Exposure Studies of Exterior House Paints Containing Kaolin Clay Pigments

Thad T. Broome—J.M. Huber Corp.*

INTRODUCTION

In the 12 months ending September 1996, 2,775 million liters (733 million gallons) of architectural coatings were manufactured in the U.S.,[1] of which exterior latex house paints represented a substantial part. While other extender pigments, such as silica and calcium carbonate, are probably most widely used in these products, there are, to the author's knowledge, a substantial number of commercial exterior latex house paints manufactured in the U.S. that utilize pigments derived from kaolin clay.

A literature search found support for such usage in several extensive studies reported about 30 years ago. These include works by Liberti,[2] Vannoy,[3,4] the Los Angeles Society for Coatings Technology,[5] and Brooks, et al.[6] Brooks specifically concentrated on varieties of clay pigments in vinyl acetate copolymer latex, acrylic latex, and linseed oil formulations exposed on 500 white pine panels with calcium carbonate and talc pigments, as well as several commercial paints as controls. In single-extender formulations, clay pigments chalked more rapidly than control extenders, but outperformed controls in mildew resistance and general appearance; a delaminated kaolin having large diameter platelets showed best overall performance among all extenders. In mixed extender formulations, a combination of two-thirds delaminated kaolin and one-third coarse calcium carbonate had the best tint retention in the study while also showing good mildew resistance, no iron staining, and enhanced opacity.

There were at least two reasons for this concentration on exposure work in the area of extender pigment selection in the 1960s. One was hope among paint technologists that basic theory might be developed on which extender selection could be based; a symposium in the late 1950s keynoted by Elm,[7] as well as the Los Angeles Society study,[5] attest to this. The other reason was the development of the first U.S. commercial exterior latex house paints for wood surfaces. Nationwide marketing of these products first occurred in 1958.[8] These products created a pressing need for more data on extender pigment performance, as evidence accumulated that talc, very widely used in oil paints at the time, had more limited usefulness in latex paints.[2-4,6]

In the ensuing 30 to 35 years, although many advances have been made in latex resins, other formula ingredients, and in the understanding of formulation principals, reports of exposure studies that included clay-extended latex paints have been lacking. This period has also seen new developments in clay pigments, particularly the introduction of additional varieties of calcined clays and of structured pigment, a pigment type produced through an inorganic hydrothermal reaction.

Exposure studies were therefore undertaken to develop exterior performance data on present day clay pigments in coatings which used currently available ingredients and present day formulation approaches. Since the usefulness and general performance characteristics of several clay pigments in exterior paints have been established by earlier investigations, the emphasis of this study was on comparison of clay pigments, including both established and more recently developed types.

EXPERIMENTAL

Selection of Formula Ingredients

While the present study was intended to update the work of Brooks et al.,[6] it did not attempt to duplicate formulas used by those investigators, since some principal ingredients are no longer available. However, formula ingredients and parameters were in fact quite similar in many respects to those used in the earlier study.

Resins used included one acrylic and three vinyl acrylic latex emulsions. *Table* 1 shows typical characteristics of these four materials. The acrylic resin and vinyl acrylic resin No. 1 are established types that have been widely used for over 20 years. The other two vinyl acrylic resins were relatively new when this work was begun; they are designed for increased pigment binding and abrasion resistance, and, among other differences, are slightly harder and somewhat smaller in particle size than resin No. 1. Selection of resins and other materials was based on recommendations of suppliers and on the author's experience, including the knowledge that while first quality exterior latex house paints usually utilize acrylic resins, large volumes of vinyl acrylic resins are presently being consumed in lower cost commercial products.

An enamel-grade titanium dioxide pigment (TiO_2)having good, but not maximum, chalk resistance was chosen to provide a degree of chalk resistance believed commonly present in U.S. commercial exterior latex house paints. Additives were chosen as representative of common U.S. industry usage.

The following extender pigments were evaluated in various combinations as described in a later section. Characteristics typical of these pigments are shown in *Table* 2.

*One Huber Rd., Macon, GA 31298.

Table 1—Typical Properties of Resins Used

Resin Properties	Vinyl Acrylic Emulsions #1	Vinyl Acrylic Emulsions #2	Vinyl Acrylic Emulsions #3	Acrylic Emulsion
Solids, % by weight	55.0	55.0	55.0	55.0
pH	5.0	5.0	4.0	8.25
Density, g/ml (lb./gal.)	1.08 (9.0)	1.08 (9.0)	1.08 (9.0)	1.07 (8.9)
Particle size, microns	0.4	0.3	N/A	0.25
Viscosity, cps	2250	600	300	300
Glass transition temp., °C	+1	+12	+5	+2
Minimum filming temp., °C	+5	+8	N/A	N/A
Surfactant type	Non-ionic	Non-ionic	Non-ionic/Anionic	Anionic
Polymer specific gravity	N/A	1.15	N/A	N/A

N/A = not available

Table 2—Typical Properties of Extender Pigments Used

Pigment Property	Calcium Carbonate	Coarse Hydrous Kaolin	Delaminated Kaolin	Conventional Calcined Clay
Type	Classified	Hydrous	Hydrous	Anhydrous
Manufacutring process	Dry Ground	Water Washed	Delaminated washed	Calcined
Form	Fine powder	Fine powder	Fine powder	Fine powder
Specific gravity	2.71	2.60	2.60	2.63
Hegman grind	4.0	4+	4+	5+
Surface area B.E.T., m^2/g	N/A	6-11	11-15	7-9
Bulking value, g/ml (lb./gal.)	2.71 (22.6)	2.60 (21.7)	2.60 (21.7)	2.62 (21.9)
Bulking value, ml/g (gal./lb.)	0.370 (0.0443)	0.385 (0.0461)	0.385 (0.0461)	0.381 (0.0457)
Moisture, % max. as produced	N/A	1.0	1.0	0.5
325 Mesh residue, % max	0.01	0.05	0.005	0.01
Average stokes equivalent particle diameter, microns	6.0	4.0	1.0	1.4
Oil absorption, g/100 g pigment, ASTM D 281	16	25-30	40-45	54
Brightness, % reflectance	87.0	81.0-83.5	87.5-89.0	90.0-93.0
pH, 100 g pigment/ 250 ml water	9.3	6.0-7.5	6.0-7.5	5.0-6.0

Pigment Property	Premium Calcined Clay	Structured Pigment	Structured Flatting Pigment
Type	Anhydrous	Structured pigment	Structured pigment
Manufacturing process	Calcined	Water washed	Water washed
Form	Fine Powder	Fine powder	Fine powder
Specific gravity	2.63	2.40	2.4
Hegman grind	5+	4.5+	4.0
Surface area B.E.T., m^2/g	13-17	N/A	6.0
Bulking value, g/m (lb./gal.)	2.62 (21.9)	2.40 (20.0)	2.40 (20.0)
Bulking value, ml/g (gal./lb.)	0.381 (0.0457)	0.417 (0.0500)	0.417 (0.0500)
Moisture, % max. as produced	0.5	3.0	4-5
325 Mesh residue, % max.	0.01	0.10	0.5
Average stokes equivalent particle diameter, microns	0.7	1.7	6.0
Oil absorption, g/100 g pigment, ASTM D 281	75-85	95	100
Brightness, % reflectance	92.0-94.0	93.0	90.0
pH, 100 g pigment/250 ml water	5.0-6.0	11.0	11.0

N/A = Not available.

