

Dispersions useful in the Coating of Substrates1. Field of the Invention

5 This invention relates to dispersions useful in the coating of substrates, e.g. in primer paints laid down directly on cleaned steel surfaces of large, exposed and previously painted tanks to prepare them for reception of a new outer protective or decorative paint or enamel coat, reducing the need for cleaning.

10 2. Prior Art

Swanson 3,136,653 illustrates a prior art primer coating.

15 Krueger 4,156,678 shows hydroxylated (meth)acrylic interpolymers and polyisocyanates useful in the invention. Krueger's ultimate products, furthermore, make excellent topcoats for the instant primers.

Simon 4,232,090 illustrates another known primer.

20 Strolle, et al, 4,281,078 (Strolle) shows hydroxylated acrylic copolymers and polyester polyols with methods for their preparation, and organic polyisocyanates.

For a full appreciation of the present invention, the reader should refer to all of the above art.

DESCRIPTION OF THE INVENTION

25 A major industrial problem is the corrosion of metallic structures, particularly those made from ferrous metals such as steel. Structural steel, for example, and other metallic objects exposed to the weather

require painting, a process which must be repeated every few years. In usual practice, the metallic surface is covered first with a primer coating and then with an outer protective and/or decorative coating of paint or enamel.

When repainting is necessary, the original coatings and any rust must be stripped off. Usually, stripping is accomplished by sandblasting, a process which is becoming more and more undesirable. In some areas, for example, it may eventually be prohibited by law. When its use is permitted, the sand particles may clog up motors, even when the latter are protected by tarpaulins or plastic sheets. Some structures may be inaccessible to equipment needed for sandblasting. Environmentalists may object to the sand and other debris from the treatment, and the sand, in fact, may cause silicosis. Finally, sandblasting is very expensive, costing some 50-100% more than the less effective hand cleaning.

Various attempts have been made to reduce the need for sandblasting but none have been commercially successful. Simple hand cleaning, while cheaper than sandblasting, has not proven satisfactory. The present invention,

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therefore, has a principal object the provision of a stable dispersion useful in the coating of substrates, particularly when it is desired to reduce the need for sandblasting.

According to the present invention, there is provided a stable dispersion in an anhydrous nonhydroxylated organic solvent containing at least 55% by weight of solids of:

a binder formed from at least one hydroxylated methacrylate or acrylate polymer or polymeric polyester having 1-10% by weight of pendant hydroxyl groups and a number average molecular weight of 500-10,000 in the case of a methacrylate or acrylate polymer, or 340-10,000 in the case of a polymeric polyester, and soluble to the extent of at least 30% by weight in the solvent; and

a pigment mixture of which at least 20% by weight is a particulate inorganic extender;

the dispersion containing additionally a lanolate; and

the pigment/binder weight ratio of said solids being in the range 60/100-600/100.

In this specification, the methacrylate or acrylate polymers and polymeric polyesters will be spoken of as "prepolymers", and the "pigment" will include extender desirable in primers.

The binder may comprise a polymer of a hydroxyalkyl methacrylate or acrylate or a polyester of at least one dicarboxylic acid or corresponding anhydride and an excess of at least one polyol. Advantageously the extender is in the form of inorganic platelets. More specifically, the extender may comprise talc and/or micaceous iron oxide. Advantageously also the pigment mixture may contain up to 5% by weight of carbon black. The lanolate mentioned may for example represent up to 6.0% by

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weight of the total pigment weight. The lanolate preferably comprises barium lanolate.

The invention also includes the substrate coating process which comprises:

A. mixing together a dispersion according to the invention and a polyisocyanate in an -NCO/-OH mole ratio of 0.1/1.0 to 2.2/1.0;

B. applying the mixture from A to a substrate; and

C. permitting the mixture from B to air-dry on the substrate.

The invention further includes a coating vehicle capable of curing in air at room temperature consisting essentially of an organic solvent and

A. an organic polyisocyanate and

B. a dispersion according to the invention, the -NCO/-OH mole ratio being in the range 0.1/1.0 to 2.2/1.0.

Additionally the invention includes an article comprising a substrate carrying adhered thereto the coating vehicle just mentioned in cured form. The article may for example comprise a ferrous substrate carrying, superimposed upon this cured coating, a layer of a second coating.

