White Hiding Lead Pigments

I-A-c

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Consultant
NL Industries, Inc.
Hightstown, New Jersey 08520

I-A-c-1 BASIC LEAD CARBONATE

Pigment White 1 (77597)
Basic Carbonate White Lead (BCWL)
White Lead, Basic Carbonate
White Lead
Dutch White Lead
Berlin White
Flake White
Krems White
Silver White
Slate White

Chemical Formula $Pb_3O_8C_2H_2$
(MW 775.67)
$2PbCO_3 \cdot Pb(OH)_2$

Chemical Composition (wt %)

Pb	80.26
O	16.51
C	3.10
H	0.13
	100.00

In the early part of the twentieth century it was a common practice for the manufacturer of white lead to add his name to the product so that white lead carried many brand names other than those listed above. As consolidations in the industry took place, brand names were replaced by manufacturing process names. Dutch, Carter, Electrolytic, Chamber, and Precipitation Process typified the designations for white lead in the era of roughly 1915 to 1940. In today's market this pigment is called basic lead carbonate, basic carbonate white lead, and white lead. Basic lead carbonate is most representative.

Government specifications often allow partial substitution of some other lead pigments for white lead in certain paint formulas. This introduced names such as basic sulphate white lead and basic silicate white lead. Such practice has added confusion since these products are not constituted of basic lead carbonate. This dubious practice appears to be phasing out of the picture as newer lead pigments and lead coated silica pigments have been brought to market.

Chemically, basic lead carbonate has the formula $2PbCO_3 \cdot Pb(OH)_2$ (equivalent to a theoretical lead carbonate content of 68.9%). Actually the chemical compositions of basic lead carbonate pigments in the trade vary with the process of manufacture. The largest tonnages of white lead were originally produced by the old Dutch stack process which produced a lead car-

bonate content ranging up to 75% by weight. This meant it contained up to 15% of free or uncombined normal lead carbonate ($PbCO_3$). Some properties of the pigment suffer as the lead carbonate content is increased.

Contemporary white leads have a lead carbonate content in the range of 62 to 66%. These lower lead carbonate contents provide a finer particle size and impart higher tinting strength, hiding power, brightness, oil absorption, and paint consistency. These low carbonate white leads presumably consist of two different basic lead carbonates: $2PbCO_3 \cdot Pb(OH)_2$ (most prevalent) and $4PbCO_3 \cdot 2Pb(OH)_2 \cdot PbO$.

X-Ray diffraction patterns verify the presence of these two lead carbonates as well as the free normal carbonate. Each lead carbonate has a distinctive X-ray pattern.[1]

Some typical chemical compositions of the basic lead carbonate formed by various manufacturing processes[2] are given in Table 1.

TYPICAL PROPERTIES

	Basic Lead Carbonate	
	Carter-Tank Finished	Precipitated
Appearance	Fine white powder	Fine white powder
Density (g/cm³)	6.75–6.85	6.85–6.95
(lb/gal)	55.6	57.5
Optical		
Brightness, glycerine rub-up (%)	92	93
Tinting strength	160–190	220–250
Hiding power (ft²/lb)	20	25
Refractive index	1.94–2.09	—
Specific surface area, BET (m²/g)	1.6	—
Crystal nature		
System	Hexagonal	Hexagonal
Habit	Lamellar to flaky	Flaky
Oil absorption (lb/100 lb)	11	15

White leads are chemically active pigments. They react with both the free acidic

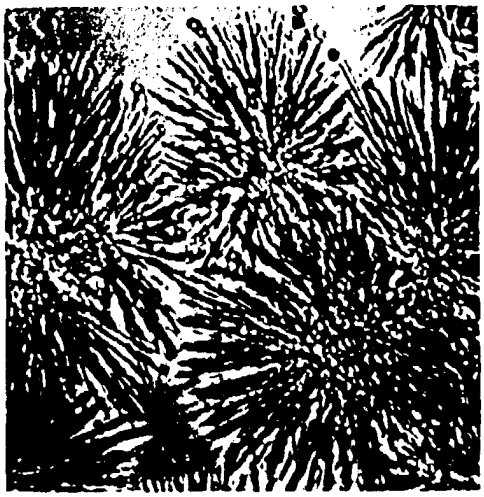

Fig. 1. White lead soaps (700×).

portions of vehicles and with the breakdown acids that develop from paint vehicles as paint films age. These reaction products shown in Fig. 1, which are called lead soaps,[3] reinforce the paint film. Fortunately these white lead reaction products are formed at a favorable (slow) rate that imparts the right type of plasticity to the paint film for good stabilization. Active white lead pigments also prevent syneresis or softening of the paint film. The beneficial rate of reaction with white lead helps to retain the film flexibility necessary for good adhesion and durability.

Legislation prohibiting the use of lead ingredients in amounts greater than 1% w of lead in interior paints (or exterior paints that are accessible to children) has been passed by several states. The Food and Drug Administration (1971) is considering ½%w as the top limit for total lead content. Such legal restrictions are discouraging the use of lead pigments in trade sales paints and to some extent in industrial paints.

ECONOMICS

The production and consumption of white lead pigments are quite dependent on the price of metallic lead. From 1926 to 1945 the New York price of metallic lead varied

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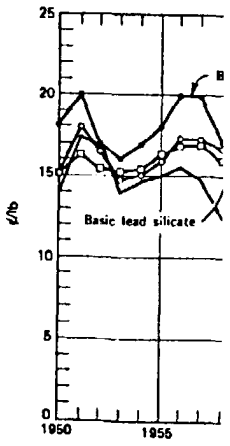

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Table 1 Chemical Composition of Lead Carbonate Pigments as Manufactured by Various Processes (%w)

	Dutch	Carter	Corrosion Tank Finished	Precipitated	Normal Lead Carbonate
PbO	86.3	86.6	87.2	88.0	83.5
CO_2	11.4	11.0	10.7	9.9	16.5
$PbCO_3$	69–74	66–68	64–67	62–64	100
Combined H_2O	2.3	2.34	2.1	2.03	—
Basic PbO	28.0	28.8	34.5	37.3	—

from 3½¢/lb to 8¢/lb. Figure 2 presents the New York prices of metallic lead for a subsequent period (1950–1968).[4] As the price of metallic lead soared from about 4¢/lb to more than 16¢/lb during this overall period, the use of white lead pigment declined correspondingly since it became increasingly uneconomical to use a high density, high price-per-pound material that had to be considered in terms of the *volume* of pigment necessary for a gallon of paint. However, the beneficial properties of white lead pigments still warrant their use in paints for specific purposes. An inspection of Fig. 2 shows the relation between metallic lead and basic lead carbonate pigment prices.[4,7] At present (1971) white lead is 21½¢/lb (it has reached a high of 23.1¢/lb).

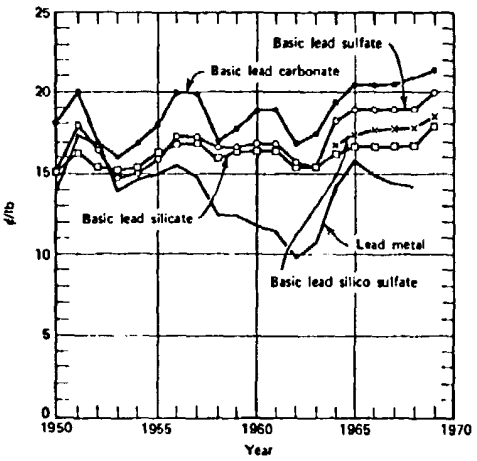

Fig. 2. United States pricing pattern for selected basic lead pigments (1950–1969). Pricing trend for lead metal also given for the same period. Basic lead carbonate was priced at 8½¢/lb and lead metal at 8¢/lb in 1946.