Table 3—Zinc-Free Vinyl Acrylic Latex Exterior House Paint Formulations

Formula Ingredients	Extender Pigment Systems					
	#1		#2	#3	#4	#5
Units	Pounds	Gallons				
Water	275.2	33.0	**NOTE: Quantities are units per 100 gallons of paint. To convert to kilograms and liters per 1,000 liters of paint, multiply pounds by 1.198 and gallons by 10.00.**			
Cellulosic thickener	4.0					
Aminomethylpropanol, 95%	2.0					
Defoamer	1.8					
Ethylene glycol	27.8	3.0				
Polyacrylate dispersant, 30%	5.2					
Potassium tripolyphosphate	1.0		This portion master-batched — same in all paints.			
Fungicide	5.0					
Nonionic surfactant	2.2					
Coalescing agent	11.8	1.5				
Total pigment volume		13.2	Same in all paints			
Titanium dioxide, enamel grade	225.0		225.0	225.0	203.9	225.0
Conventional calcined clay	100.0		—	—	100.0	—
Premium calcined clay	—		67.7	67.7	—	—
Structured pigment	—		—	—	33.7	—
Delaminated kaolin	—		—	—	—	100.0
Calcium carbonate	45.6		78.9	—	21.5	—
Coarse hydrous kaolin	—		—	75.6	—	—
Structured flatting pigment	—		—	—	—	33.4
Water	66.4	8.0				
Cellulosic thickener	1.0					
Aminomethylpropanol, 95%	1.0		This portion master-batched — same in all paints.			
Defoamer	1.8					
Vinyl acrylic emulsion, 55%	354.2	39.4				
PVC,%	40.0		40.0	40.0	40.0	40.0
Solids by volume, %	33.0		33.0	33.0	33.0	33.0

Calcium carbonate was a natural dry-ground coarse pigment, a type widely used in exterior latex house paints in the U.S. and similar to that used by Brooks.

Hydrous kaolin was a coarse water-washed clay, very similar to that used by Brooks.

Delaminated kaolin consisted of large-diameter hexagonal platelets from water-washed clay, very similar to that used by Brooks.

Conventional calcined clay, as described in *Table* 2, was a type of anhydrous clay offered by all U.S. clay suppliers; hence, the name used here. Brooks used a very similar pigment.

Premium calcined clay was a finer particle size, high oil absorption, high brightness anhydrous clay, presently widely used in paper coatings with limited use in paints. Not used by Brooks.

Structured pigment requires explanation. Calcined clays, produced by subjecting hydrous kaolins to high temperatures in order to drive off water of hydration and fuse kaolin booklets (stacks of platelets) into a structure that provides increased light scattering, can be termed structured pigments, as can some other extender pigments. However, the material referred to here belongs to a recently introduced class of clay pigments produced by slurrying selected hydrous kaolin in a caustic soda solution at elevated temperatures and pressures. Particles are fused into house-of-cards structures without loss of water or hydration. Resulting pigments have many internal air interfaces for light refraction, with bluer undertone, lower density, higher oil absorption, and higher pH than are found in calcined clays (see *Table* 2). These pigments were not available to Brooks.

Structured flatting pigment was a version of the class of pigments just described, which has a large particle size, making it effective as a flatting agent.

Basic Formulations

Two basic formulations were used. They were chosen based on the author's experience and advice of resin suppliers to represent current industry practice. Initial work and screening exposure tests utilized a zinc-oxide-free basic formula made with vinyl acrylic latex resins and intended to represent lower cost paints (see *Table* 3). The balance of exposures involved a higher solids, and thus higher cost, basic formula containing zinc oxide, with revision of the dispersant system necessary to provide stability with this reactive pigment. This latter formula was used with both vinyl acrylic and acrylic resins (see *Tables* 4 and 5).

These formulations were not intended to represent optimum conditions for any single ingredient, but rather to provide direct comparisons of the extender pigment combinations being evaluated. Dispersion and letdown portions of each formula were accordingly master batched, to the extent possible, to minimize compounding variables. Thus formula adjustment of, for example, coalescent, might have provided improved performance with a given resin. The results obtained may therefore be of limited value for drawing conclusions concerning ingredients other than the extender pigments.

Control Paints

The first pigmentation shown in *Table* 3, having an extender pigment mixture of approximately two-thirds

Table 4—Zinc-Bearing Vinyl Acrylic Latex Exterior House Paint Formulations

Formula Ingredients	Extender Pigment Systems				
	#6		#7	#8	#9
Units	Pounds	Gallons			
Water	240.0	28.8	NOTE: Quantities are units per 100 gallons of paint. To convert to kilograms and liters per 1,000 liters of paint, multiply pounds by 1.198 and gallons by 10.00.		
Cellulosic thickener	4.0				
Ethylene glycol	27.9	3.0			
Fungicide	6.0				
Defoamer	2.0				
Polyacrylate dispersant, 30%	8.0				
Aminomethylpropanol, 95%	1.0				
Nonionic surfactant	2.0				
Potassium tripolyphosphate	1.5		This portion master-batched — same in all paints.		
Total pigment volume		16.4			
Titanium dioxide, enamel grade	250.0				
Zinc oxide	25.0				
Coarse hydrous kaolin	87.7				
Delaminated kaolin	96.2		—	—	
Premium calcined clay	—		64.8	—	—
Coarse hydrous kaolin	—		32.1	48.1	—
Structured pigment	—		—	44.4	—
Conventional calcined clay	—		—	—	97.3
Water	69.4	8.3	This portion master-batched — same in all paints.		
Cellulosic thickener	1.2				
Defoamer	2.0				
Coalescing agent	11.3	1.4			
Vinyl acrylic emulsion, 55%	357.9	39.8			
PVC,%	45.0		45.0	45.0	45.0
Solids by volume, %	36.4		36.4	36.4	36.4

Table 5—Zinc-Bearing Acrylic Latex Exterior House Paint Formulations

Formula Ingredients	Extender Pigment Systems				
	#6		#7	#8	#9
Units	Pounds	Gallons			
Water	240.0	28.8	NOTE: Quantities are units per 100 gallons of paint. To convert to kilograms and liters per 1000 liters of paint, multiply pounds by 1.198 and gallons by 10.00.		
Cellulosic thickener	4.0				
Ethylene glycol	27.9	3.0			
Fungicide	6.0				
Defoamer	2.0				
Polyacrylate dispersant, 30%	8.0				
Aminomethylpropanol, 95%	1.0				
Nonionic surfactant	2.0				
Potassium tripolyphosphate	1.5		This portion master-batched — same in all paints.		
Total pigment volume		16.4			
Titanium dioxide, enamel grade	250.0				
Zinc oxide	25.0				
Coarse hydrous kaolin	87.7				
Delaminated kaolin	96.2		—	—	
Premium calcined clay	—		64.8		—
Coarse hydrous kaolin	—		32.1	48.1	—
Structured pigment	—		—	44.4	—
Conventional calcined clay	—		—	—	97.3
Water	76.1	9.1	This portion master-batched — same in all paints.		
Cellulosic thickener	1.2				
Defoamer	2.0				
Coalescing agent	11.3	1.4			
Acrylic emulsion, 55%	343.0	38.5			
PVC,%	45.0		45.0	45.0	45.0
Solids by volume, %	36.4		36.4	36.4	36.4

Table 6—Latex Exterior House Paint Primer Formulation

Formula Ingredients	Primer Used on Huber Panels		Primer Used on Huber Test Wall	
Units	Pounds	Gallons	Pounds	Gallons
Water	143.9	17.3	135.4	16.2
Cellulosic thickener	2.1		2.1	
Polyacrylate dispersant, 30%	5.2		5.2	
Nonionic surfactant	1.0		1.0	
Defoamer	1.0		1.0	
Ethylene glycol	35.0	3.8	35.0	3.8
Titanium dioxide, enamel grade	150.0	4.4	150.0	4.4
Microcrystalline silica	75.5	3.4	—	—
Coarse hydrous kaolin	77.3	3.6	217.2	10.0
Fungicide	9.0		9.0	
Defoamer	1.0		1.0	
Coalescing aid	5.5		5.5	
Acrylic emulsion, 55%	450.4	50.6	395.0	44.4
Water	111.5	13.4	143.8	17.3
Cellulosic thickener	1.0		2.5	
Ammonia, 28%	2.0		2.0	
PVC,%	30.0		38.0	
Solids by volume, %	38.0		38.0	

NOTE: Quantities are pounds per 100 gallons of paint. To convert to kilograms and liters per 1,000 liters of paint, multiply pounds by 1.198 and gallons by 10.00.

conventional calcined clay and one-third calcium carbonate, served as a control pigmentation for the zinc-free formula series. The author has had much successful experience with this combination, and earlier investigators have reported good results from calcined clays in exterior coatings[2-4,6,13]

The first pigmentations, with an extender pigment mixture of approximately one-half coarse hydrous kaolin and one-half delaminated kaolin, shown in *Tables* 4 and 5, were used as control pigmentations for the zinc-bearing exposure series. These pigments have also been established by earlier investigators as dependable performers in exterior latex paints.[2-4,6]

A control paint was prepared for each formula/resin combination used in the study. Control paints on exposure panels, described in a later section, were in all cases made with the same resin as the paints for which they served as controls.