More specifically, the substrate coating process may for example be one in which a previously painted metal surface is hand cleaned (if necessary) as by scraping, wire brushing, power tool abrading or the like, and a two-package primer of good adhesion is applied, the primer's first package ("Package 1") comprising a dispersion according to the invention and its second package ("Package 2") comprising an orthodox polyisocyanate crosslinking agent. Packages 1 and 2 or in other words the dispersion and the poly-

isocyanate are mixed at the time of use, and the mixture is applied to the surface to be protected by any convenient means, e.g., by brush, roll or spray, etc. The primer dries in air very rapidly, minimizing the time when tacky material is vulnerable to soil and allowing rapid recoat. This primer can be used directly as a topcoat but, if only because of appearance, is generally a primer for a topcoating of polyurethane, epoxide or the like.

DETAILED DESCRIPTION

The prepolymers of Package 1, as indicated, either (1) hydroxyl-containing acrylic (i.e., acrylate or methacrylate) polymers or (2) hydroxyl-containing polymeric polyesters. They contain 1-10% by weight of pendant OH groups, with 1.5-6% preferred, and are soluble to the extent of at least 30% by weight in the solvent specified.

The acrylic prepolymers usable are preferably formed from two or more conventional ethylenically unsaturated polymerizable monomers, one of which is always at least one hydroxyalkyl acrylate or methacrylate in which the alkyl group contains 2-18 carbons. Such monomers include hydroxyethyl methacrylate, hydroxypropyl methacrylate, hydroxybutyl methacrylate, hydroxyoctyl methacrylate and corresponding acrylates and the like. Preferred are hydroxyalkyl methacrylates or acrylates in which the alkyl groups contain 2-4 carbons, e.g., 2-hydroxyethyl acrylate and the like.

Typical ethylenically unsaturated monomers that can be used with the hydroxylated monomer are, for example: vinyl and vinylidene chlorides; olefins such as ethylene, propylene and the like; vinyl acetate; conjugated dienes having 4 to 10 carbon atoms, such as butadiene; aromatic hydrocarbons having vinylene groups, such as styrene and alkyl-substituted styrene, such as α -methyl styrene; alkyl maleates, such as dibutyl maleate; vinyl pyrrolidone; methacrylonitrile and acrylonitrile; esters of methacrylic acid and acrylic acid, preferably alkyl esters having 1-12 carbon atoms in the alkyl group, such as methyl methacrylate, ethyl methacrylate, propyl methacrylate, butyl methacrylate, hexyl methacrylate, 2-ethylhexyl methacrylate, lauryl methacrylate and the like, and corresponding acrylates; or mixtures of these monomers. Particularly useful monomers include styrene, methyl methacrylate, butyl methacrylate, ethyl acrylate, acrylonitrile, and vinyl pyrrolidone.

For the purposes of this invention, the preferred acrylic prepolymer contains at least three monomers, i.e., the hydroxylated acrylic monomer, at least one other acrylic monomer, and styrene. Preferred monomers are 2-hydroxyethyl acrylate, a lower alkyl methacrylate and a longer-chain alkyl methacrylate, e.g., ethyl and lauryl methacrylates, and styrene, with the hydroxylated monomer comprising at least 6.6% by weight and styrene, 20-70%.

The acrylate or methacrylate prepolymer is made by conventional methods: see, for example, the Strolle patent noted. The precursor monomers and catalyst (azo, peroxide) are mixed together in an organic solvent such as Cellosolve® acetate or ethyl acetate and refluxed until polymerization is

complete. Both monomers and prepolymers are very soluble in organic solvents and the product need not be separated from solvent used in its preparation. The result is a high-solids solution, usually ca. 60-80% by weight, with 70% preferred. High concentration of prepolymer is useful in applications to reduce pollution caused by solvent. The molecular weight $\bar{M}_n$ (number average molecular weight) is preferably about 4,000.

10 Any organic solvent used either for the preparation of the prepolymer or for forming a dispersion therewith should, of course, be anhydrous and free from hydroxyl. Otherwise, general organic solvents may be used. Cellosolve® acetate and ethyl acetate are preferred. Aromatics such as xylene are usable but are less preferred because of pollution problems.