Figure 3 illustrates the estimated United States consumption pattern for basic lead carbonate pigment for 1968 in terms of white lead shipments.[6]

The production of *all* lead pigments now approximates only 160 million lb whereas in the early part of the twentieth century well over 400 million lb of lead pigment were produced annually in the United States.[5]

As shown in Fig. 4, the consumption of basic lead carbonate has also fallen from about 90 million lb in 1950 to a low of 24 million lb in 1968. Imports of white lead approximate 4,000,000 lb/yr, but ordinarily exports pretty well balance out imports.

White lead in oil was the backbone of the exterior house paint business up until about 1940. By that time both titanium dioxide and zinc pigments had established their merits and two-coat systems were initiated.

However, the use of basic lead carbonates on a reduced scale has still persisted. The use of basic lead carbonate is still showing a decline and, in view of the present concern with toxic materials,[8,9] this trend is not likely to be reversed.

HISTORICAL BACKGROUND

One of the earliest descriptions of white lead is given by Theophrastes[10] (originating in the fourth century B.C.). Similar data relative to the production of white lead, then known to the Greeks as "Psimithium" and to the Romans as "Cerusa," are found in Pliny[11] and Vitruvius.[12]

The white lead industry began to expand

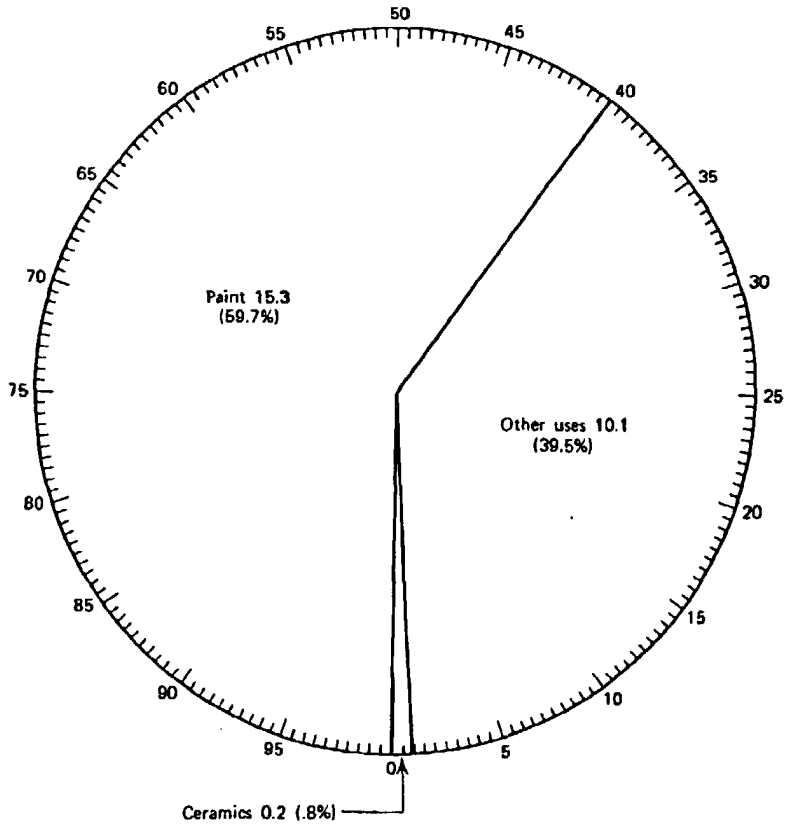

Fig. 3. Estimated United States consumption pattern for basic lead carbonate pigment (million lb) for 1968. Total: 25.6 million lb.

tremendously in the early part of the seventeenth century due to the introduction of the Dutch stack process. Subsequently, various processes to manufacture white lead were developed such as the German chamber process, the French precipitation process, and the Carter, Electrolytic, Euston, and Thompson-Stewart processes. Many variations of these processes were also adopted.

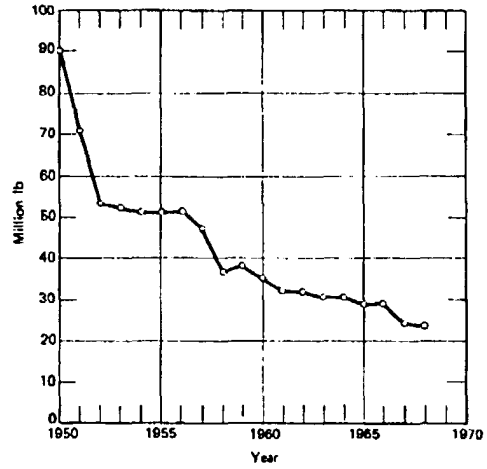

Fig. 4. Annual United States shipments of basic lead carbonate for the period 1950–1968.

In the early part of the twentieth century, white lead production was at an all time high of approximately 340,000,000 lb/year.[5] Approximately 40 plants manufactured white lead in the United States by the Dutch stack process. When the price of lead doubled in the late 1940s, the Dutch stack process became obsolete since it was uneconomical to operate.

The Carter process for making white lead from atomized lead was pursued as the Dutch process was phased out of production. The manufacture of white lead from a leady litharge was introduced about 1940 and finally a combination process of corrosion-tank finishing became the main

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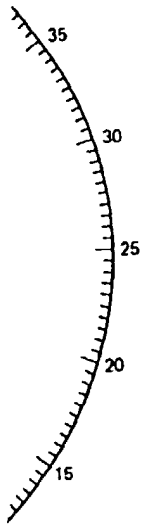

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method of manufacture. Along with this, a small amount of white lead is made by the precipitation process using litharge as a starting material (used primarily for the dry white lead pigment business).

MAJOR REASONS FOR USE

Basic lead carbonate is one of the most foolproof pigments to use in linseed oil base paints.[13] Three of its more beneficial properties are its slow rate of reactivity with acidic components of vehicles (thereby imparting good stabilization), its ability to impart adhesion, and its unusual ease of brushing. Oil-based primers for wood normally have a goodly portion of white lead present. White lead contributes hiding power, possesses a high affinity for linseed oil, is readily wet (and easily flushed to give lead-in-oil compositions), provides a high degree of absorption for near-UV light below 310 nm, and imparts some fungicidal properties. It lowers moisture vapor transmission, imparts blister resistance, and keeps water solubles at low levels. Since white lead is a film building pigment, it is many times too durable during the first year of weathering to provide good self-cleaning by slow chalking. For this reason, a portion of a chalking grade of titanium dioxide is often used with basic lead carbonate in linseed oil base paints to overcome this deficiency.

Since lead sulfide (PbS) is black, basic lead carbonate paints tend to become stained black when exposed to industrial environments with hydrogen sulfide (H_2S) present.

