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ULTRAVIOLET RADIATION CURABLE PAINTS

Final Report for the Period June 28, 1979—September 30, 1981

By
A. M. Grosset
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September 30, 1981

Work Performed Under Contract No. AC01-79CS40210

Westinghouse R&D Center
Pittsburgh, Pennsylvania

MASTER

U. S. DEPARTMENT OF ENERGY

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ULTRAVIOLET RADIATION CURABLE PAINTS

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ABSTRACT

In product finishing lines, ultraviolet radiation curing of paints on prefabricated structures could be more energy efficient than curing by natural gas fired ovens, and could eliminate solvent emission. Diffuse ultraviolet light can cure paints on three dimensional metal parts. In the UV curing process, the spectral output of radiation sources must complement the absorption spectra of pigments and photoactive agents. Photosensitive compounds, such as thioxanthones, can photoinitiate unsaturated resins, such as acrylated polyurethanes, by a free radical mechanism. Newly developed cationic photoinitiators, such as sulfonium or iodonium salts (the so-called "onium" salts) of complex metal halide anions, can be used in the polymerization of epoxy paints by ultraviolet light radiation.

One-coat enamels, topcoats, and primers have been developed which can be photoinitiated to produce hard, adherent films. This process has been tested in a laboratory scale unit by spray coating these materials on three-dimensional objects and passing them through a tunnel containing UV lamps.

1. INTRODUCTION

The Westinghouse Electric Corporation has completed a program to develop ultraviolet light curable paints for application on three dimensional objects representing typical industrial items. This program was sponsored by the U.S. Department of Energy in an effort to evaluate a more energy efficient technique than direct fired gas ovens which traditionally have been used for curing paint films on metal substrates, such as appliances, metal furniture, automobiles, and other fabricated metal parts.

High quality, durable paint finishes are generally achieved by a high temperature baking cycle, e.g., 20 to 30 minutes at 175 degrees C. A high temperature cure results in a higher molecular weight and a more highly cross-linked polymer. Only about 0.2% of the heat input is actually used in the curing of the paint film in conventional ovens (see Appendix I).

Since most manufactured products are painted for decorative and protective purposes, there are large volumes of industrial finishes used. In 1976, approximately 81.24 billion cubic feet of natural gas were used to bake paints in product finishing lines in the United States.*¹ However, the energy shortage of the 70's and the increasing energy costs of the 70's and 80's have led to renewed interest in more efficient alternative curing methods using energy sources other than oil or natural gas.

Alternative systems based on electricity, which can be generated from a variety of sources, present the opportunity for meeting the needs of an efficient and economical curing system. One of the most promising and most widely accepted paths for translating electricity into chemically usable energy is radiation, and specifically ultraviolet (UV) light radiation (see Appendix II). UV curing itself or followed by some degree of thermal curing, consumes only one-third to one-fourth the energy used in natural gas direct fired oven

*U.S. Department Comm. Bureau of Census, Annual Survey of Manufacturers, 1976, "Fuels and Electric Energy Consumed," Code M76(AS)-4.1.

heating because most of the energy is used for curing the resin and not in heating the part. Also, the air mass in the curing tunnel is not heated nor is any heated air for dilution of solvent vapors needed, because the coatings are solvent-free and completely reactive. Due to its greater chemical excitation specificity, UV curing is more energy efficient than thermal.*² Furthermore, there is no need for incineration or other pollution controls, since there is no volatile organic emission. This process allows very rapid cure (sometimes in the order of 3 to 30 seconds) as opposed to the longer time (3 to 30 minutes) required for a thermal cure so that throughput is increased; it also saves plant space since UV curing does not require the huge ovens necessary for conventional processes. As a further benefit, UV coatings can be cured on heat sensitive substrates. Unlike electron beam irradiation, no inert atmosphere in the curing chamber nor special shielding for personnel or equipment is required for UV curing. Furthermore, the capital equipment costs of UV are lower. Despite higher material costs, the overall costs are lower for UV curing of paints than for curing by direct gas fired ovens as illustrated in Table 1. Operating experience with UV light curing processes has indicated an overall operating cost which is approximately one-fourth that of natural gas.