20 To a prepolymer solution prepared as above or diluted is added the pigment necessary for the primer. This pigment is devoid of heavy toxic metals, notably lead and chromium, and preferably contains inorganic coloring matter such as red iron oxide and zinc oxide, carbon or lamp black and, particularly as the present composition is a primer, an extender comprising at least one relatively inert inorganic material preferably shaped in the form of platelets. Several chemicals are commercially available in platelet form, e.g., silicates (magnesium and sodium silicates, hydrous aluminum silicate, glass, mica, quartz); metals (aluminum, stainless steel, nickel); oxides (iron and zinc oxides, alumina); salts (organic salts of calcium, barium); etc. A preferred extender is talc (magnesium silicate) but commercially available micaceous iron oxide (metallic black-brown platelets) is equally effective, although expensive. Heterogeneous roughly hewn nodular extender particles such as calcium carbonate or barium sulfate can be used, but they

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are not as effective as the shaped lamellar materials. In the pigment mixture, at least 20%, and usually between 20 and 95%, by weight, is a particulate inorganic extender.

The total composition of the preferred pigment mixtures is given by the following Table I:

TABLE I

<u>Pigment Mixtures</u>	<u>Percentage by Weight</u>			<u>Preferred (%)</u>
Calcium Carbonate	-	-	20-90	-
Talc	20-90	-	0-90	70
Micaceous Iron Oxide	-	20-95	-	-
Zinc Oxide	5-25	5-25	5-25	14
Red Iron Oxide	0-50	0-50	0-50	14
Carbon Black	0-5	0-5	0-5	2
	<u>100</u>	<u>100</u>	<u>100</u>	<u>100</u>

In addition to the pigment components mentioned, a lanolate, preferably barium lanolate, is included in Package 1. This is a corrosion inhibitor which acts also as a pigment dispersant and anti-settling agent. Barium lanolate usually employed forms about 0-6.0% by weight of the total pigment weight (0-4.2% total weight of solids).

The ingredients which make up Package 1 are readily assembled. A solution of prepolymer prepared as noted is adjusted to the proper solids content, e.g. ca. 37%, by means of an anhydrous nonhydroxylated organic solvent as specified, e.g. Cellosolve acetate, and the various solid materials added thereto. The mixture is then ground for about 1 day in a ball mill until a substantially homogeneous nonsettling or stable suspension is obtained. Table II gives some characteristics of the pigmented Package 1 materials, in preferred and most preferred cases.

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TABLE II

	<u>Preferred</u>	<u>Most Preferred</u>
P/B (Pigment to Binder Weight Ratio)*	213/100	
5 PVC (Pigment Volume Concentration)	15-65%	42%
% Vol. Solids	45-82%	50%
% Wt. Solids	55-92%	67%

*Here B (the binder) includes only the prepolymer.

10 Package 2 may be a solution in an organic solvent of a polyisocyanate as disclosed in above-mentioned patent 4,156,678, especially at col. 2, line 37, through col. 3, line 18. Some of these compounds
15 are known as Desmodurs® and Mondurs®, readily available commercially and quite useful here.

The polyisocyanate of Package 2 is used with the hydroxylated prepolymer of Package 1 in
20 the mole ratio -NCO/-OH 0.1/1.0-2.2/1.0, with 1/1-1.15/1 being preferred. It is dissolved in an organic solvent as is the prepolymer but, since some aromatics can be tolerated, a Cellosolve® acetate/ethyl acetate/xylene mixture is generally employed
25 (69/23/8% by weight). Data on the mixed Packages are given in Table III:

TABLE III

	<u>Preferred</u>	<u>Most Preferred</u>
30 P/B*	50/100-400/100	143/100
PVC	15%-60%	33%
% Vol. Solids	45%-80%	50%
% Wt. Solids	50%-90%	67%

35 *Here the binder includes polyisocyanate as well as prepolymer.

When it is desired to use the present coating composition, it is merely necessary to mix the two packages and apply the mixture to the prepared surface. Although the mixture dries rapidly in air, it will remain sufficiently fluid for application, e.g. with a viscosity below about 1000 cps, for up to about 8 hours. In general, any substrate, particularly a metallic substrate, can be protected by the present primer. Ferrous surfaces, e.g., of iron or steel, can readily be coated. A rusty surface, for example, need only be hand cleaned although performance is improved with improved cleaning. The primer can also be put down directly on a previously painted surface. In any event, structural steel girders, steel oil storage tanks, pipes, steel sheeting for ships, trucks, etc., are easily protected by the primers.

Application can be by any of the conventional means such as spraying where permitted, rollers or brush. The primer dries rapidly in air, e.g., in about 5-8 hours, at 20-25°C, and is then ready for application of the other protective coat. The superimposed coat can be of usual commercial varieties, e.g., acrylic urethane such as Imron® or epoxide such as Corlar®.