PIGMENT MANUFACTURE

In today's economy there are primarily two processes used for the manufacture of white lead: the corrosion-tank finishing process and the precipitation process. In the corrosion-tank finishing process a leady oxide is charged into slowly revolving cylinders.[14] Moisture is sprayed on the surface of the charge (about 3 tons of material) as the cylinder slowly rotates. Catalytic quantities of acetic acid are also automatically sprayed on the charge as new surfaces are exposed. Carbon dioxide from flue gas is metered into the center of the rear end of the cylinder. The moisture activates the metallic lead particles, helping to oxidize them to litharge, and the carbon dioxide from the flue gas reacts to form the basic lead carbonate. These reactions heat up the charge to about 185°F. After about 2½ days in this corrosive steaming atmosphere, the charge is balled up into small lumps and at this point it is emptied, disintegrated, and water classified. The classified product is pumped to large circular reaction tanks where the final stage of carbonation is achieved. Flue gas is pumped into the bottom of these reaction tanks while the slurry is agitated with a strong turbine mixer. The final carbonation takes about ½ day, so that the batch is completed in a total of about 3 days.

The second process for the manufacture of white lead in the present market is the precipitation process.[15] A litharge slurry is fed to a large turbine agitated mixing tank and oxidized by blowing air into the slurry to eliminate any metallic lead (if present). When the metallic lead is eliminated, flue gas is pumped into the slurry to carbonate the product, usually to a carbonate content of 64 to 66%. This is a short-time reaction, taking less than 1 day for completion. Time of completion is dependent on the size of the batch being carbonated. Instrument control is very important for this process.

PIGMENT GRADES

There are primarily two grades of basic lead carbonates marketed. One is based on the corrosion-tank finishing process and the other on the precipitation process. The greater volume of white lead is made by the corrosion-tank finishing process. In both processes particles are generally formed with a hexagonal outline. If the particles are allowed to grow during processing, the hexagonal outline is generally well established. The controls in processing nor-

Fig. 5. Photomicrographs (945×) of normal lead carbonate (left), corrosion-tank finished H.T.S. White Lead (center), and precipitated white lead (right).

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Fig. 5. Photomicrographs (945X) of normal lead carbonate (left), corrosion-tank finished H.T.S. White Lead (center), and precipitated white lead (right).

mally keep the particles fine so as to develop better optical and physical properties. The hexagonal outline is not always well developed with finer particle size.

Micrographs[2]

The third or depth dimension of a basic lead carbonate particle is much less than the other two dimensions that are visible under the microscope. White lead particles are decidedly flaky. The coarser particles may be a little thicker than the finer ones. A flaky particle imparts the effect of a much finer particle size than is indicated from the two dimensions observable under the microscope. Photomicrographs at a magnification of 1000 diameters of the two types of white lead and normal lead carbonate are illustrated in Fig. 5. The particles of H.T.S. (high tinting strength) White Lead have a pronounced hexagonal outline. Their particle size is a little coarser than in the precipitated white lead. From the thickness of the contour band (the dark line outlining the edge of the particle) it is evident that the particles are inclined to be a little thicker than the high basicity precipitated white lead.

By comparing the micrographs of Fig. 5 it is evident that the particles in precipitated white lead are less distinct, more indefinite, irregular in shape, of finer particle size, and possess a thinner third or depth dimension. Because of these characteristics, the precipitated white lead normally has a higher tinting strength, higher hiding power, higher oil absorption, and higher paint consistency.

Normal lead carbonate particles usually grow to a larger particle size than the basic lead carbonate particles. They are prismatic in shape, as shown in Fig. 5, and their crystal habit would be classed as chunky. Their crystal outline is usually quite well defined.

White lead particles wet easily with linseed oil and there is little tendency for particles to aggregate or produce a rough textured film from agglomerates. Agglomerates need to be above approximately 15 μ in size for the eye to detect their presence in a film.

Cumulative Particle Size Distribution Curve[2]

Figure 6 gives the cumulative particle size distribution curve for H.T.S. White Lead particles as measured from two dimensions of the particles. The average diameter by surface mean is 1.1 μ. This is an ideal average diameter to use for comparison of many types of materials. White lead might be made of a little finer particle size for higher optical properties. However, it is a little difficult to achieve because of the general tendency of basic lead carbonate

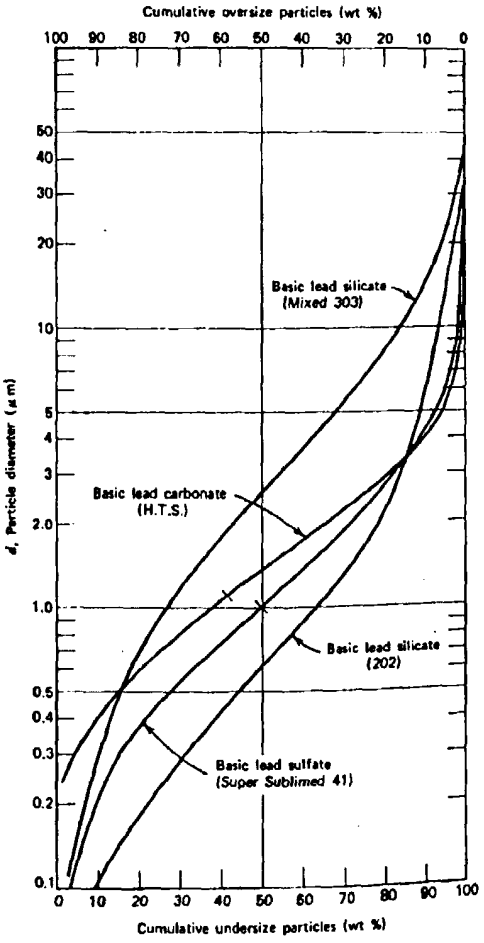

Fig. 6. Cumulative particle size distribution curves for selected basic lead pigments (white hiding types).

particles to grow during precipitation and carbonation.

The range of particle size is fairly broad, which allows tight packing of the particles for a structurally strong paint film. The flaky character of the particles impart body and paint consistency. The number of particles in 1 cm^3 (solid) approximates 3.6 trillion. In general, the higher the number of particles per cubic centimeter, the higher the hiding power, tinting strength, and oil absorption. A particle size distribution curve for precipitated white lead would be of slightly finer particle size than for the H.T.S. White Lead.

Spectral Reflectance Curves[2]

The spectral reflectance curves for two commercial grades of basic lead carbonate are given in Fig. 7 for both the visible and the near-UV range. These curves were obtained from the dry packed powders.

The reflectance curves for the two types of white lead are quite similar, with relatively high reflection of UV light (over 80%) through the 310 to 400 nm range. Below 310 nm the UV reflection falls off rapidly (becoming less than 10% below 270 nm). From 210 to 270 nm, white lead is a strong absorber of UV light.

In the visible range of the spectrum, from 400 to 700 nm, white lead provides very high reflectance, making it a good white pigment (most reflectance values are above 97%).

Over the near-IR range, white leads have relatively high reflection values compared to magnesium oxide (set at 100% reflection).

