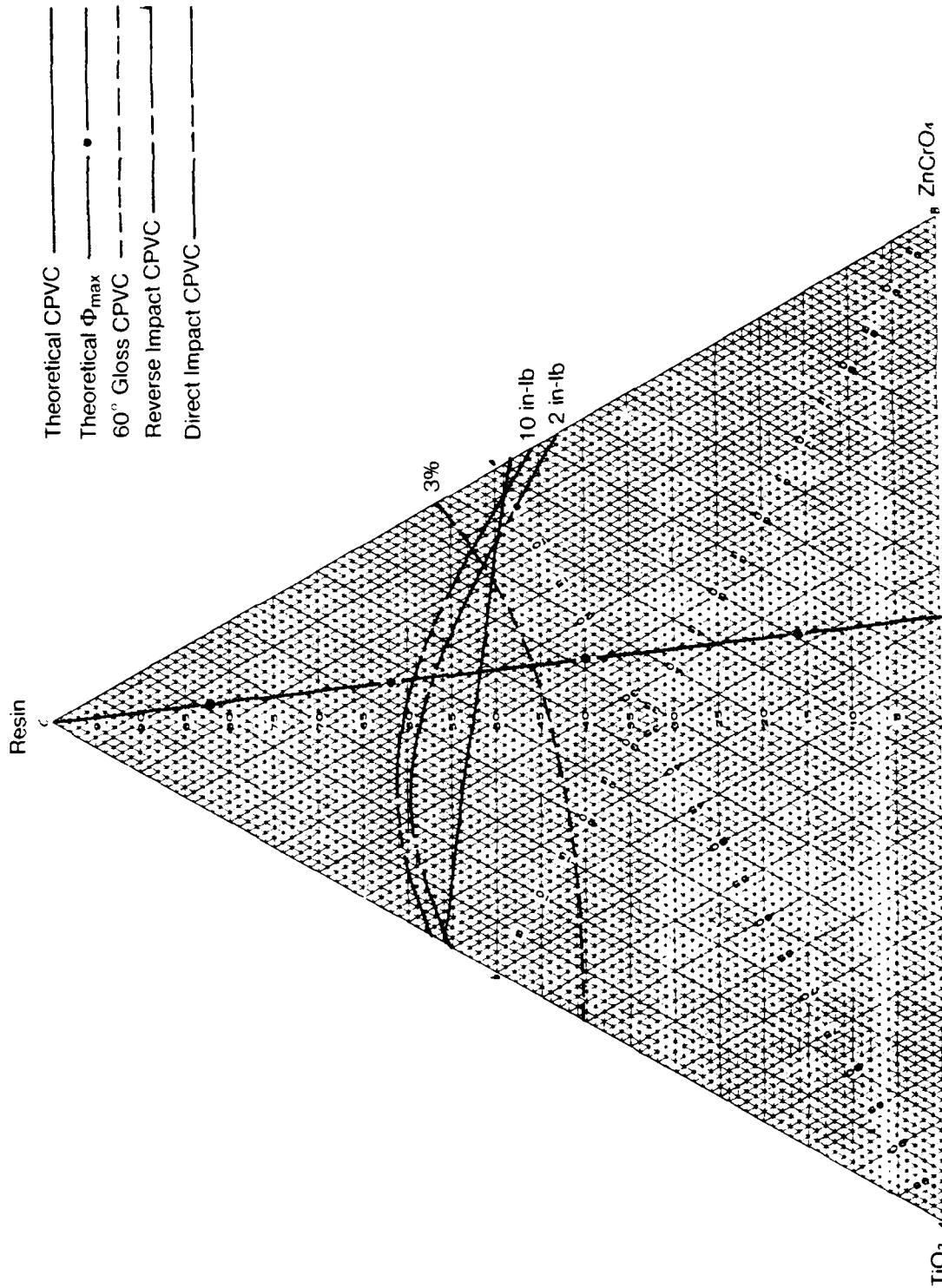


Figure 36: Experimental and Theoretical CPVC's for TiO₂/ZnCrO₄