It is sometimes desirable to accelerate the reaction between isocyanate and hydroxyl, particularly in cold weather or when a quick-drying shop primer is needed. Conventional tertiary amines such as N,N-dimethyl dodecylamine; N,N,N',N'-tetramethyl-1,3-butane diamine; N,N,N',N'-tetramethylethylene diamine; triethylamine; N-ethyl morpholine; etc., or organo-metallic compounds such as stannous octoate, etc., can be used.

For the purposes of this invention a mixture of di-
butyltin dilaurate (DBTDL) and 1,4-diaza(2,2,2)bicyclo-
octane (DABCO; triethylenediamine) has been satisfactory
(see Example 4 below). The mixed catalyst is preferably
5 added separately to the polyisocyanate and polyester
polyol components as substantially in a third package.

In the alternative embodiment of the
invention, the hydroxyacrylic prepolymer of Package
1 is replaced by a polyester polyol with
10 pendent hydroxyl groups. The polyesters employed
are made usually from dicarboxylic acids (or
anhydrides, where they exist) such as the phthalic
acids and the like and an excess of monomeric
glycol. Branching as produced by a branching
15 polyol such as trimethylolpropane or pentaerythritol
is preferred but not necessary. The degree of
branching is determined by the mole ratio of the
ingredients as shown in Example 3. Small amounts
of monocarboxylic acids, e.g., benzoic acid, may
20 be used if desired (to defunctionalize some hydroxyl
groups). The resulting polyester should have
about 1-10% by weight of pendant OH. The cited
Strolle patent(4281078) shows a usable polyester
polyol with an $\bar{M}_n$ of 340. The polyester
25 polyol is packaged and applied like the hydroxyl-
acrylic prepolymer above and with similar results.
It is found in practice that the polyester aspect
of the invention can give a somewhat higher con-
centration of solids than the acrylate.

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EXAMPLES

There follow some examples illustrating
the practice of the invention. Unless otherwise noted,
in these examples all parts, proportions and percentages

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are in terms of weight and temperatures, in degrees centigrade.

Example 1

5 A. Hydroxylated Resin. A hydroxyacrylate polymer was prepared at reflux (ca. 145-155°) in Cellosolve® acetate from premixed styrene (31.5%), ethyl methacrylate (31.5%), lauryl methacrylate (25.0%) and 2-hydroxyethyl acrylate (12.0%) with t-butyl per-
10 acetate as catalyst. The product was a 70% solids solution of the hydroxyacrylate polymer of the composition determined by the monomers: $\bar{M}_n = 4000$; Vis = 2500 cps; -OH = 1.76%.

15 B. Dispersion. Resin of A (27.91%) is diluted with Cellosolve® acetate (18.60%) and ethyl acetate (6.04%), and pigment (45.59%) and corrosion inhibitor (1.86%) were added thereto. Pigment consist of the extender talc (69.38%), zinc oxide (14.28%), iron oxide (14.28%) and carbon (2.06%; furnace black). The corrosion
20 inhibitor is barium lanolate. All of these materials are mixed and milled together for about 18 hours to a fineness of 0.5 mils. This process forms a stable brown dispersion: Solids = 67.0%.

25 C. Crosslinking Agent. A mixture of polypropylene glycol (49%), trimethylolpropane (8%) and toluene diisocyanate (43%) were reacted together during reflux in a nonaqueous solvent mixture of xylene/Cellosolve® acetate (70/30) under a nitrogen
30 blanket. An isocyanate-terminated prepolymer of about 75% gravimetric solids was obtained with an average -NCO content of about 9.4% based on solids.

This prepolymer was further diluted with ethyl acetate to yield a final composition of 58.14%
35 solids in a solvent blend of Cellosolve® acetate (17%), xylene (40%), and ethyl acetate (43%).

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D. The dispersion of B and the cross-linking agent of C were mixed together in a volume ratio of four to one (4:1), respectively. This two-component mixture is stable for use as a paint (or has a "usable potlife") in excess of twenty-four (24) hours during which time it must be used. Upon further aging, reaction of the two polymers introduced through their respective package components leads to increasing polymeric molecular weights accompanied by an increase in viscosity ultimately producing an insoluble gel.