PIGMENT SPECIFICATIONS AND MANUFACTURERS

Table 2 lists the basic lead carbonate pigments that are currently manufactured by

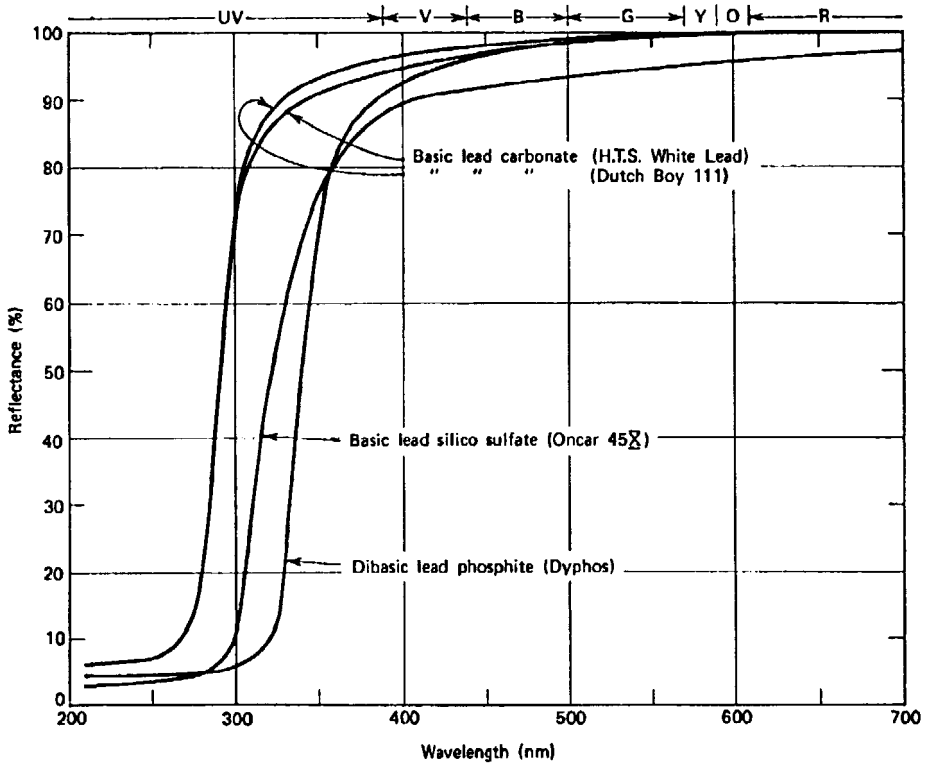

Fig. 7. Spectral reflectance curves for selected basic lead pigments in the near UV and visible spectrums. Data courtesy Titanium Pigments Division, NL.

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Table 2 Commercial Pigments Meeting Two Standard Specification for Basic Lead Carbonate

Company Coding	ASTM D81-43(55) or Federal TT-W-00251g
BNK	AAA
EP	E-PA
	E-PAAA
HMD	HLP-A
	HLP-AA
	HLP-AAA
MGL	Scotch Laddic
NL	Dutch Boy HTS
	Dutch Boy 111

the five major United States producers to meet two industrial specifications.[16]

Regulations on Lead Content

Prior to the middle 1930s, paints for interior surfaces contained a high lead content. During the 1930s the more economical titanium dioxide/extender pigments began to replace white lead pigments for interior application. Subsequently a health-hazard problem arose in that occasional lead poisoning in children was traced to the chewing and eating of paint film chips from the walls of improperly maintained old buildings in slum areas.

Discussion of this situation led to the establishment in 1955 of the voluntary standard of the American National Standards Institute (Z66.1). This standard limited the lead content in paints intended for childrens' toys, furniture, and interior surfaces to a maximum of 1.0% lead based on the total paint solids.

More recently the Food & Drug Administration (FDA), U.S. Department of Health, Education, and Welfare, issued a proposed regulation that would declare certain heavy-metal-containing paints as hazardous substances that require special labeling or warning for child protection under the Federal Hazardous Substance Act. This proposal reduces the acceptable level of lead (as metal) in certain paints to 0.5% of the nonvolatile content of the paint. It also limits the levels of antimony, arsenic, cadmium, mercury, and selenium in the nonvolatile coating portion to 0.05%, individually or in total (reduction from the earlier 0.06% limit specified by the ANSI Standard Z66.1 of 1964). The acceptable level of water-soluble barium at 1.0% of the barium present is reaffirmed. It is expected that this FDA regulation will become operational in late 1972.

These rulings have caused changes in certain paint formulations and will result in further changes in the future. However, the present major uses of lead pigments in primers and finishes for *exterior* house surfaces and for various metal protective systems for *outside* use have not been essentially affected by this legislation except for the requirement of a warning label. These regulations will supersede any state requirements.

I-A-c-2 BASIC LEAD SULFATE

Pigment White 2 (77633)
Basic Sulfate, White Lead
Sublimed White Lead
Super Sublimed White Lead
Bartlet White Lead
Lewis White Lead

Chemical Formula (Mixture)
Normal lead sulfate
$PbSO_4$ (MW 303.27)
Monobasic lead sulfate
$PbSO_4 \cdot PbO$ (MW 526.48)

Chemically, basic lead sulfate is a broad term that embraces many compounds with varying degrees of basicity above the normal (nonbasic) lead sulfate. The lowest basicity lead sulfate (stoichiometric proportions) is monobasic lead sulfate with 42.4% combined or basic PbO present. The lead sulfate pigment marketed to the paint industry has a basic or combined PbO content usually in the range of 15 to 28%.[2] Therefore, the usual basic lead sulfate that

is sold is a mixture of normal lead sulfate and monobasic lead sulfate.

Government specifications allow the substitution of basic lead sulfate for basic lead carbonate in certain paint formulas. This has introduced pigment names such as basic sulfate white lead and basic silicate white lead even though these products do not contain white lead (basic lead carbonate). It is expected that such names may be phased out of paint terminology as new and more specific lead pigments are brought to market.

Blue Basic Lead Sulfate

A variant of the white basic lead sulfate pigment is the older product called blue basic lead sulfate that was and still is marketed as a metal protective pigment. Blue basic lead sulfate is a mixture of compounds consisting of approximately 78% of monobasic lead sulfate, 10% lead sulfide (PbS), 4% lead sulfite ($PbSO_3$), 4% zinc oxide (ZnO), plus 4% carbon and undetermined material. The use of blue basic lead sulfate as a metal protective pigment seems to be rapidly declining. A concise survey of this slate-gray pigment is given by Rose.[17]

TYPICAL PROPERTIES[16]

	Basic Lead Sulfate
Appearance	White powder
Density (g/cm³)	6.4
(lb/gal)	53.3
Bulking value (gal/lb)	0.0188
Brightness in oil (%)	83.0
Oil absorption (lb/100 lb)	8.0
Screen analysis through 325 mesh (%)	99.9

ECONOMICS

The price of basic lead sulfate pigment tends to vary with the price of metallic lead. During the 1950s it varied by about 3¢/lb from an average price of 16½¢/lb. During the 1960s it averaged 18½¢/lb and in 1971 (May) was quoted at 21½¢/lb.[7]

Annual United States consumption of basic lead sulfate pigment by the paint industry has declined from about 10 million lb in 1965 to less than half this amount in 1970. In view of the concern relative to lead toxicity (as contributed by paints), further erosion in sales can be anticipated.[8,9]

HISTORICAL BACKGROUND

White basic lead sulfate was first manutured about 100 years ago. It was obtained as a portion of zinc oxide pigment because in the early days both zinc oxide and lead sulfate pigments were made directly from the ores by sublimation using controlled atmospheres.

At that time a by-product was obtained during the lead ore smelting that was called blue lead and basic sulfate blue lead. Subsequently it was made under better controlled furnace conditions to produce a white powder having pigment properties. In recent years the demand for the basic lead sulfate product has declined.

In 1935 a new basic lead sulfate was made by a precipitation process[18] that gave the pigment improved optical and physical properties and higher basicity. However, it was not particularly economical and was taken off the market about 1960.