Extender Pigment Combinations

Selection of extender pigment combinations was based on principles established by earlier investigators. Coarse particle extenders are known to give generally better tint retention and chalk resistance than fine particle extenders.[2-6] However, inclusion of a significant proportion of finer particle extenders is common practice due to the considerable enhancement of TiO_2 efficiency, and thus opacity, which they have been found to contribute.[9-13] Calcium carbonate is known to be more chalk resistant than clay pigments, but also more susceptible to staining.[2-6] Large platelet delaminated kaolin has shown promise of better chalk resistance and color retention than other hydrous kaolins.[6]

The various extender pigment combinations which were substituted into the two basic formulas are shown in *Tables* 3-5. Each is a combination of a coarse extender—defined for this paper as having an average Stokes equivalent particle diameter of 4.0 microns or more—and a fine particle extender having an equivalent diameter of less than 2.0 microns. Coarse pigments included calcium carbonate, hydrous kaolin, and structured flatting pigment; fine pigments were the two calcined clays, delaminated kaolin, and the finer structured pigment, all as previously described.

Extender pigment ratios in the individual paint formulas, that is, within the individual two- or three-extender combinations, were chosen to provide desired paint properties, as follows. PVC was held constant within each basic formula series, therefore, it was believed important to adjust extender pigment ratios in such a way that critical pigment volume concentrations (CPVCs) were also the same, to the extent that this was possible to determine, in order to insure that exposed films had similar PVC/CPVC ratios and thus similar amounts of free binder. This was done by assuming, based on the established principle that optical as well as physical properties of films pass through significant discontinuities at CPVC,[14,15] that, for constant PVC formulations that differ only in extender pigment types and quantities, equal optical properties will indicate equal PVC/CPVC ratios and thus equal binder demand. Paints within a basic formula series were therefore assumed to be similar in binder demand (that is, in PVC/CPVC ratio, and thus in CPVC, since PVCs were equal) when their reflectances after tinting and their contrast ratios before tinting were respectively very similar. Tinting was accomplished by the addition of one percent by weight of commercial glycol-based black colorant. Several trial batches of each basic formulation with each extender pigment combination were therefore prepared and tested using each control pigmentation as standard in its own series for these optical properties, and thus for binder demand and CPVC, until binder demands of all pigment combinations within each basic formula series were believed to be as similar as was practical to achieve. In the zinc-free formula series, tinted reflectances fell within a range of +/-0.7 reflectance units and contrast ratios within a range of +/-0.0075 units. In the zinc-bearing formula series, the range for tinted reflectance was +/-0.9 and that for contrast ratio was +/-0.0015.

Tables 3-5 show that extender systems evaluated included the following.

Among paints based on the zinc-free formulation,

- two used primarily conventional calcined clay as the fine particle extender;
- two used primarily premium calcined clay as the fine particle extender;
- one used primarily delaminated clay as the fine particle extender;
- two used primarily calcium carbonate as the coarse particle extender (comparing the two calcined clays); and

Table 7—Panel Exposure Ratings at Huber, GA

Southern yellow pine, south vertical, two coats WHITE over latex primer. Each value is derived from five or more panel ratings.

Extender Pigment Systems in Zinc-Free Formulations		Exposure Time, Months 10	25	34	42	69
General Appearance						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	9.6	7.5	7.7	6.3	6.4
	Std. dev.*	0.3	0.8	0.8	1.5	1.2
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	10	9.2	7.7	7.7
	Std. dev.	0	0	0.6	0.6	0.2
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	10	7.5	7.9	6.8	7.1
	Std. dev.	0	0.8	0.9	1.0	0.6
Chalk Resistance						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	10	9.7	8.8	8.5	6.0
	Std. dev.	0	0.5	0.7	0.5	0.6
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	8.7	7.3	7.9	4.0
	Std. dev.	0	0.9	1.4	0.8	0
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	9.3	8.7	7.7	8.1	6.2
	Std. dev.	0.4	0.9	0.5	0.4	0.4
Resistance to Dirt Collection						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	10	7.0	8.7	6.8	8.0
	Std. dev.	0	0	0.5	0.7	0.6
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	10	9.5	8.6	8.8
	Std. dev.	0	0	0.5	0.3	0.2
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	10	8.0	8.7	7.8	8.4
	Std. dev.	0	1.4	0.5	0.6	0.6
Mildew Resistance						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	10	7.0	8.6	6.7	7.8
	Std. dev.	0	1.4	0.8	1.2	1.0
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	10	9.5	8.2	8.6
	Std. dev.	0	0	0.5	0.2	0.5
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	10	7.5	7.7	7.5	7.9
	Std. dev.	0	0.8	0.9	0.5	0.7
Resistance to Darkening						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	8.0	6.8	7.5	6.0	7.7
	Std. dev.	0	0.4	1.0	0.6	0.9
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	10	9.5	9.1	9.0
	Std. dev.	0	0	0.5	0.2	0
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	10	9.2	9.3	8.0	8.2
	Std. dev.	0	0.4	0.5	1.0	0.8

*Standard deviation

- two were free of calcium carbonate.

Paints based on the zinc-bearing formulation utilized only clay extenders, with hydrous clay as the coarse extender in all cases and with each of the following fine particle extenders used in one paint: delaminated clay; conventional calcined clay; premium calcined clay; and structured pigment.

In one case—the zinc-free combination of conventional calcined clay, calcium carbonate, and structured pigment—titanium dioxide was reduced in order to evaluate exterior performance of this combination when used as partial TiO_2 replacement. This variation in TiO_2 loading invalidates the previously described assumption, and the binder demand of this paint must be considered as unknown relative to its control.

The combination of delaminated kaolin and structured flatting pigment was chosen because use of delaminated kaolin can result in an objectionable degree of sheen if an effective flatting pigment is not present.

Primers

As described in the following, latex primers were used on most exposure panels at Huber, GA, while an alkyd primer was used at Charlotte, NC. Formulas for these latex primers are shown in *Table* 6. The first formula shown was used on south

Table 7—Panel Exposure Ratings at Huber, GA, *Continued*

Southern yellow pine, south vertical, two coats GRAY over latex primer. Each value is derived from five or more panel ratings.