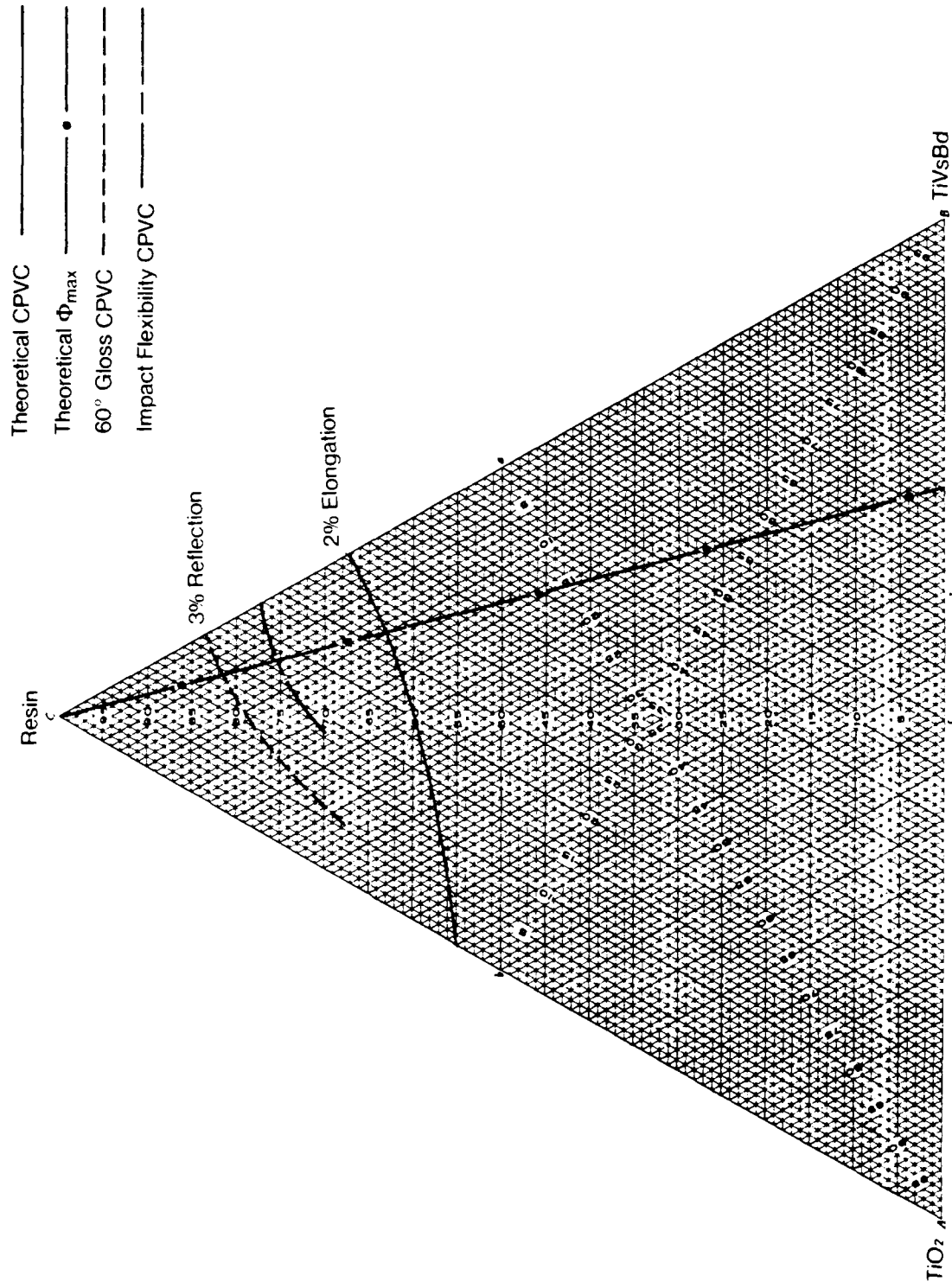


Figure 37: Experimental and Theoretical CPVC's for TiO₂/TiVsBd/Resin (5)