MAJOR REASONS FOR USE

White basic lead sulfate is used only in multiple or mixed pigment formulations. Its low basicity, and therefore low reactivity, makes it a good pigment to mix with zinc oxide. Leaded zinc oxides may contain up to 50% basic lead sulfate and a popular level is 35% (35% leaded zinc oxide). Because of its lower density compared with basic lead carbonate, basic lead sulfate is often substituted for the carbonate pigment. Basic lead sulfate is used in both prime and finish coats.

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PIGMENT MANUFACTURE

White basic lead sulfate is currently produced by a fume process. Molten lead is atomized into a jet flame in the presence of excess air and sulfur dioxide to form the lead sulfate. The lead sulfate forms a fume that is drawn off through a long cooling system by suction to a bag house where it is collected (the bags are periodically shaken to relieve the pigment so that it falls into bins for packaging).

PIGMENT GRADES

Only one grade of white basic lead sulfate is marketed (Super Sublimed E-P No. 41) by EP. Its chemical composition is given in Table 3.

An electron photomicrograph (1000 ×) of basic lead sulfate pigment is given in Fig. 8 and reveals the pigment particles as primarily roundish or irregular in shape, transparent, and characterized by a slightly roughened surface.[2]

The cumulative particle size distribution curve for the commercial basic lead sulfate pigment is given in Fig. 6.[18] The median diameter is 1 μm and the particles are reasonably uniform in size.

When mixed with linseed oil, basic lead

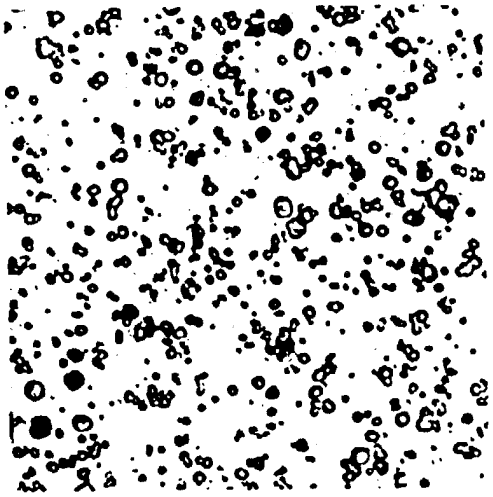

Fig. 8. Basic lead sulfate (1000×).

sulfate exhibits an average brightness of 83% across the visible spectrum. This makes it a good white pigment for house paints. Below 290 nm, basic lead sulfate has a low reflectance and acts primarily to absorb UV light.

PIGMENT SPECIFICATIONS AND MANUFACTURERS

There are three main specifications for the procurement of basic lead sulfate:

Military	MI 23-46
ASTM	D82-44 (1955)
Federal	TT-W-261c

All are met by Super Sublimed E-P No. 41 produced by the one United States manufacturer of this pigment (EP).[16]

Table 3 Chemical Composition of a Commercial Basic Lead Sulfate Pigment (EP)

	$PbSO_4$ (%)	Combined or Basic PbO (%)
Normal lead sulfate (theoretical)	100.0	0.0
Super Sublimed E-P No. 41 (commercial)	83.5	16.5
Monobasic lead sulfate (theoretical)	57.6	42.4

I-A-c-3 BASIC LEAD SILICATE

Pigment White 16 (77625)
Basic Silicate White Lead

Chemical Formula, Representative
$Pb_3Si_2H_2O_8$ (MW 807.8)
$3PbO{\cdot}2SiO_2{\cdot}H_2O$

Chemical Composition, (wt %)		Representative	
Pb	77.00		
Si	6.95	PbO	83.0%
H	0.25	SiO_2	14.8%
O	15.80		
	100.00		

Basic lead silicates that are used for pigmentary purposes are generally variations of monobasic lead silicate. A wide range of compositions may be produced, but commonly they have a molecular composition close to that given above. The commercial *mixed* lead silicate pigment, however, usually has a much higher silica content as given in Table 4.

Table 4 Chemical Composition of Commercial Basic Lead Silicates

	PbO (%)	SiO_2 (%)
High Lead (E-P 202)	83–85	14–16
High Silica (E-P 303)	49	51

X-Ray diffraction analyses show these two products to be quite different. The hydrated lead silicate pigment (E-P 202) has a characteristic pattern that distinguishes it from the mixed pigment (E-P 303) containing the free silica. The National Bureau of Standards has published an article describing the various lead silicates that have been prepared as definite compounds.[20]

TYPICAL PROPERTIES

	Basic Lead Silicates	
	E-P 202 (Low Silica)	E-P 303 (High Silica)
Appearance	White powder	White powder
Density (g/cm³)	6.4	4.0
(lb/gal)	53.3	33.3
Bulking value (gal/lb)	0.0188	0.030
Brightness in oil (%)	86.0	
Oil absorption (lb/100 lb)	16	13
Screen analysis through 325 mesh (%)	99.5	99.9

ECONOMICS

Over a period of many years the basic lead silicate pigments, in general, have been priced at slightly lower levels than the basic lead sulfate pigments. The price of basic lead silicate has remained relatively stable for a 20-year period (1950–1969) and as with the other lead pigments has paralleled the pricing pattern for metallic lead as shown in Fig. 2.

As of early 1971 the high lead (85% PbO content) silicate pigment was quoted at 24¢/lb whereas the low lead (48% PbO content) silicate pigment was priced lower at slightly less than 20¢/lb. These prices compare with a price of 21½¢/lb quoted for basic lead sulfate at that time.

HISTORICAL BACKGROUND

In 1942 a United States patent[21] was issued disclosing the manufacture of lead silicate pigments. Hence these pigments represent relatively new products that were first marketed in the United States in the early 1940s. The *mixed* basic lead silicate variant was introduced in the early 1950s.

The first basic lead silica pigment was promoted as a stabilizer for vinyl chloride

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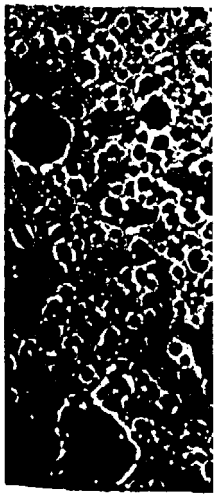

Fig. 9. Photo
303 (right).

E. J. Dunn, Jr.

ES

Basic Lead Silicates	
E-P 202 Low Silica)	E-P 303 (High Silica)
White powder	White powder
6.4	4.0
53.3	33.3
0.0188	0.030
86.0	
16	13
99.5	99.9

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plastics since it was particularly suitable for use in insulated polyvinyl chloride wire coating formulations where it contributed to high electrical resistivity. Subsequently it was adapted for utilization as a white hiding pigment. Research and practical experience has shown that basic lead silicates act to provide durable films. Around 1950 the mixed lead silicate pigment[18] comprising about 51% silica and 49% basic lead silicate was brought on the market. Both products, however, are referred to as basic silicate white leads.

MAJOR REASONS FOR USE

Basic lead silicates are reactive pigments that react with acidic components and breakdown acids and thereby act to stabilize industrial compositions (paint films, polyvinyl chloride plastics).

They are low tinting strength whites and have low hiding power and hence their contributions in these areas are of secondary importance.

Basic lead silicates tend to produce durable paint films since they impart resistance to chalking (although photo-sensitivity is occasionally a problem). Basic lead silicate is helpful in water-base primers for wood by reducing and controlling cedar and redwood staining. This high lead content pigment is also claimed to confer a degree of corrosion resistance to ferrous substrates.[22]

The mild reactivity of basic lead silicate helps to stabilize linseed oil films. This pigment is also an effective stabilizer for polyvinyl chloride plastic compositions.