Although UV curing of clear and lightly pigmented coatings, such as inks is well established in many applications from flooring to metal decorating, UV curable industrial quality paints had not been developed previously. There are two obstacles to the use of heavily pigmented coatings on three-dimensional objects. One difficulty is the through cure of a largely opaque coating and the other difficulty is that the shape of the object itself can permit non-uniform irradiation unless precautions are taken.

*"In contrast (to a thermal process), the energy associated with UV is higher than with visible or infrared radiation and excited states are formed directly when irradiated with UV without excitation of intermediate vibrational, rotational and translational levels," (Hulme, B. E., "Some Aspects of the Pigmentation of UV Curing Systems," given at the Mass. Society for Coatings Technology Seminar.)

TABLE 1

COMPARATIVE ECONOMICS OF HIGH SPEED CURING SYSTEMS
(FROM THE 1974 FORD FOUNDATION STUDY)

Parameter	Thermal Oven			UV		
A. System Step						
Line Speed (fpm)	100	200	300	100	200	300
Beam Power (kW)	---	---	---	6.2 ^a	12.4 ^a	18.6 ^a
Power Consumption (kW)	1,700 ^b	3,400 ^b	5,100 ^b	170	340	510
B. Operating Costs (\$/h)						
Power	6.00 ^c	12.00 ^c	18.00 ^c	3.40 ^d	6.80 ^d	10.20 ^d
Maintenance	0.90	1.80	2.70	1.50	3.00	4.50
Total Costs	6.90	13.80	20.70	4.90	9.80	14.70

a - Based on UV cure requirements of 1 joule/cm².

b - For gas fired ovens with afterburner and heat recovery system.

c - Based on gas cost in 1974 of \$1.00/1,000 cu ft (0.35¢/kWh).
Proportionally higher in 1978.

d - Based on electrical power costs in 1974 at 2¢/kWh.
Proportionally higher in 1978.

The starting point of this program was the matching of resins, photoinitiators, and pigments which resulted in coatings that cured by UV radiation. The end point was the application of these coatings to pre-fabricated metal structures to evaluate the viability of this technique in producing commercially acceptable painted products. These paints produced films that were hard, adherent, and opaque at a nominal thickness of 1 mil (0.001 in.).

2. CONCLUSIONS

1. UV radiation can cure paints. It requires only one-third the energy of the conventional thermal method of curing.
2. UV curable one-coat enamels, topcoats and primers have been developed which can produce hard and adherent films after UV radiation. The paints are cured by either photoinduced free radical polymerization or photoinduced cationic polymerization, depending on the formulation.
3. Unfocused ultraviolet radiation can cure paint on three dimensional metal objects which indicates its potential use for finishing industrial products such as appliances, metal furniture, automobiles, and other manufactured goods.

3. RECOMMENDATIONS

Since UV radiation curing of paint could replace conventional thermal curing in industrial product finishing lines, the further study of UV curable paint formulations for specific end uses is recommended.

4. EXPERIMENTAL

In order to develop UV curable paints suitable for application on prefabricated structures, the following tasks were performed:

- identify materials
- formulate and test paints
- evaluate paints
- apply paints
- pilot plant testing.

In a normal paint development, the resin binder is chosen first, then suitable pigments are added, followed by various modifiers such as extenders and flow control agents. In the development of UV curable paints, the procedure is reversed owing to the interrelationship of those components with each other and with the absorption spectra of the photoinitiators and the UV radiation source output spectra.

Experimental data confirmed that these systems require testing in combination. Furthermore, this work has confirmed the previously drawn conclusion that optimization data on clear coatings are not necessarily helpful for optimizing pigmented coatings.

The spectral absorbance of the rutile titanium dioxide (Fig. 1)²⁻⁷ screens light through most of the ultraviolet and visible ranges leaving only a small window through which the UV radiation can penetrate.⁸ Therefore, it is necessary to select photosensitizers or photoinitiators which absorb strongly in this region and also to select lamps which have a large output in this area, such as iron iodide lamps, other metal halide lamps, Xenon lamps, and the Fusion Systems Corp. non-mercury D or V bulbs (Fig. 2).⁹ On the other hand, use of a medium pressure mercury lamp resulted solely in surface cure. The non-mercury bulbs were chosen and mounted on a modified conveyorized UV processor.