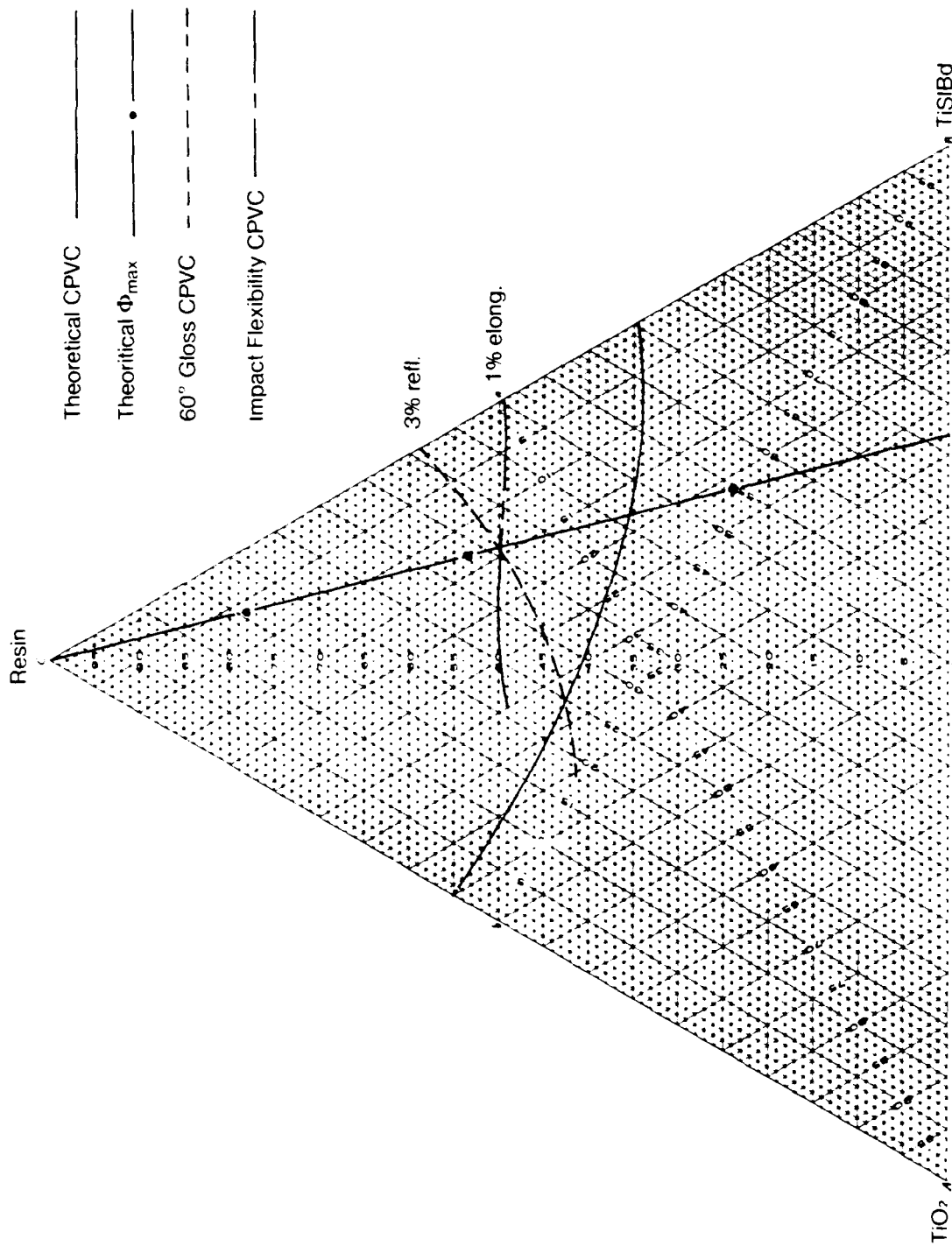


Figure 38: Experimental and Theoretical CPVC's for TiO₂/TiSiBd/Resin

Table I: Pigment Properties

	TiO ₂	SiO ₂	Sb ₂ O ₃	ZnCrO ₄	TiVsBd	TiSiBd
Manufacturer and Designation	Dupont R-960	Johns-Man-ville Celite 499	Harshaw Kr-325	Hercules Y-2082	Enterprise Chemical	Enterprise Chemical
Appearance	White spherical	White platelet	White nodular	Yellow rectangular	White sperical	White spherical
Density (g/ml)	4.0	2.36	5.6	3.5	1.30	1.62
Oil absorption ASTM D281	29.3	111.2	9.3	25.5	146.8	29.9
Refractive Index	2.76	1.48	2.00	---	---	---
Particle Diameter (microns)	0.3	2.0	0.3	0.25	5.0	4.0
CPVC, Theoretical	0.448	0.267	0.641	0.513	0.317	0.658