The two-component primer mixed as described was sprayed onto 4" x 12" hot rolled steel (AISI-1010, 16 gauge) panels to a dry film thickness of 2 to 3 mils. Simultaneously, panels of the same substrate were prepared by applying commercial red lead oil-alkyd metal primers as well as two-package epoxy/polyamide metal primers to the same dry film thickness. The panels were air dried at ambient temperatures for seven days and were then coated with a urethane enamel to 2 mils dry film thickness. These painting systems were subjected to accelerated corrosion testing in a salt fog chamber controlled at $95 \pm 2^\circ\text{F}$ using an aqueous 5% sodium chloride solution. This test is well established in the industry and is based on ASTM B-117 method.

After 816 hours' exposure in the salt fog chamber, the painting system using the commercial control red lead oil-alkyd primer had developed severe blistering and rusting on the face of the panel. The commercial control epoxy/polyamide primer system showed face-blistering at 1000 hours of exposure, while the two-component urethane primer system showed no face-blistering after 2300 hours of exposure.

Example 2

A. A hydroxyacrylate polymer was made up as in Example 1 except that the styrene/ethyl methacrylate/lauryl methacrylate/2-hydroxyethyl acrylate weight ratio was 25/25/20/30: -OH = 4.4%; $\bar{M}_n = 4000$.

B. A brown dispersion was prepared as in Example 1 except that the resin of Example 2 A was substituted.

10 C. Desmodur® N-75, a 75% solids solution of an aliphatic polyisocyanate prepolymer commercialized by Mobay Chemical Corp., was diluted to 53.6% solids with ethyl acetate for use in conjunction with the primer prepared in Example 2 B.

15 D. Four volumes of the dispersion of Example 2 B were mixed with one volume of crosslinker of Example 2 C. This primer, together with commercial control metal primers such as epoxy/polyamide as well as several red lead and oil primers, were
20 applied to 4" x 12" panels of hot rolled steel (AISI-1010; 16 gauge). These panels had been previously exposed to the elements for nine months for rusting and subsequently cleaned with a wire brush to simulate actual field conditions in the
25 industry. These primed panels were allowed to dry for a week and then coated with a urethane enamel. These systems were evaluated by salt spray testing as in Example 1 D. The system based on the urethane primer of this invention showed no face-blistering
30 after 2100 hours of exposure. All the other systems started blistering at a 768-hour check and showed failure due to blistering at the 1250 hour check.

Example 3

A. A polyester was prepared by reacting
35 under reflux in ca. 3% xylene, pentaerythritol,

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benzoic acid, neopentyl glycol, isophthalic acid, phthalic anhydride and adipic acid in the mole ratio 3.2/6.4/12.8/4.0/4.0/2.0. Heating was continued with removal of water until the pot temperature reached 215°. The batch was cooled to 80° and thinned with ethyl acetate: Solids = 85%; -OH = 6.32%; Acid No. = 20-30.

- 5 B. A stable dispersion was prepared by grinding in steel ball media to a fineness of 0.5 mils: polyester resin as prepared in Example 3 A (32.67%); Cellosolve® acetate (16.83%); talc (33.66%); zinc oxide (Kadox 515) (6.93%); red iron oxide (6.93%); furnace black pigment (Raven 500) (1.00%); and barium lanolate (1.98%).
- 15 C. A crosslinker was prepared by diluting Desmodur E-21, a 100% solids polyisocyanate pre-polymer available from Mobey Chemical Corp. to 75% solids with xylene.

- 20 D. The dispersion of Example 3 B was blended with the crosslinker of Example 3 C in the weight ratio 2:1. This primer was sprayed together with control commercial red lead and oil/alkyd metal primers over pre-rusted hand/wire brush-cleaned 4" x 12" steel panels as described in Example 2 above.
- 25 After drying, these panels were coated with the same urethane enamel and placed in Florida at 5° angle from horizontal facing south. After six months of exposure, the commercial red lead and oil/alkyd primed system had developed considerable blistering. The urethane primed system of this invention was in immaculate condition.
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Example 4

- 35 A series of tests were made on the mixed primer of Example 1 D to determine the effect of

catalysts on drying time. The catalyst used was dibutyltin dilaurate (DBTDL) alone and in conjunction with triethylenediamine (DABCO). The paint samples were equilibrated to 25° and paint-cure was measured at 25°.

The mixed primer was further mixed with various catalyst concentrations. Films were cast from these samples on 5" x 7" glass plates with a 10 mil clearance Bird film applicator in accordance with ASTM-D-823-53 except that the applicator blade was hand drawn. When the primer films were tack free, they were overcoated with a commercial polyurethane enamel and a commercial epoxy/polyamide enamel, respectively, both containing high levels of strong and potent solvents. The primer films were not attacked by these strong solvents, and the enamels dried out flawlessly and at high-gloss levels.