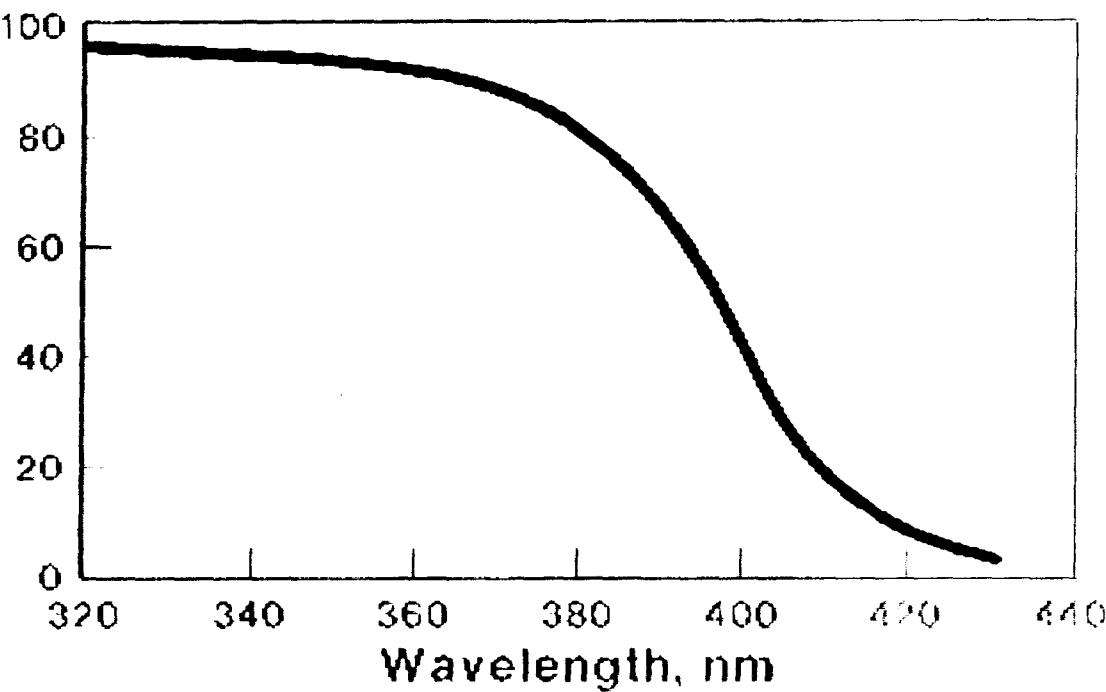
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ABSORPTION BY RUTILE TiO_2