Extender Pigment Systems in Zinc-Free Formulations		**Exposure Time, Months: 10**	**25**	**34**	**42**	**69**
General Appearance						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	10	9.0	9.0	7.3	7.4
	Std. dev.*	0	0	0.8	0.5	0.5
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	9.3	8.8	7.5	6.6
	Std. dev.	0	0	0.9	0.5	0.5
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	10	7.3	7.6	7.8	7.6
	Std. dev.	0	0.5	0.7	0.4	0.6
Chalk Resistance						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	10	10	8.5	8.3	5.4
	Std. dev.	0	0	1.0	0.5	0.8
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	10	8.0	8.5	4.0
	Std. dev.	0	0	1.6	0.8	0
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	10	9.0	7.3	8.3	6.2
	Std. dev.	0	0	0.5	0.5	0.4
Resistance to Dirt Collection						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	10	10	9.3	8.0	8.2
	Std. dev.	0	0	0.5	0	0.4
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	10	9.3	8.0	9.0
	Std. dev.	0	0	0.5	0	0
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	10	8.3	8.8	8.1	8.6
	Std. dev.	0	0.9	0.4	0.2	0.4
Mildew Resistance						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	10	7.0	9.3	8.2	7.9
	Std. dev.	0	0	0.5	0.4	0.5
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	10	8.7	8.3	9.0
	Std. dev.	0	0	0.8	0.5	0
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	10	6.8	7.7	8.3	8.8
	Std. dev.	0	0.4	0.5	0.5	0.2
Resistance to Fading						
Premium calcined clay/calcium carbonate (Pigment system #2)	Mean	10	10	9.5	9.0	9.2
	Std. dev.	0	0	0.5	0	0.8
Premium calcined clay/hydrous kaolin (Pigment system #3)	Mean	10	9.3	8.8	8.8	7.6
	Std. dev.	0	0.5	0.7	0.4	0.8
Conventional calcined clay/struct. pigment/calcium carbonate (Pigment system #4)	Mean	9.2	8.2	8.0	9.2	8.9
	Std. dev.	0.2	0.7	0	0.4	0.2

* Standard deviation

vertical panels at Huber. By the time the test walls at Huber were prepared, however, information on the possible carcinogenicity of crystalline silica had been published, and this pigment was therefore removed as a precaution, resulting in the second formula shown. The alkyd primer used at Charlotte was a commercial product.

Characterization of Paints

All paints were characterized and compositions confirmed by testing for density, nonvolatile content by weight, viscosity, fineness of grind, reflectance, opacity, reflectance after tinting, sheen, and gloss using appropriate ASTM test methods.

Paints were also tested for scrub resistance, a test normally associated with interior paints. However, the author has encountered a substantial number of attempts by paint technologists to expedite exterior performance evaluations through the use of such tests. Development of data necessary to demonstrate the degree of correlation which actually exists between exterior exposure and scrub resistance was felt to be worthwhile.

Preparation and Evaluation of Exposure Panels

In accord with recognized good practice in exterior exposure testing of paints,[16,17] wood panels were selected to mini-

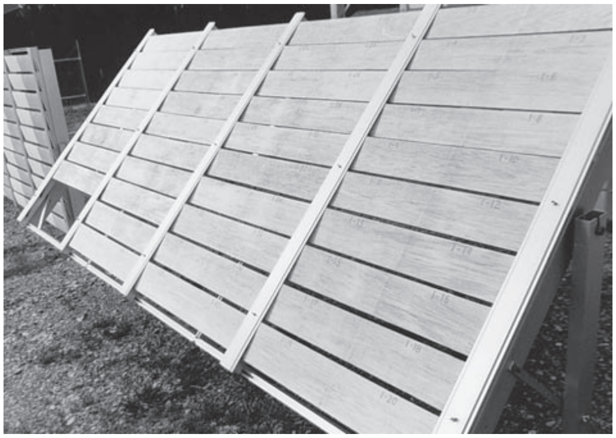

Figure 1—White test paints and controls applied one coat without primer on southern yellow pine, exposed on aluminum racks at 45° south in Huber, GA.

Figure 2—White and gray test panels and controls applied two coats over latex primer on southern yellow pine, exposed south vertical in Huber, GA.

Table 8—Test Wall Exposure Ratings* at Huber, GA

Southern yellow pine, south, two coats WHITE over latex primer.						
	Exposure Time, Months					
Extender Pigment Systems in Zinc-Free Formulation	5	14	23	34	58	71
General Appearance						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	9.2	8.5	8.5	8.1	7.2	6.1
Premium calcined clay/hydrous kaolin (Pigment system #7)	9.2	8.8	8.5	8.5	7.2	6.2
Structured pigment/hydrous kaolin (Pigment system #8)	9.2	9.0	8.5	8.0	6.8	4.8
Conventional calcined clay/hydrous kaolin (Pigment system #9)	9.2	8.5	8.5	8.8	7.8	6.8
Resistance to Chalking						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	10	9.8	10	9.5	8.5	8.0
Premium calcined clay/hydrous kaolin (Pigment system #7)	10	10	10	9.5	8.0	6.0
Structured pigment/hydrous kaolin (Pigment system #8)	10	10	10	9.5	8.5	7.2
Conventional calcined clay/hydrous kaolin (Pigment system #9)	10	9.5	10	9.0	8.5	7.8
Resistance to Mildew						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	10	10	9.5	8.1	7.2	7.2
Premium calcined clay/hydrous kaolin (Pigment system #7)	10	10	9.5	8.5	7.5	7.8
Structured pigment/hydrous kaolin (Pigment system #8)	10	10	9.5	8.5	7.0	6.5
Conventional calcined clay/hydrous kaolin (Pigment system #9)	10	10	9.5	8.8	7.5	7.8

mize surface irregularities and were prepared by a single trained technician at each exposure location. Paints were applied in the laboratory at normal spreading rates. All panels were allowed to dry under ambient conditions for 24 hr before being recoated, and for seven days before being placed on exposure. All panels were prepared in duplicate and a portion of each panel was coated with a control paint previously described. Panels were exposed at Huber, GA; Charlotte, NC; Cary, NC; and Miami, FL followed by additional exposure of these latter panels at Cary, NC. Panels were mounted in a variety of positions as described. Paints were exposed in both whites and tints. Panel ratings based on accepted pictorial standards[18] were made by the author at all locations with the exception of ratings of the Miami and Cary panels, which were made by experienced local technicians. Panels were rated for general appearance, chalking, erosion, cracking, flaking, dirt accumulation, mildew, darkening (whites only), and fading (tints only). These conditions were rated on a scale of 0 to 10 where 0 represented total failure and 10 represented no detectable deterioration.

The rating of white latex paints for darkening requires explanation. To the maximum extent possible, this rating excludes discoloration from dirt and mildew. It represents tannin staining and other discoloration believed to result from interaction between wood substrates and paint ingredients, particularly calcium carbonate.[6]

Three series of panels were exposed at Huber, GA. The first of these included only white zinc-free paints, all of which were based on vinyl acrylic resins as previously described. These paints were exposed at 45° south on 15 cm (6 in.) smooth-sawn beveled southern yellow pine siding cut to 91 cm (36 in.) lengths and mounted on aluminum racks. Paints were applied in a single coat on bare, back-primed panels. This series was designed to fail quickly, thus yielding early, coarsely differentiated results. Ratings were made at 4, 10, and 16 months (see *Figure* 1).

The second series included the same formulation and extender systems as the first series, on the same type of back-primed yellow pine panel, but exposed south vertical, two coats over latex primer. Gray tints as well as whites were exposed, grays being made by addition of one percent by weight of commercial glycol-based black colorant to the white paints. Ratings were made at 4, 10, 16, 25, 34, 44, and 69 months (see *Figure* 2).

The third series at Huber included the four extender pigment combinations in the zinc-bearing formula, each made up in one vinyl acrylic resin (resin No. 2) and in the acrylic resin, and exposed in whites and gray tints. These exposures were not on conventional test fence panels, but rather the paints were applied to a two-sided test wall, having vertical walls approximately 1.5 meters (5 ft) high facing north and south. Each side had a 31 cm (12 in.)

Table 8—Test Wall Exposure Ratings* at Huber, GA, *Continued*

Southern yellow pine, north, one coat WHITE over latex primer extender pigment.