Table II: Polyurethane Resin Formulation

Component I	Weight Percent
Mobay Desmophen 651A-65	7.1
Mobay Desmophen 670-90	23.2
Dibutyltin Dilaurate (2% in MEK)	0.7
Monsanto Modaflow (10% in MEK)	1.3
Methyl Ethyl Ketone (MEK)	28.7
Component II	
Mobay Desmodur N-75	24.7
Propylene Glycol Monomethyl Ether Acetate	7.2
Toluene	7.2

APPENDIX A

Lee's Algorithm

$$(\Phi_p)_i = \sum_{j=1}^{i-1} \Phi_{ij} X_j$$

$$\sum_{j=1}^{i-1} x_j = 1$$

$$\Phi_{ii} = 0.639$$

$$\Phi_{ij} = \Phi_{ii} + [\Phi_{\max}(D_i/D_j) - \Phi_{ii}]/0.265$$

$$\Phi_{ji} = \Phi_{ii} + [\Phi_{\max}(D_i/D_j) - \Phi_{ii}]/0.735$$

where

Φ_{ii} = packing factor for uniform or mono sphere size i

Φ_{ij}, Φ_{ji} = binary packing factor coefficient of i and j sphere sizes

$\Phi_{\max}(D_i/D_j)$ = maximum packing factor of binary sized spheres with the diameter ratio of $D_i/D_j \geq 1$

x_j = volume fraction of j sphere size

$(\Phi_p)_i$ = packing factor of n -component mixtures of spheres when $(\Phi_p)_i$ is a minimum

APPENDIX B

CPVC Calculations for Pigment Mixtures

Calculation of CPVC is based on two physical properties of pigments: the particle size distribution and the oil absorption value of the pigment. The pigment particles are assumed to be inert spheres embedded in a resin matrix; this assumption is also extended to cases when pigment mixtures are used. The oil absorption value of a pigment is that amount of oil necessary to coat all of the pigment particles, fill all of the interstitial spaces between the pigment, and be absorbed by the pigment leaving no excess oil or pigment. Therefore, packing of the pigment and the fraction of volume which the pigment particles occupy relative to the total volume affects the CPVC.

The CPVC has the same definition as oil absorption where it applies to a pigment-in-resin system. The CPVC can be mathematically expressed as:

$$\text{CPVC} = \frac{V_{\text{pig}}}{\text{Total Volume}} = \frac{V_{\text{pig}}}{V_{\text{pig}} + V_{\text{res-min}}} = \frac{1}{1 + (V_{\text{res-min}}/V_{\text{pig}})} \quad \text{B-1}$$

where V_{pig} is the volume of pigment and $V_{\text{res-min}}$ is the volume of resin at the CPVC point. Since the oil absorption equals $V_{\text{res-min}}/V_{\text{pig}}$, we can express CPVC as:

$$\text{CPVC} = \frac{1}{1 + \text{OA}} \quad \text{B-2}$$

where oil absorption, OA, is expressed as volume of oil per unit volume of pigment. Another assumption is that the oil used in the oil absorption determination wets the pigment similar to the resins used in the paint formulation.

The above equation, B-2, is strictly correct for a single pigment. However, if one is dealing with a multi-component system, the packing of one pigment with another becomes an important factor to consider. If one pigment has a mean particle size of 10 microns and a second pigment has a mean particle size of 0.5 microns, very efficient packing will take place due to the smaller pigment particles filling the interstitial spaces between the larger pigment particles. This, in turn, affects the oil absorption value of the mixture because of the decreased amount of interstitial volume.