PIGMENT MANUFACTURE

Lead silicate pigments are prepared by fusing silica and litharge at a temperature of approximately 1800°F. When the fusion is complete, the fluid melt is allowed to run from the furnace into a large volume of water which granulates the product. The granulated compound is wet ground in a ball mill for 24 hr, reducing it to pigmentary particle size. The ground slurry is removed from the ball mill, flocculated, dried, and pulverized. The pigment can be dried at high temperatures since it is not heat sensitive (although it is important to retain some water of hydration).

PIGMENT GRADES

There are essentially only two grades of basic lead silicate for the paint trade as listed under the section on Typical Proper-

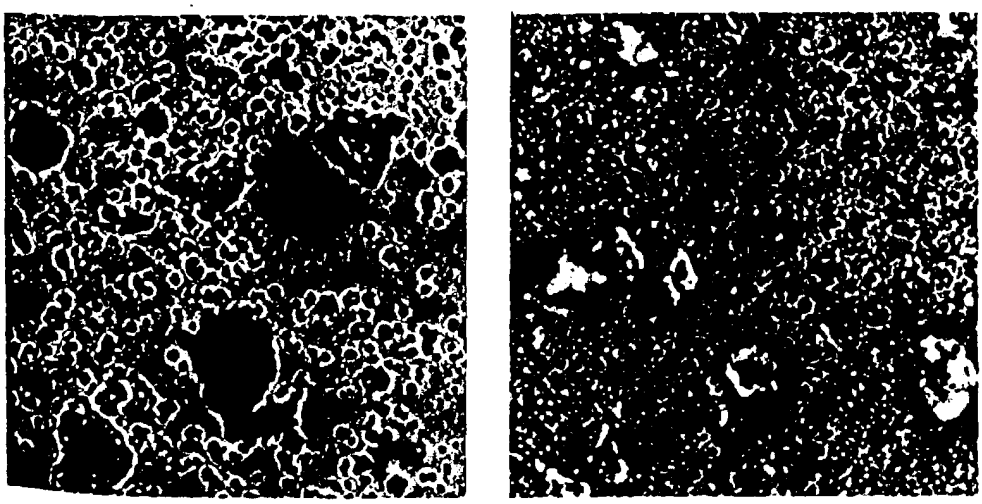

Fig. 9. Photomicrographs (1000×) of Basic Lead Silicate 202 (left) and Mixed Lead Silicate 303 (right).

ties. A third grade (E-P 201) is made primarily for the stabilization of plastics.

The photomicrographs (1000×) shown in Fig. 9 illustrate the appearance of two types of basic lead silicate pigment. In general these pigments have a coarser particle size than either the basic lead carbonate or the basic lead sulfate pigments. This is shown by making a comparison of the percent comulative oversize particles above 10 μm among the three basic lead pigments in Fig. 6.

PIGMENT SPECIFICATION AND MANUFACTURERS

There are no industry specifications for the procurement of basic lead silicate pigments.

The only United States manufacturer of lead silicate pigment is Eagle-Picher Industries, Inc.

I-A-c-4 BASIC LEAD SILICO SULFATE

Oncor 45X
Coated Silica Pigment
Basic Silicate White Lead

Basic lead silico sulfate is a coated silica pigment comprising three chemical compounds; a core of silica (SiO_2) and a coating (mixture) of gamma tribasic lead silicate ($PbSiO_3 \cdot 3PbO$) and monobasic lead sulfate ($PbSO_4 \cdot PbO$).

Chemical Composition, Overall (wt %)

PbO	47.9
SiO_2	47.9
SO_3	4.2
	100.0

Basic lead silico sulfate pigment is unique in that it has a core of silica and a coating of mixed basic lead salts.[23] It was the first in a series of such pigments developed for the paint industry.

TYPICAL PROPERTIES

	Basic Lead Silico Sulfate
Appearance	White powder
Density (g/cm³)	4.0
(lb/gal)	33.3
Bulking value (gal/lb)	0.030
Hiding power (ft²/lb)	10
Tinting strength	70
Oil absorption (lb/100 lb)	15

The composition of basic lead silico sulfate is roughly equivalent to ⅓ gamma tribasic lead silicate, ⅓ monobasic lead sulfate, and ⅓ silica (core). X-Ray diffraction patterns demonstrate the presence of silica and verify the formation of both the gamma tribasic lead silicate and the monobasic lead sulfate.[1] Microscopical analyses confirm the presence of the lead compounds as a strongly adherent composite coating on the silica core.

ECONOMICS

Basic lead silico sulfate was designed as an economical lead-base white pigment.[24] Pigments are formulated in paint compositions primarily on a volume basis and the usual substitution of one pigment for another is also on a volume basis. Thus a formulation requiring 170 lb of basic lead carbonate (6.8 g/cm³) requires only 100 lb of basic lead silico sulfate (4.0 g/cm³) on a volume replacement basis. This substitution represents a tremendous saving in raw material cost since not only are fewer pounds used but the price per pound is also lower. Clearly basic lead silico sulfate is more economical than the more dense (higher specific gravity) basic lead sulfate or basic lead carbonate pigments.

From 1950 to 1963 the price of basic lead silico sulfate was substantially the same as the basic lead silicate. From 1964 to 1969

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the basic lead silico sulfate pigment was priced somewhat higher as shown in Fig. 2 (17¢/lb to 18½¢/lb).[2,7]

HISTORICAL BACKGROUND

Basic lead silico sulfate is a relatively new lead pigment that was first marketed in 1948. Research had established about 1930 that the stabilizing reactions of pigments in paint films were primarily surface reactions as evidenced by microscopical techniques[3] and chemical analyses.[25] The natural sequence to these findings was the development of solid phase reactions to produce pigments consisting of an inactive core (such as silica) and a reactive outside coating or shell (such as an inorganic active lead salt).

Basic lead silico sulfate represented a pioneer pigment where the gamma tribasic lead silicate was put in the surface coating (over the silica core) to impart durability to a paint film and monobasic lead sulfate was added to stabilize the whiteness of the lead silicate.[23]

Basic lead silico sulfate (Oncor 45X/NL) was manufactured commercially in the late 1940s so that experience with this pigment in the field is now over 20 years. Practical exposure has confirmed that basic lead silico sulfate is a good durable pigment with its greatest utilization developing in multipigment house paints (both water-base and solvent-base types).

MAJOR REASONS FOR USE

Aside from cost savings, basic lead silico sulfate is used for the many benefits it confers to paint systems.

It is unique in that it reduces the bleedthrough of redwood and cedar stains in latex base paints.[26] The water from latex paints penetrates into redwoods, dissolves the stain, and then immediately bleeds back through the paint film and badly discolors it. The basic lead silico sulfate may allow some discoloration of the *primer* coat but it prevents the stain from progressing into the finish or subsequent coats. If severe bleeding is evident in the prime coat, a safe practice is to apply a second coat of primer before applying the finishing coat(s).

The commercial basic lead silico sulfate (Oncor 45X) mixes readily with most colors and provides good stabilizing properties. It can replace basic lead carbonate in oil-base primers and basic lead sulfate in multipigment paints (whether the basic lead carbonate is present as such or as a part of leaded zinc oxide).