	Exposure Time, Months					
Systems in Zinc-Free Formulations	**5**	**14**	**23**	**34**	**58**	**71**
General Appearance						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	9.0	6.5	5.2	4.2	5.5	6.2
Premium calcined clay/hydrous kaolin (Pigment system #7)	9.0	6.0	5.0	5.5	5.5	6.0
Structured pigment/hydrous kaolin (Pigment system #8)	9.0	6.5	5.5	5.0	5.5	6.0
Conventional calcined clay/hydrous kaolin (Pigment system #9)	9.0	6.0	5.5	4.5	5.5	6.0
Resistance to Chalking						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	10	10	9.5	10	9.8	10
Premium calcined clay/hydrous kaolin (Pigment system #7)	10	10	9.5	10	9.8	10
Structured pigment/hydrous kaolin (Pigment system #8)	10	10	9.5	10	9.8	10
Conventional calcined clay/hydrous kaolin (Pigment system #9)	10	10	9.5	10	9.8	10
Resistance to Mildew						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	10	9.5	6.8	6.2	5.5	6.9
Premium calcined clay/hydrous kaolin (Pigment system #7)	10	9.5	7.5	7.5	5.5	7.2
Structured pigment/hydrous kaolin (Pigment system #8)	10	9.5	7.0	6.5	5.5	7.2
Conventional calcined clay/hydrous kaolin (Pigment system #9)	10	9.0	7.0	6.5	5.5	7.0

Table 8—Test Wall Exposure Ratings* at Huber, GA, *Continued*

Southern yellow pine, south, two coats GRAY over latex primer.

Extender Pigment Systems in Zinc-Free Formulation	Exposure Time, Months					
	5	14	23	34	58	71
General Appearance						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	9.5	9.2	9.1	8.0	7.1	5.0
Premium calcined clay/hydrous kaolin (Pigment system #7)	9.5	9.2	8.5	8.0	7.5	5.0
Structure pigment/hydrous kaolin (Pigment system #8)	9.5	9.0	8.5	8.2	7.8	5.2
Conventional calcined clay/hydrous kaolin (Pigment system #9)	9.5	9.5	9.2	8.0	7.5	5.8
Resistance to Chalking						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	10	10	10	9.0	7.5	7.4
Premium calcined clay/hydrous kaolin (Pigment system #7)	10	10	10	8.5	6.0	6.0
Structured pigment/hydrous kaolin (Pigment system #8)	10	10	10	9.0	7.0	5.8
Conventional calcined clay/hydrous kaolin (Pigment system #9)	10	10	10	9.0	8.5	8.0
Resistance to Mildew						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	10	10	10	8.0	7.0	6.8
Premium calcined clay/hydrous kaolin (Pigment system #7)	10	10	10	8.0	7.5	7.5
Structured pigment/hydrous kaolin (Pigment system #8)	10	10	10	8.0	7.5	8.0
Conventional calcined clay/hydrous kaolin (Pigment system #9)	10	10	10	7.8	7.2	6.8
Resistance to Fading						
Delaminated kaolin/hydrous kaolin (Pigment system #6)	10	9.8	10	9.0	9.0	7.5
Premium calcined clay/hydrous kaolin (Pigment systems #7)	10	8.5	9.0	8.0	8.0	6.5
Structured pigment/hydrous kaolin (Pigment system #8)	10	7.8	8.0	7.8	8.0	6.2
Conventional calcined clay/hydrous kaolin (Pigment system #9)	10	10	10	9.0	9.0	6.8

*Values for delaminated kaolin/hydrous kaolin are means of four area ratings. Other values are means of two area ratings.

overhang, boxed and faced with exterior grade plywood and roofed with roofing shingles. Walls were southern yellow pine novelty siding. Test areas were coated with a latex primer and two coats of test paints on the south wall, primer and single coats of test paints on the north wall. Each test paint was applied in a stripe from bottom to top of the wall, including boxing and facia boards. Stripes were 25 cm (10 in.) wide on the south wall and 51 cm (20 in.) wide on the north wall. Whites and tints were applied to the southern exposure of the wall, and whites only to the northern exposure. Siding was cut in 1.5 meter (5 ft) lengths, and each 1.5 meter wall section included a stripe of control paint in each appropriate color. Test walls were rated at 5, 14, 23, 34, 58, and 71 months (see *Figure* 3).

Since each test area on these test walls included a series of panels at varying distances from the ground and from the overhang and since each test area was rated as a whole, most ratings are subjectively judged averages. Chalking was evaluated at the vertical midpoints of these test areas.

In Charlotte, paints exposed were the same as in the first two series at Huber—that is, only zinc free vinyl acrylic based paints. Panels were again 15 cm (6 in.) smooth-sawn beveled siding of southern yellow pine, cut to 91 cm (36 in.) lengths. Two coatings of paint were applied over a commercial alkyd primer, with a control paint on each panel. Only white paints were exposed in Charlotte. Exposures were at 45° south. Ratings were made at 12, 22, and 38 months (see *Figure* 4).

Table 9—Statistical Analysis of Panel Exposure Ratings at Huber, GA

Southern yellow pine, south vertical, two coats WHITE over latex primer.

Pigment System Pairs Compared	General Appearance	Resistance to Chalking	Resistance to Dirt Collection	Resistance to to Mildew	Resistance Darkening
#1 vs. #2	#1>#2*	#2>#1	Equal	Equal	#1>#2
#1 vs. #3	#3>#1	#1>#3	#3>#1	#3>#1	#3>#1
#1 vs. #4	Equal	#1>#4	Equal	#1>#4	#4>#1
#1 vs. #5	Equal	#1>#5	Equal	#1>#5	#5>#1
#2 vs. #3	#3>#2	#2>#3	#3>#2	#3>#2	#3>#2
#2 vs. #4	Equal	#2>#4	#4>#2	#2>#4	#4>#2
#2 vs. #5	#5>#2	#2>#5	Equal	Equal	#5>#2
#3 vs. #4	#3>#4	Equal	#3>#4	#3>#4	#3>#4
#3 vs. #5	#3>#5	Equal	#3>#5	#3>#5	#3>#5
#4 vs. #5	Equal	Equal	Equal	Equal	#4>#5*

Comparisons of pairs of extender pigment systems, combining data for all time periods and all resins.
For example, #1>#2 means Pigment System #1 was rated superior to System #2 to a statistically significant degree.
EQUAL means no statistically significant difference.
Tests marked * are at 90% confidence level; otherwise 95% confidence level.

Table 10—Statistical Analysis of Panel Exposure Ratings

	Statistical Ranking of Pigment Systems				
Characteristics of Exposed Films	First	Second	Third	Fourth	Fifth
Huber, GA Series 1, southern yellow pine, 45° South, one coat WHITE unprimed, three ratings over 16 months					
General appearance	#3	#2	#5	#1	#4
Huber, GA Series 2, southern yellow pine — south vertical, two coats WHITE over latex primer, seven ratings over 69 months					
General appearance	#3	#1,#5		#4	#2
Resistance to chalking	#2	#1	#3,#4,#5		
Resistance to dirt collection	#3	#4	#1	#2,#5	
Resistance to mildew	#3	#1	#2	#5	#4
Resistance to darkening	#3	#4	#5	#1	#2
Huber, GA Series 2, southern yellow pine — south vertical, two coats GRAY over latex primer, seven ratings over 69 months					
General appearance	#1,#2		#3,#5		#4
Resistance to chalking	#1	#2	#3,#4,#5		
Resistance to dirt collection	#3	#2	#4	#1,#5	
Resistance to mildew	#3	#1	#2,#4,#5		
Resistance to fading	#1	#2	#3,#5		#4
Cary, NC, white pine — south and north vertical, and southern yellow pine — 45° South, two coats BLUE unprimed, three ratings over 24 months					
General appearance	#3	#1	#2,#5		#4
Resistance to chalking	#1	#2	#3,#4,#5		
Resistance to mildew	#1,#2#3			#5	#4
Resistance to fading	#1	#2,#5		#4	#3
Miami, FL & Cary, NC, white pine — south vertical, two coats BLUE unprimed, four ratings over 72 months					
General appearance	#2	#1,#5		#3	#4
Resistance to chalking	#1	#2	#4,#5		#3
Resistance to mildew	#2	#1,#3		#4,#5	
Resistance to fading	#1	#2	#4,#5		#3
Charlotte, NC, southern yellow pine — 45° south, two coats WHITE over alkyd primer, three ratings over 38 months					
Resistance to mildew	#1,#2		#3,#5		

Statistical tests combined all time periods and all resins.