The approach taken in this study was to separate the oil absorption value into its three volume components: V_{ab} , V_{ad} , and V_{i} representing the volume of absorbed oil, adsorbed oil, and interstitial oil, respectively. This can be mathematically represented as:

$$\text{OA} = V_{\text{ab}} + V_{\text{ad}} + V_{\text{i}} \quad \text{B-3}$$

It is assumed that, upon mixing pigments, the values of V_{ab} and V_{ad} will not change. In other words, pigment 'A' does not affect the absorbing or the adsorbing of oil into or onto pigment 'B'. However, the value of V_i will change, unless the two pigments have identical particle size distributions. Thus, an equation for computing the oil absorption value of a pigment mixture is:

$$OA_{mix} = X_A(V_{ab} + V_{ad})_A + X_B(V_{ab} + V_{ad})_B + \dots + X_n(V_{ab} + V_{ad})_n + (V_i)_{mix} \quad B-4$$

Once OA_{mix} is computed, the CPVC of a pigment mixture follows from equation B-2:

$$CPVC_{mix} = \frac{1}{1 + OA_{mix}} \quad B-5$$

Let us now examine how to determine each of the terms in equation B-4, such that $CPVC_{mix}$ for a multi-pigment system may be computed from equation B-5. For simplicity, we shall start with the particle size distribution of a binary mix of pigments A and B. A computer program was written to perform the computations (see Appendix C.). Using an algorithm described and presented in reference (4), a packing factor (Φ) could be determined for any given particle size distribution. Φ represents the percent of actual volume the pigment particles occupy; $(1 - \Phi)$ represents the interstitial volume between the pigment particles. The algorithm, based on random dense packing of spheres, assigns a Φ of 0.640 for a mono-sized distribution. A wider range of particle size distribution yields a higher value of Φ , due to the filling of the interstitial volume of the larger particles by the smaller particles. Φ reaches a maximum value of 0.870 for an infinitely wide particle size distribution.

Once Φ is calculated for each individual pigment, V_i is simply:

$$V_{iA} = 1 - \Phi_A \text{ for pigment A} \quad B-6$$

$$V_{iB} = 1 - \Phi_B \text{ for pigment B} \quad B-7$$

for equation B-3, there are still two unknowns for each pigment: V_{ab} and V_{ad} . These two values can be treated as one unknown ($V_{ab} + V_{ad}$). Because oil absorption is known, ($V_{ab} + V_{ad}$) can be determined from equation B-3:

$$(V_{ab} + V_{ad}) = OA_A - V_{iA} \text{ for pigment A} \quad B-8$$

$$(V_{ab} + V_{ad}) = OA_B - V_{iB} \text{ for pigment B} \quad B-9$$

Now we can combine both particle size distributions (mathematically) and compute a packing factor for the mixture of pigments Φ_{mix} . From equations B-6 and B-7, V_{imix} can be computed. All of the necessary terms in equation B-4 are now known for the calculation of OA_{mix} and thus $CPVC_{mix}$. Expansion of these equations to pigment mixtures of more than two pigments is straightforward.