The effect of catalyst on the film cure is reflected in the tack-free time and the pot life summarized in Table IV:

TABLE IV

	Catalyst*	Pot Life	Tack Free
	(%DBTDL/%DABCO)		
25	None	48 hrs	4.5 hrs
	0.05/0.00	8 hrs 16 hrs	3.25 hrs
	0.05/0.06	6 hrs	2.45 hrs
	0.10/0.00	6 hrs	2.45 hrs
	0.10/0.06	3.5 hrs	2 hrs
30	0.10/0.12	1.5 hrs	1.5 hrs

* % Catalyst based on resin solids.

The catalyst level of 0.05% DBTDL/0.06% DABCO based on resin solids of mixed primer of Example 1 was used to spray a few structural steel I-beams and crossmembers in a chemical installation

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at 6°C. By night fall the temperature had dropped
to -4°C. Next morning the primer was tack free and
cured hard. It was then overcoated with commercial
urethane enamel with excellent results and appear-
ance. The pot life at these temperatures exceeded
an eight-hour work shift.

From these results it is considered that,
between 50-100°F (10-37.78°C), no catalyst is
needed; between 30-50°F (-1.11-37.78°C), the
0.05/0.06 ratio is desirable; and between 10-30°F
(-12.22-1.11°C), the 0.10/0.12 ratio is desirable.

The words "Cellosolve", "Desmodur", "Mondur",
"Dabco", "Corlar", "Imron" and "Raven" appearing in the
foregoing description are registered Trade Marks.

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CLAIMS:

1. A stable dispersion in an anhydrous nonhydroxylated organic solvent containing at least 55% by weight of solids of:

a binder formed from at least one hydroxylated methacrylate or acrylate polymer or polymeric polyester having 1-10% by weight of pendant hydroxyl groups and a number average molecular weight of 500-10,000 in the case of a methacrylate or acrylate polymer, or 340-10,000 in the case of a polymeric polyester, and soluble to the extent of at least 30% by weight in the solvent; and

a pigment mixture of which at least 20% by weight is a particulate inorganic extender;

the dispersion containing additionally a lanolate; and

the pigment/binder weight ratio of said solids being in the range 60/100-600/100.

2. A dispersion of claim 1 wherein the binder is a polymer of a hydroxyalkyl methacrylate or acrylate or a polyester of at least one dicarboxylic acid or corresponding anhydride and an excess of at least one polyol.

3. A dispersion of claim 1 or claim 2 wherein the extender is in the form of ⁱⁿ⁻organic platelets.

4. A dispersion of any one of claims 1 to 3 wherein the extender is at least one of talc or micaceous iron oxide.

5. A dispersion of claim 1, 2, 3 or 4 wherein the pigment mixture contains up to 5% by weight of carbon black.

6. A dispersion of any one of claims 1 to 5 wherein the lanolate represents up to 6.0% by weight of the total pigment weight.

7. A dispersion of any one of claims 1 to 6 wherein the lanolate comprises barium lanolate.



8. The substrate coating process which comprises
 - A. mixing together a dispersion of any one of claims 1 to 7 and a polyisocyanate in an -NCO/-OH mole ratio of 0.1/1.0 to 2.2/1.0;
 - B. applying the mixture from A to a substrate; and
 - C. permitting the mixture from B to air-dry on the substrate.
9. A coating vehicle capable of curing in air at room temperature consisting essentially of an organic solvent and
 - A. an organic polyisocyanate and
 - B. a dispersion of any one of claims 1 to 7, the -NCO/-OH mole ratio being in the range 0.1/1.0 to 2.2/1.0.
10. An article comprising a substrate carrying adhered thereto the coating vehicle of claim 9 in cured form.
11. An article of claim 10 comprising a ferrous substrate carrying superimposed upon the cured coating a layer of a second coating.



12. A stable dispersion in accordance with claim 1 substantially as hereinbefore described in any one of the Examples.

13. A process in accordance with claim 8 substantially as hereinbefore described in any one of the Examples.

14. A coating vehicle in accordance with claim 9 substantially as hereinbefore described in any one of the Examples.

15. An article in accordance with claim 10, substantially as hereinbefore described in any one of the Examples.