Table 11—Panel Exposure Ratings* at All Sites After Two Years Exposure

White Paints

Extender Pigment Systems in Zinc-Free Formulation	Location and Substrate: Huber, GA: SYP—S. Vertical, 2 Ct. on Latex Primer	Location and Substrate: Charlotte, NC: SYP—S. 45°, 2 Ct. on Alkyd Primer
General Appearance		
Conventional calcined clay/calcium carbonate (System #1)	8.2	8.0
Premium calcined clay/calcium carbonate (System #2)	7.5	7.3
Premium calcined clay/hydrous kaolin (System #3)	10.0	6.3
Conventional calcined clay/structured pigment/calcium carbonate (System #4)	7.5	7.7
Delaminated kaolin/structured flatting pigment (System #5)	8.3	7.7
Resistance to Chalking		
Conventional calcined clay/calcium carbonate (System #1)	9.3	7.0
Premium calcined clay/calcium carbonate (System #2)	9.7	7.0
Premium calcined clay/hydrous kaolin (System #3)	8.7	6.3
Conventional calcined clay/structured pigment/calcium carbonate (System #4)	8.7	6.7
Delaminated kaolin/structured flatting pigment (System #5)	8.7	7.7
Resistance to Mildew		
Conventional calcined clay/calcium carbonate (System #1)	8.5	7.8
Premium calcined clay/calcium carbonate (System #2)	7.0	8.3
Premium calcined clay/hydrous kaolin (System #3)	10.0	5.2
Conventional calcined clay/structured pigment/calcium carbonate (System #4)	7.5	6.8
Delaminated kaolin/structured flatting pigment (System #5)	8.2	6.7
Resistance to Darkening		
Conventional calcined clay/calcium carbonate (System #1)	8.2	6.7
Premium calcined clay/calcium carbonate (System #2)	6.8	5.8
Premium calcined clay/hydrous kaolin (System #3)	10.0	7.7
Conventional calcined clay/structured pigment/calcium carbonate (System #4)	9.2	7.7
Delaminated kaolin/structured flatting pigment (System #5)	9.3	7.2

Paints exposed in Cary were the same as in Charlotte. However, all paints were tinted with four fluid ounces of phthalo blue glycol based commercial colorant per gallon; no white paints were exposed. All paints were exposed north and south vertical on white pine panels, and at 45° south on southern yellow pine panels, two coats without primer in all cases. Panels were rated at 6, 19, and 24 months.

Paints exposed in Miami and Cary were the same as in the earlier Cary, including the blue tint. All paints were exposed south vertical on white pine panels, two coats without primer. Panels were rated at six months, one year, and two years in Miami, after which they were transferred to Cary and were rated there at six years.

The three northern Georgia and North Carolina exposure environments differ somewhat. The Huber site is rural, on open, flat land; the Charlotte site is industrial (adjacent to a polymer plant); the Cary site is located in an office park surrounded by hardwood trees. The Miami site is a commercial weathering station.

Statistical Analysis of Data

Analysis of data was done by use of the sign test* to a 95% level of confidence where possible and to a 90% level of confidence when necessary to obtain significant differences. This statistical method allowed comparison of samples of weathered film characteristics, as represented by exposure panel ratings, from the several populations of films represented in this work, where a population was postulated to be all films of a given formulation exposed under a given set of conditions now and in the future. This test does not require assumption that the populations are normally distributed or have equal variances.[19]

Extender pigments were compared by testing general appearance ratings from each of the three exposure series at Huber and from each of the other locations, as well as ratings for the individual film characteristics of chalk resistance, resistance to dirt collection, mildew resistance, resistance to darkening, and fade resistance, to the extent that this was judged to be productive from the various locations.

Vinyl acrylic resins were compared by testing general appearance ratings from the one coat, 45° south exposures and the south vertical panel exposures at Huber, GA. The vinyl acrylic and acrylic resins used on the test walls in Huber were also compared against each other using general appearance ratings.

To the extent necessary to provide sufficient sample sizes for analysis, data for multiple resins, multiple periods of

* The sign test compares two groups of ratings or rankings, typically where quantitative measurements are not possible, using the signs, positive or negative, of observed differences rather than quantitative magnitudes. Each extender pigment system was compared to each other system by determining the sign of the difference from each comparable pair of panel ratings. The sign test was then applied to the resulting groups of signs, to determine whether, in each system pair, one system rated higher than the other at the desired level of confidence. *Tables* 9 and 10 show how these results were translated into pigment system rankings. Resins were ranked in the same way.

Table 11—Panel Exposure Ratings* at all Sites after Two Years Exposure, *Continued*

Colored Paints

	Location and Substrate		
Extender Pigment Systems in Zinc-Free Formulation	Huber, GA: SYP, 2 Vert., 2 Ct. Gray on Latex Primer	Cary, NC: S. & N. Vert. + SYP S. 45°, 2 Ct. Blue Umprimed	Miami, FL: WP, S. Vert., 2 Ct. Blue Umprimed
General Appearance			
Conventional calcined clay/calcium carbonate (System #1)	9.0	8.4	8.0
Premium calcined clay/calcium carbonate (System #2)	9.0	8.2	7.0
Premium calcined clay/hydrous kaolin (System #3)	9.3	8.4	6.3
Conventional calcined clay/structured pigment/calcium carbonate (System #4)	7.3	7.9	5.3
Delaminated kaolin/structured flatting pigment (System #5)	8.0	8.0	6.7
Resistance to Chalking			
Conventional calcined clay/calcium carbonate (System #1)	10.0	9.1	7.7
Premium calcined clay/calcium carbonate (System #2)	10.0	8.8	6.7
Premium calcined clay/hydrous kaolin (System #3)10.0	8.1	5.7	
Conventional calcined clay/structured pigment/calcium carbonate (System #4)	9.0	8.2	6.3
Delaminated kaolin/structured flatting pigment (System #5)	9.0	8.2	5.0
Resistance to Mildew			
Conventional calcined clay/calcium carbonate (System #1)	7.6	8.6	8.7
Premium calcined clay/calcium carbonate (System #2)	7.0	8.5	8.7
Premium calcined clay/hydrous kaolin (System #3)	10.0	8.6	8.7
Conventional calcined clay/structured pigment/calcium carbonate (System #4)	6.8	7.9	6.0
Delaminated kaolin/structured flatting pigment (System #5)	7.2	8.1	8.3
Resistance to Fading			
Conventional calcined clay/calcium carbonate (System #1)	10.0	8.7	7.0
Premium calcined clay/calcium carbonate (System #2)	10.0	8.2	4.7
Premium calcined clay/hydrous kaolin (System #3)	9.3	7.7	3.3
Conventional calcined clay/structured pigment/calcium carbonate (System #4)	8.0	8.2	5.3
Delaminated kaolin/structured flatting pigment (System #5)	8.3	7.9	4.0

SYP = southern yellow pine, WP = white pine.
* Values are means of three or more panel ratings.

exposure time, and (at Cary) multiple substrates and panel positions, were combined in pigment tests. Data from multiple extender pigments, multiple periods of exposure, and (on the test wall) multiple panel positions were combined in resin tests. Resistance to erosion, flaking, and cracking are not included in reports of statistical analyses, because panel ratings for these characteristics seldom had values below eight, and differences between the pigment systems were not statistically significant. Resistance to mildew and dirt collection were omitted from the Miami and Cary analyses for similar reasons and because the number of data points available from these panels was marginal for meaningful results. The limited number of data points available from Charlotte permitted only comparison of paints containing calcium carbonate with those containing none.