Basic lead silico sulfate pigment is the most versatile and economical pigment for formulating white or light tint exterior coatings (including primer and finish coats for both oil- and water-base coatings). Since it has the lowest density (specific gravity) of all the active white (hiding) lead pigments. its utilization results in substantial cost savings.

The chemical activity of basic lead silico sulfate is similar to that of basic lead carbonate. Thus it stabilizes by forming lead soaps with acidic components and degradation products that normally would soften a paint film. The low tinting strength of this pigment materially aids the formulation of a wide range of colored paints.

PIGMENT MANUFACTURE

Basic lead silico sulfate is manufactured from a mixture of silica, litharge, and sulfuric acid. The silica is wet ground overnight in a ball mill. The ground slurry is then pumped to a reaction tank containing a slurry of litharge. Acetic acid in catalytic amounts (0.1% wt) is added to the slurry. Dilute sulfuric acid is prepared and slowly added to the litharge/silica slurry mixture to form the required amount of monobasic lead sulfate. Mixing is continued for an additional hour after all the sulfuric acid has been added to insure homogeneity. The precipitate is filtered, dried, and calcined in a rotary kiln to produce the lead-coated silica particle. The calcined product is disintegrated and packed in 50 lb bags for the trade.

PIGMENT GRADES

There are two grades of basic lead silico sulfate pigment for the paint industry (Oncor 45X Green Code and Red Code).

The Green Code product is the more broadly used pigment. The Red Code, designed for blister-resistant wood primers, has 4.0% P_2O_5 substituted for the 4.2% SO_3 in the Green Code grade. Approximate average compositions for the two grades are given in Table 5.

The particle size characteristics of the two Oncor 45X pigments are similar. The particle size is very dependent on the particle size of the ground silica used for the particle core. The average surface mean diameter is 5 to 6 μ.

Table 5 Average Chemical Composition of Basic Lead Silico Sulfate Pigments (wt %)

	Oncor 45X(NL)	
	Green Code	Red Code
PbO	47.9	48.0
SiO_2	47.9	48.0
SO_3	4.2	—
P_2O_5	—	4.0

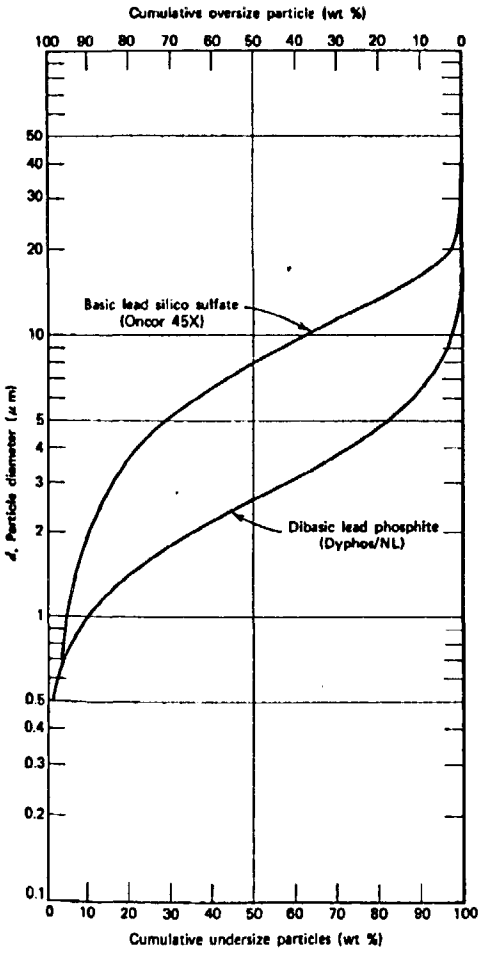

Fig. 10. Cumulative particle size distribution curves for basic lead silico sulfate (Oncor 45X/NL) and dibasic lead phosphite (Dyphos/NL).

The cumulative percent distribution curve for the basic lead silico sulfate pigment particles as made by the microscopical projection method is given in Fig. 10. The specific surface area of this Oncor 45X sample is 1.2 m^2/cm^3.

Figure 11 is a photomicrograph (1000×) illustrating the appearance of the uncoated and coated silica pigments. The reaction of the uncoated silica to form a coated product is quite evident.[27] The crystal habit of both pigments would be classed as irregular or chunky.

The spectral reflectance characteristics of basic lead silico sulfate (Oncor 45X) are graphed in Fig. 7 and show that this pigment absorbs UV light up to 285 nm. Above that, the reflectance rapidly increases to 76% at 360 nm and on up to 87% at 400 nm. In the visible range it provides an average reflectance of 94% but falls a little lower in the blue wavelength region. In the near-IR it is as highly reflective as magnesium oxide.

PIGMENT SPECIFICATIONS AND MANUFACTURERS

There are no standard industrial specifications for the procurement of basic lead silico sulfate. It is manufactured and sold only by NL Industries, Inc. as Oncor 45X.

Fig. 11. Phc original uncoa

I-A-c-5 DIBA

Dibasic Lead Pho
Dyphos

Chemical Formula
$Pb_3PO_{5.5}H_2$ (M
$2PbO \cdot PbHPO_3$

Chemical Compos

Pb	8
P	
O	1
H	
	10

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quite close to its
90.2–91.7%; P 3.
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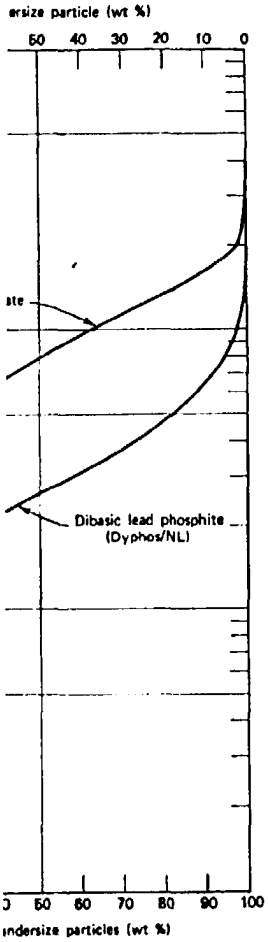

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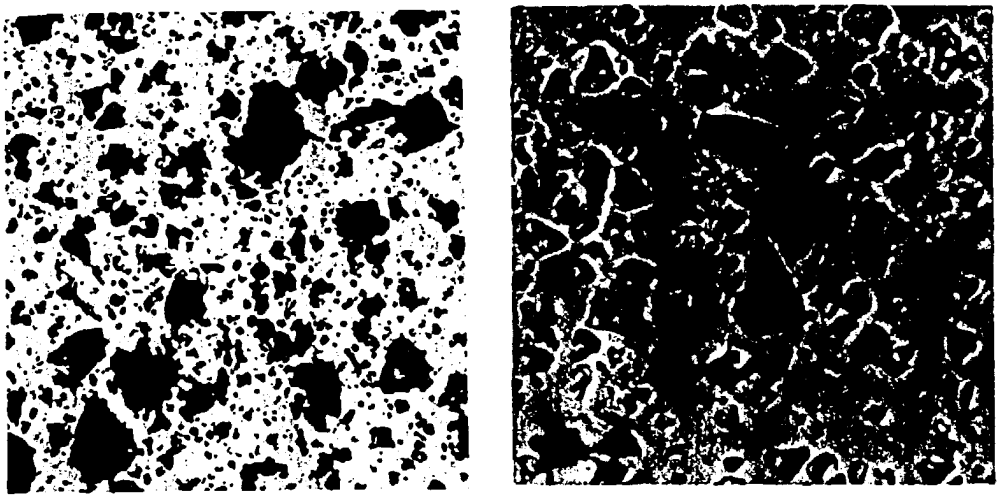

Fig. 11. Photomicrographs (1000×) of basic lead silico sulfate (Oncor 45X) (left) and original uncoated silica (right).