APPENDIX C

COMPUTER PROGRAM TO CALCULATE CPVC AND $\Phi_{\max}$

The following is a computer program, example input data, and corresponding output for the calculation of critical pigment volume concentration (CPVC) and maximum packing factor ($\Phi_{\max}$). The program is written in Fortran IV for use on a Control Data Corp. 6600 computer. The example is for a pigment system of 70% TiVsBd (Pigment 1) and 30% titanium dioxide (Pigment 2) by volume.

```

      PROGRAM AB(INPUT,OUTPUT,TAPE5=INPUT)
      DIMENSION NPART(100),SIZ(100,4),VOL(100),VOLSIZ(100,4)
      DIMENSION VOLPER(100,4),PIGPER(4),TOTVOL(4),NSIZ(4)
      DIMENSION VARS(4)
      DIMENSION COMBSZ(400),VADS(4),PCK(5),DIL(4)
      DIMENSION PHI(100,100),COMBPR(400),VINTER(4)
C THIS PROGRAM COMPUTES THE PACKING FACTOR OF A SINGLE DRY PIGMENT.
C ULTIMATELY, IT WILL COMPUTE THE PACKING FACTOR OF A MULTI-PIGMENTED
C MIXTURE, AND THEN COMPUTE THE CPVC BASED UPON THIS AND THE DIL
C ABSORPTION VALUES OF THESE PIGMENTS
C
C NPIS = NUMBER OF PIGMENTS IN SYSTEM
C VADS (1) = VOLUME OF RESIN ADSORBED IN SINGLE PIGMENT
C VABS (1) = VOLUME OF RESIN ADSORBED IN SINGLE PIGMENT
C VINTER (1) = VOLUME OF REIN IN INTERSTICIES OF SINGLE PIGMENT
C PHIMIX = PH OF MIXTURE
C DIL = D.A. VALUE IN VOL DIL/VOL PIG
C TOTVOL (1) = TOT VOL OF THE ENTIRE PARTICLE DIST INCLUDING ADS LAY
C NSIZ (1) = NUMBER OF PARTICLE SIZE RANGES
C PIGPER (1) = PIGMENT VOLUME FRACTION
C ADSLAY = ADSORBED LAYER THICKNESS (IN MICRONS)
C NPART (1) = NUMBER OF PARTICLES
C SIZ (2) = DIAMETER OF PARTICLES
C VOL (1) = VOLUME OF ONE PARTICLE
C VOLSIZ (2) (RANGE,PIGMENT) VOL OF THE ENTIRE PARTICLE RANGE
C VOLPER (2) (RANGE,PIG) VOL FRACTION DISTRIBUTION
C PCK (1) PF OF SINGLE PIGMENT
C COMBSZ (1) MAKES SIZ INTO A 1 DIMENTIONAL ARRAY
C COMBPR (1) MAKES VOLPER INTO A 1 DIMENTIONAL ARRAY
C PHI (2) = MAY PF FOR A SINGLE PIGMENT
C TEND = TEMPORARY STORAGE OF PF TIMES VOL & DIST
C DIAMAT = DIAMETER RATIO
C PHIMIX = PF OF MIXTURE
C VINTP = VOL FRACTION INTERSTITIAL OF PIG COMBINATION
C VDS = VOL FRAC ADSORBED (PIG VOL & TIMES THE ADS VOL)
C VBS = VOL FRAC ADSORBED (PIG VOL & TIMES THE ABS VOL)
C
C READ IN THE PARTICLE SIZE DISTRIBUTION FOR THE PIGMENT
C AND CONVERT THIS INTO A VOLUME PERCENTAGE DISTRIBUTION
C
      PI=3.1415
      READ *, NPIS
      DO 2 II=1,NPIS
C
C INITIALIZE TO ZERO
C
      VADS(II)=0.0
      VABS(II)=0.0
      VINTER(II)=0.0
      PHIMIX=0.0
      DIL(II)=0.0
      TOTVOL(II)=0.0
      READ *, NSIZ(II),PIGPER(II),ADSLAY,DIL(II)
      M=NSIZ(II)
      DO 1 I=1,M
      READ *, NPART(II),SIZ(I,II)
      VOL(II)=PI*(SIZ(I,II)**2.0*ADSLAY)**3/6.0
      VOLSIZ(I,II)=NPART(II)*VOL(II)
1    TOTVOL(II)=TOTVOL(II)+VOLSIZ(I,II)
      DO 9 I=1,M
      VOLPER(I,II)=(VOLSIZ(I,II)/TOTVOL(II))*PIGPER(II)
9    CONTINUE
      M=0
      MQ=0