RESULTS AND DISCUSSION

Ratings of Exposure Panels and Test Wall Areas

One of the most striking results of this work is the similarity in performance of all the paints tested. Of 23 paints reported, each exposed as white and one or two colors, and each exposed on 16 or more panels or on two or more test wall areas, all gave reasonably good performance over the two- to six-years period.

This is demonstrated for panel exposures in *Table* 7, which reports arithmetic means and standard deviations of panel ratings from the Huber south vertical panel series for extender pigment systems No. 2, premium calcined clay/calcium carbonate; No. 3, premium calcined clay/hydrous kaolin; and No. 4, conventional calcined clay/structured pigment/calcium carbonate. The first two of these were two of the better systems evaluated, while the third system produced the lowest overall ratings. As this table shows, the spread between performances of these paints was remarkably small and ratings were quite good over the six-year test period.

This situation was particularly notable on the test wall, where none of the four white or four gray paints tested could be shown statistically to have an advantage over the six years of exposure. While fewer data points were available from the test wall than from the panel exposures, the 30 data pairs which were in most cases available for each sign test should have permitted detection of all but small differences. *Table* 8 reports arithmetic means of test wall ratings of the eight paints for four of the most significant film characteristics.

Statistical Comparisons of Extender Pigments

Table 9, which compares each pair of extender pigment systems for each of several film characteristics of white paints

Figure 3—White and gray test paints and controls applied two coats over latex primer on southern yellow pine, exposed on test wall at south vertical in Huber, GA.

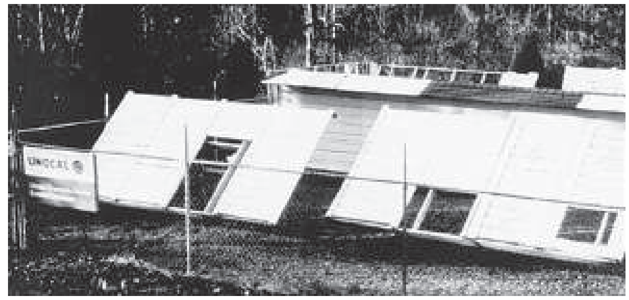

Figure 4—White test paints and controls applied two coats over alkyd primer on southern yellow pine, exposed on aluminum racks at 45° south in Charlotte, NC.

on the Huber south vertical panel exposures, illustrates the type of results generated by the sign test. Confidence levels achieved for each test are indicated. *Table* 10 reports rankings derived from this type of data, for all sites and exposures series for which data were sufficient to reveal significant statistical differences.

As previously stated, attempts at statistical analysis of test wall data found no significant differences between extender pigment systems. Various approaches were tried, but no meaningful ranking of these paints has been achieved. All four performed well. The balance of this discussion, therefore, is derived from panel exposure results and pertains to the zinc-free formulation in vinyl resins.

The following overall rankings of pigment systems are based on equal weightings for general appearance and for resistance to chalking, mildew, dirt collection, darkening of whites, and fading of colors. White paint No. 3, premium calcined clay/hydrous clay, ranked first for all characteristics except chalk resistance, in which it ranked equal to the other all-clay systems. Thus it was the best overall white paint tested. White paint No. 1, conventional calcined clay/calcium carbonate, and No. 5, delaminated clay/structured flatting pigment, made up the middle range, ranking below average for mildew resistance and dirt collection in the former case, and for mildew resistance and general appearance in the latter. Although white paint No. 2, premium calcined clay/calcium carbonate, ranked first for chalk resistance, problems with darkening and dirt collection brought it down to a tie for last place with paint No. 4, conventional calcined clay/structured pigment/calcium carbonate. This last paint suffered from mildew and had poorer general appearance.

Colored paints No. 1, conventional calcined clay/calcium carbonate, ranked as the best overall colored paints despite some tendency to dirt collection. (Note that the plural is used because both gray and blue paints were tested in each pigment system.) Their strengths were resistance to chalking and fading. Colored paints No. 2, premium calcined clay/calcium carbonate, were a close second besting No. 1 in mildew resistance and resistance to dirt collection. Among the all-clay systems in colored paints, paint No. 3, premium calcined clay/hydrous clay, showed good mildew resistance and general appearance but chalked and faded considerably. None of the other colored paints exhibited above average characteristics. It should be noted that the data from Miami had a significant effect on rankings of colored paints. Chalking and fading were dramatically greater at that location than at the other sites (see *Table* 11).

Comparison of Performance of Vinyl Paints after Two-Years Exposure at Different Sites

Table 11 presents site comparisons for the maximum period for which all panels were exposed by showing arithmetic means of panel ratings at two years from each exposure location. In examining it, however, one must bear in mind that differences go considerably beyond geographical location. In fact, climatic differences were not great with the exception of Miami. Wood species and panel positions, and

Table 12—Correlation Between Scrub Resistance and Exterior Performance

Extender Pigment Systems in Zinc-Free Formulation	Scrub Resistance Means of Three Resins		General Appearance Rankings (See Explanation Below)	
	Cycles	Rank	Whites	Colors
Conventional calcined clay/ calcium carbonate (System #1)	1841	Second	Second (tie)	First (tie)
Premium calcined clay/ calcium carbonate (System #2)	1710	Third	Fifth	First (tie)
Premium calcined clay/ hydrous kaolin (System #3)	1212	Fourth	First	Third (tie)
Conventional calcined clay/ structured pigment/calcium carbonate (System #4)	1899	First	Fourth	Fifth
Delaminated kaolin/ structured flatting pigment (System #5)	629	Fifth	Second (tie)	Third (tie)

Sources of exterior performance rankings: Whites from statistical analysis of Huber, GA panel series #2, general appearance ratings; Colors from mean of statistical analysis rankings of Huber, GA series #2, Cary, NC and Miami,Fl/Cary,NC exposures, general appearance ratings.

Table 13—Scrub Resistance of Five Pigment Systems in Three Vinyl Acrylic Resins

	Scrub Cycles		
Extender Pigment Systems in Zinc-Free Formulation	Resin #1	Resin #2	Resin #3
Conventional calcined clay/calcium carbonate (System #1)	1085	2426	2011
Premium calcined clay/calcium carbonate (System #2)	964	2285	1880
Premium calcined clay/hydrous kaolin (System #3)	677	1581	1378
Conventional calcined clay/structured pigment/calcium carbonate (System #4)	1400	2294	2002
Delaminated kaolin/structured flatting pigment (System #5)	397	738	752

particularly primers used, may well be the most significant differences. While all panels compared had two coats of finish, Huber panels were latex primed, Charlotte panels were alkyd primed, and Cary and Miami panels were unprimed.

At least three interesting observations can be made from these data.

(1) All-clay extender systems were particularly susceptible to mildew growth when applied over alkyd primer, but paints having a substantial loading of calcium carbonate showed much better mildew resistance on these panels. This observation is supported by the statistical pigment system ranking reported from Charlotte in *Table* 10, where limited data permitted only a ranking for mildew resistance of paints containing relatively high loadings of calcium carbonate (systems #1 and #2) versus those containing no calcium carbonate (systems #3 and #5). The calcium-carbonate-bearing paints ranked higher.

(2) Latex-primed panels generally outperformed alkyd-primed and unprimed panels of the same finish coats.