I-A-c-5 DIBASIC LEAD PHOSPHITE

Dibasic Lead Phosphite PG
Dyphos

Chemical Formula
$Pb_3PO_{5.5}H_2$ (MW 742.7)
$2PbO \cdot PbHPO_3 \cdot \frac{1}{2}H_2O$

Chemical Composition (wt %)

Pb	83.7	
P	4.2	PbO 90.2%
O	11.8	
H	0.3	
	100.0	

Dibasic lead phosphite PG (pigment grade) is the dibasic lead salt of phosphorous acid. Its commercial chemical composition is quite close to its theoretical formula (PbO 90.2–91.7%; P 3.7–4.5%; moisture loss at 105°C, 0.3% maximum).

TYPICAL PROPERTIES

	Dibasic Lead Phosphite
Appearance	White powder
Density (g/cm³)	6.67
(lb/gal)	55.5
Tinting strength, minimum	250
Refractive index	2.25
Hiding power (ft²/lb)	38
Oil absorption (lb/100 lb)	14
Average diameter surface mean (μm)	2.5

ECONOMICS

Dibasic lead phosphite has remained very stable in price over the past 20 years (within a price range from 56¢/lb to 59¢/lb from 1950 to 1970). It was first used for the stabilization of plastics but its effective anticorrosive properties and its ability to eliminate the bleed-through of cedar and redwood stains in emulsion primers have since broadened its use. It is presently produced by three manufacturers.

HISTORICAL BACKGROUND

Dibasic lead phosphite was patented in 1949[28] and marketed shortly thereafter. Its early use was primarily as a stabilizer for vinyl-type plastics with a chlorine content. This use has expanded with the years.

Since basic lead phosphite is basic in nature, it is an active pigment that stabilizes films in a manner similar to other basic lead pigments. Its anticorrosive properties are good and this white pigment is being utilized in increasing amounts in light or pastel colored anticorrosive primers and finishes (especially useful on highway guard rails).

MAJOR REASONS FOR USE

Dibasic lead phosphite pigment is a versatile product and is consumed by both the plastics and paint industry. In the plastics industry it acts as an effective stabilizer for polyvinyl chloride resins or other vinyl resins having a chlorine content.

In the paint industry it acts as an anticorrosive agent in metal protective coatings and as an antistaining agent in latex coatings (over cedar and redwoods).

When dibasic lead phosphite is introduced in modest amounts into white or pastel colored metal protective coatings, it improves the blister and corrosion-resistant qualities of the paint. Usually the incorporation of 0.6 to 1.0 lb of the phosphite pigment per gallon of paint will provide improved resistance to corrosion for exposures to saline environments. It is effective in alkyd, linseed oil, and epoxy ester vehicles or their combinations. Dibasic lead phosphite will also impart anticorrosive properties to emulsion primers designed for the protection of steel. In latex primers for wood, it prevents the nail head rusting that often makes a paint job unsightly.

The effectiveness of dibasic lead phosphite in reducing the wood staining of latex paints is in proportion to the level of pigment present. Usually 0.4 to 1.0 lb/gal eliminates the bleed-through when incorporated in the latex primer.

Dibasic lead phosphite is recommended for addition to white and pastel fast-drying enamels when applied to well-cleaned metal surfaces as an insurance against corrosion problems.

PIGMENT MANUFACTURE

Dibasic lead phosphite is made by preparing a litharge slurry to which acetic acid in catalytic amounts (0.1%) is added. The slurry is heated to 100°F. A clear solution of phosphorous acid is then prepared using phosphorous acid crystals. This is heated to 100°F and slowly added to the well-mixed heated slurry until a pH in the range of 7.0 to 7.1 is reached. Agitation is continued for an added half-hour to insure equilibrium conditions. The batch is flocculated, the excess water is siphoned off, and the residue is filtered and dried. The product is cooled to room temperature and carefully disintegrated. Since the compound is sensitive to the heat generated by the disintegration process, there is the possibility of it taking fire. If this happens, there is a reddish-yellow glow in the product and that portion of the batch must be isolated and removed or the whole batch will burn (and spoil) by changing composition.

PIGMENT GRADES

There is only one grade of dibasic lead phosphite for pigment use (Dyphos/NL).

The crystal habit of this pigment grade is acicular as shown in the electron photomicrograph (1,400 ×) of Fig. 12. As better resolution is applied to the ultra-fine particles, they also are shown to be acicular (only a small percentage by weight of the particles appear roundish or irregular).

A cumulative percent particle size distribution curve for the commercial dibasic lead phosphite (Dyphos) is given in Fig. 10. The average diameter is about 3.0 μ and the specific surface area is 2.0 m^2/cm^3.

Fig. 12. Electronmicro
grade of Dibasic Lead Ph

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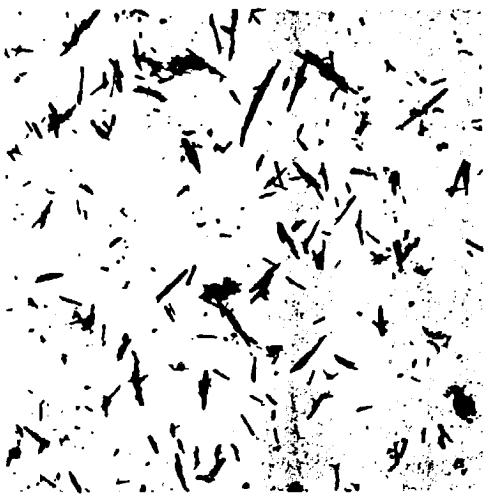

Fig. 12. Electronmicrograph (1400×) of pigment grade of Dibasic Lead Phosphite PG/NL.

Both the particle shape and size are important to the properties imparted to a paint film. Acicular particles add strength in the long direction of the particle and tend to relieve stress without rupture of the film. Acicular particles also add body (paint consistency) due to their natural interlocking tendency that produces resistance to flow as compared to roundish particles.

The spectral reflectance curve for dibasic lead phosphite is given in Fig. 7 and reveals that this pigment strongly absorbs the UV wavelengths up to 310 nm. From this point the reflectance increases rapidly until it reaches a value of 70% at 350 nm and 94% at 400 nm.

Dibasic lead phosphite is a high brightness white pigment possessing a dry powder reflection of approximately 98% across the full visible spectrum. The near-IR reflection for this pigment is quite similar to magnesium oxide.

PIGMENT SPECIFICATIONS AND PIGMENT MANUFACTURERS

There are no standard specifications for the procurement of dibasic lead phosphite.

Dibasic lead phosphite is produced by three United States manufacturers (NL, MGL, FER) but only NL supplies a pigment grade.

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Anti

W. A. GL

Pigments An
NL Industrie
Hightstown,

Pigment White 11
Antimony Trioxid
Antimony White
Antimony Pigmen
Antimony Bloom
Antimony Sesquic
Antimonious Oxic
Flowers of Antim

Chemical Formul
Sb_2O_3 (MW 291

Chemical Compos

Sb	8
O	1
	10