```

```

      NPIG1=NPIG+1
      DO 11 IA=1,NPIG1
      PCK(IA)=1.0
      IF(IA.EQ.NPIG1) GO TO 3
      MOF=NSIZ(IA)
      DO 13 K=1,MOF
      COMBSZ(K+M)=SIZ(K,IA)
12  COMBPR(K+M)=VOLPER(K,IA)
      MD=MD+NSIZ(IA)
      M1=M+1
      3  IF(IA.EQ.NPIG1) M1=1
C
C  COMPUTE PACKING FACTOR FOR PIGMENTS SEPARATELY AND THEN TOGETHER
C
      DO 8 I=M1,MD
      PHI(I,I)=0.639
      TEMP=0.0
      DO 70 J=M1,MD
      IF(I.EQ.J) GO TO 70
      IF(COMBSZ(I).LT.COMBSZ(J)) GO TO 20
      DIAMAT=COMBSZ(I)/COMBSZ(J)
C
C  THIS PART COMPUTES PHIMIX OF BINARY MIXTURE
C
      IF(DIAMAT.GT.15.0) GO TO 22
      PHIMIX=0.872459-0.307909*0.814182**DIAMAT
      GO TO 32
22  PHIMIX=.000022449*DIAMAT+.347551
32  PHI(I,J)=0.639+(PHIMIX-0.639)/(1.15-1.017*PHIMIX)
      GO TO 70
20  DIAMAT=COMBSZ(J)/COMBSZ(I)
      IF(DIAMAT.GT.15.0) GO TO 21
      PHIMIX=0.872459-0.307909*0.814182**DIAMAT
      GO TO 31
21  PHIMIX=.000022449*DIAMAT+.347551
31  PHI(I,J)=0.639+(PHIMIX-0.639)/(1.017*PHIMIX-0.15)
70  TEMP=TEMP+PHI(I,J)*COMBPR(J)
      IF(TEMP.LT.PCK(IA)) PCK(IA)=TEMP
8  CONTINUE
      IF(IA.EQ.NPIG1) GO TO 11
C
C  COMPUTE INTERSTITIAL, ABSORBED AND ADSORBED OIL AMOUNTS FOR
C  EACH PIGMENTS INDIVIDUALLY
C
      VADS(IA)=PI/6.0*((SIZ(I,I)+2.0*ADSLAY)**3-SIZ(I,I)**3)
      PCK(IA)=PCK(IA)/PIGPER(IA)
      VINTER(IA)=1.0-PCK(IA)
      VARS(IA)=DIL(IA)-VADS(IA)-VINTER(IA)
      WRITE 100,IA,PIGPER(IA),PCK(IA),DIL(IA),VINTER(IA),VARS(IA),
      VADS(IA)
      M=M+NSIZ(IA)
11  CONTINUE
100  FORMAT(/,*, PIGMENT NO.,I2,*, AT VOL. *,
      *,F5.4,*, PACKING FACTOR *,F9.6,/,T17,*O.A. VALUE **,
      *,F7.4,*, INTERSTITIAL DIL **,F7.4,/,T17,*ABSORBED OIL **,
      *,F7.4,*, ADSORBED OIL **,F7.4)
C
C  COMBINE ALL THE DIFFERENT PIGMENT OIL VALUES TOGETHER
C
      VNTR=1.0-PCK(NPIG1)
      VDS=VRS=C.0
      DO 5 I=1,NPIG
      VDS=VDS+PIGPER(I)*VADS(I)
      VRS=VRS+PIGPER(I)*VARS(I)
5

```

```

C
C  COMPUTE CPVC
C
      CPVC=1.0/(1.0+VNTR+VDS+VRS)
      WRITE 120,PCK(INPIG1)
120  FORMAT(//,T20,* PIGMENT COMBINATION*,//,* PACKING FACTOR **,
      $ F10.5)
      WRITE 300,VNTR,VDS,VRS,CPVC
300  FORMAT(* VOL. INTERSTITIAL **,F10.6,//,* VOL. ADS. **,F10.6,
      $ //,* VOL. ABS. **,F10.6,//,* CPVC **,F10.6)
      STOP
      END

```

INPUT DATA

2
 37 0.7 0.05 2.16
 7 0.5
 5 1.
 9 1.5
 30 2.0
 37 2.5
 50 3.0
 57 3.5
 72 4.0
 61 4.5
 72 5.0
 57 5.5
 61 6.0
 53 6.5
 37 7.0
 32 7.5
 31 8.
 23 8.5
 23 9.
 20 9.5
 14 10.
 14 10.5
 7 11.
 5 11.5
 8 12.
 6 12.5
 5 13.
 7 13.5
 3 14.
 2 14.5
 2 15.
 3 16.
 2 16.5
 1 17.5
 1 18.5
 1 19.
 1 24.5
 2 30.
 5 0.3 0.05 1.23
 1 0.1
 35 0.2
 143 0.3
 29 0.4
 1 0.5

SAMPLE OUTPUT

PIGMENT NO. 1 AT VOL. % .7000 PACKING FACTOR .964990
D.A. VALUE = 2.1600 INTERSTITIAL OIL = .3351
ABSORBED OIL = 1.8244 ADSORBED OIL = .0005

PIGMENT NO. 2 AT VOL. % .3000 PACKING FACTOR .540232
D.A. VALUE = 1.2300 INTERSTITIAL OIL = .3598
ABSORBED OIL = .3597 ADSORBED OIL = .0005

PIGMENT COMBINATION
PACKING FACTOR = .845139
VOL. INTERSTITIAL = .154861
VOL. ADS. .000524
VOL. ABS. 1.537969
CPVC = .371284

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