(3) The previous observation concerning latex primer did not apply to mildew resistance of unprimed panels, which exceeded that of latex-primed panels, although it should be noted that this comparison is between blue panels (unprimed, at Cary) and gray panels (latex-primed, at Huber). Also, the Cary two-year results are arithmetic means of ratings from white pine panels exposed both south and north vertical as well as southern yellow pine exposed at 45° south, while the Huber results are from south vertical exposures of southern yellow pine only.

Statistical Comparison of Resins

Differences found between resins were surprisingly minor. In fact, analysis of all data from paints based on the three vinyl resins totaling 80 or more panels for each resin and exposures at four sites revealed no statistically significant differences between these three resins at the 95 or 90% confidence levels, although, based on inspection of the raw panel ratings, specific resins may have shown advantages or disadvantages for certain film characteristics at specific exposure time periods.

Comparisons of vinyl resin No. 2 and the acrylic resin used on the test wall ranked the acrylic better for general appearance overall and in colored paints, better for chalk resistance in white paints, and better for resistance to dirt collection in colored paints. But the vinyl resin ranked better for chalk resistance in colored paints and for resistance to dirt collection in white paints, and 15 other tests showed no statistically significant differences.

Comparison with Results of Earlier Investigators

It is interesting to compare these results with those of the 30-year old study by Brooks et al.,[6] who exposed similar paints for five years at the same principal location used in this present study. The present study obtained ratings which are generally higher, at five years, by about two rating units, for the characteristics reported by Brooks (general appearance, mildew resistance, resistance to chalking). This may indicate substantial improvement in performance of present day raw materials. It is also noteworthy that Brooks used an oil-based primer on two-thirds of each panel. The remainder of each panel was self-primed. Results with an alkyd primer in this present study suggest that an oil primer could have detracted from performance of some of the finish coats evaluated.

Scrub Resistance and Exterior Performance

Table 12 shows with considerable clarity the lack of correlation between scrub resistance and performance on exterior exposure. *Table* 13 shows scrub test results for each resin and pigment system. Scrub resistance varied greatly with resin used in identical pigmentations and varied consistently with pigmentation regardless of resin (possibly correlating with pigment hardness). However, exterior performance, as represented by statistical rankings of general appearance of exposures, showed no apparent relationship to scrub resistance ranking.

CONCLUSIONS

Several clay pigments provided good exterior performance in exterior latex house paints applied to wood when exposed at multiple locations for up to six years.

The relatively small differences in exterior performance between the pigment systems tested, which was confirmed by statistical analysis of a rather large number of exposures, may be related to the effort made to standardize the free binder level in the test paints. This possibility points to areas for future work, to establish possible optimum exterior formulations in terms of free binder (PVC/CPVC), and to further test the standardization of free binder through measurement of optical properties.

Calcined clay provided the best white paint in the study when combined with coarse hydrous kaolin, and the best colored paints when combined with coarse calcium carbonate. This white paint was notable for freedom from darkening and low dirt collection, while these colored paints exhibited the best tint retention in the study. All paints containing calcined clay ranked high for mildew resistance, except when used in a calcium carbonate free pigment system applied over alkyd primer.

In formulations used in this study, premium calcined clay did not show a performance advantage sufficient to justify its higher cost. It should be noted, however, that for a given binder demand, premium calcined clay is used at a lower level with a higher level of coarse extender. This permits an increased loading of calcium carbonate, which should provide improved mildew resistance over alkyd primer and improved tint retention in colors, and will help to offset the higher cost of the calcined clay.

The promising exterior performance of delaminated clay which had been reported by earlier investigators was neither confirmed nor refuted by this study. The middle-quality performance of this pigment may have been due to the presence of structured flatting pigment with the delaminated clay in one formulation, as well as to the lack of detectable performance differences between pigment systems in the second formulation in which it was used.

Additional work is needed if structured clay pigments are to be established as suitable for exterior use. They should not be written off at this point, however, for two reasons. First, the zinc-free pigment system which contained the finer particle size structured pigment in this study was not standardized for binder demand. High binder demand, rather than structured pigment performance, may have accounted for the relatively poorer performance of this paint. Second, the zinc-bearing pigment system in which this pigment was used was standardized for binder demand and performed equally with the other zinc-bearing systems evaluated.

Some conclusions seemed inescapable to the investigator when the actual panels were examined. One of these is that calcium carbonate is a very useful ingredient for paints which will be applied over alkyd primer, if they are to resist mildew; this is reflected in the two-year ratings of the white paints at Huber as compared to those at Charlotte, as shown in *Table* 11. Another conclusion is that exposure at Miami must be viewed as an accelerated test, which is excellent for establishing relative chalk and fade resistance, but is not realistic for paints which will not be used in such climates. A third conclusion is that latex primer generally enhances performance considerably as compared to either no primer or alkyd primer. However, there was an anamoly at Cary, where mildew resistance of unprimed blue-tinted panels was superior to that of latex-primed gray panels at Huber.

Differences in overall exterior performance among the four resins used in this study were small to a degree which was surprising to the investigator. Statistical analysis of the data indicates that choice of resins from this group is not a major consideration, even though both vinyl and acrylic resins were included.

Scrub resistance showed no significant correlation with exterior performance. Its use in evaluation of exterior paints should be discouraged.

References

(1) "Business Activity Indicators," *Mod. Paint Coat.*, 87, No. 2, 14 (1997).

(2) Liberti, F.P., "The Function of Extender Pigments in Exterior Vinyl Emulsion Paints," *Official* Digest, *33*, No. 434, 390 (1961).

(3) Vannoy, W.G., "Extenders in Outside House Paints," *Official* Digest, *31*, No. 441, 1215 (1961).

(4) Vannoy, W.G., "Paint and Pigment Variations as Tested on Wood," *Official* Digest, *35*, No. 461, 537 (1963).

(5) Los Angeles Society for Coatings Technology, "Surface Area/PVC Relationships for Various Extender Pigments," *Official* Digest, *33*, No. 442, 1461 (1961).

(6) Brooks, L.E., Sennett, P. and Morris, H.H., "Kaolin—Five Years on the Test Fence," Journal of Paint Technology, *40*, No. 520, 240 (1968).

(7) Elm, A.C., "Size and Shape Properties of Representative White Hiding and Extender Pigments," *Official* Digest, *31*, No. 413, 720 (1959).

(8) Lowrey, E.J., personal communication.

(9) Stieg, F.B., "The Geometry of White Hiding Power," *Official* Digest, *34*, No. 453, 1065 (1962).

(10) Cuthrone, Luigi, "Influence of Fine-Particle Size Extenders on the Optical Properties of Latex Paints II," Journal of Coatings Technology, *58*, No. 736, 83 (1986).

(11) Braum, J.H., "Crowding and Spacing of Titanium Dioxide," Journal of Coatings Technology, *60*, No. 758, 67 (1988).

(12) Brooks, L.E. and Morris, H.H., "The Function of Modified Kaolins in Flat Wall Paint," *Official* Digest, *37*, No. 489, 1204 (1965).

(13) Madson, W.H., "White Hiding and Extender Pigments," *Federation Series on Coatings Technology*, Federation of Societies for Paint Technology, Philadelphia, 1967.

(14) Paul, S., *Surface Coatings Science and Technology*, John Wiley & Sons, New York, 1985.

(15) Patton, T.C., *Paint Flow and Pigment Dispersion*, John Wiley & Sons, New York, 1979.

(16) Storer, R.H. (Ed.), *1988 Annual Book of ASTM Standards*, Vol 6.01, American Society for Testing and Materials, Philadelphia, 1988.

(17) Sward, G.G. (Ed.), *Paint Testing Manual*, American Society for Testing and Materials, Philadelphia, 1972.

(18) *Pictorial Standards of Coatings Defects*, Federation of Societies for Coatings Technology, Philadelphia, 1979.

(19) Hamburg, M., *Statistical Analysis for Decision Making*, Harcourt Brace Jovanovich, New York, pp 542-545, 1983.