Absorption, %



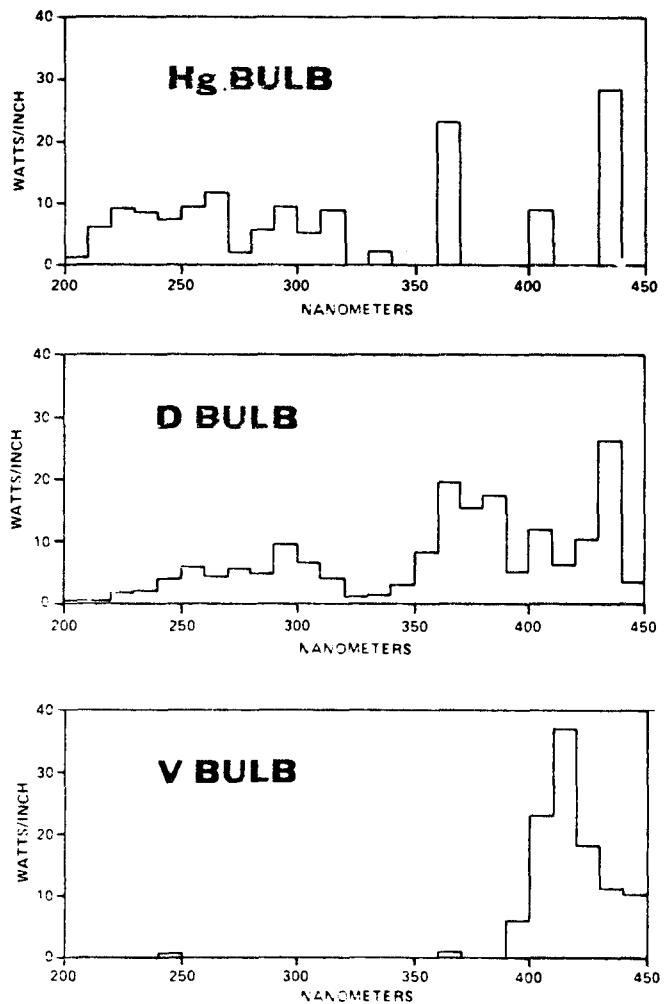


Figure 2
OUTPUT SPECTRA OF FUSION BULBS

For the preliminary screening tests, paints were applied on three different substrates: iron phosphated, zinc phosphated, and untreated cold rolled steel panels. They were applied at a thickness of 1 mil using a Bird film applicator. The coated panels were passed through the processor where they were irradiated.

The initial screening tests conducted were crosshatch adhesion and pencil hardness. Coatings passing these tests were evaluated by more intensive screening using Taber abrasion, conical mandrel bend, and impact resistance tests. A more complete evaluation of UV curable paints was conducted by subjecting them to salt spray, humidity, Weather-o-meter, and Fade-o-meter tests.

To simulate the shapes, angles and configurations of parts painted industrially, 3 inch by 5 inch metal file boxes were chosen. The exteriors and interiors of the file boxes were spray painted on a laboratory scale finishing line which was set up to demonstrate the practicality of the system and to allow economic data to be generated.

5. RESULTS AND DISCUSSION

5.1 Identification of Materials; Formulation and Testing of Paints

In Tasks 1 and 2 of the program, suitable photosensitive materials were identified and formulated into paints. The pigments, which largely determined the spectral absorptivity of the coatings were selected first.

5.1.1 Pigments

An important criterion in the choice of pigments is their effect on UV light penetration of films. High opacity at a thickness of about 1 mil was desired. The hiding power of a pigment (Appendix II) is determined by its ability to scatter and absorb visible radiation. For white pigments, hiding is accomplished primarily by light scattering which depends on the difference in refractive index between pigment and binder. Pappas and Kuhhirt⁸ have examined the pigments under consideration for their effect on the ability of UV to cure pigmented films. Some results of this work are given in Table 2.

In Table 2 the amount of pigment and film thickness were adjusted to provide films with approximately the same hiding power. This table illustrates that, as expected, films with greater hiding power allow less penetration of UV radiation.

Cure times of formulations using three grades of titanium dioxide, which was selected for pigmenting white topcoats and one-coat enamels in a free-radical system, are similar. Test results on an anatase TiO_2 (Unitane 0-310), rutile TiO_2 (Tioxide RHD6X), and rutile TiO_2 (Titanox 2020) are

TABLE 2

HIDING POWER OF WHITE PIGMENTS IN UV-CURED PAINTS*

<u>Pigment</u>	<u>PVC^a</u>	<u>Film Thickness, mils.</u>	<u>SX(550 nm)^b</u>	<u>Cure %^c</u>
ZnO	20	0.82	0.83	11
Sb ₂ O ₃	20	0.96	1.1	94
PKT	20	0.41	0.72	92
ZnS	20	0.34	0.83	94
TiO ₂ (anatase)	10	0.36	0.88	67
TiO ₂ (rutile)	10	0.24	0.88	74
TiO ₂ (anatase)	20	0.43	1.8	42
TiO ₂ (rutile)	10	0.65	2.2	57

^aPVC is pigment volume concentration.^bSX is Kubelka-Munk measure of hiding power.^c% of film cured by 200 w/in. UV-lamp.*Pappas and Kuhhirt, J. Paint Tech. 47, p. 43, 1975.

shown in Table 3. Rutile titanium dioxide (TiO_2) is used universally by the paint industry because it provides superior hiding power at lower pigment volume concentration and thickness, and has less chalking than anatase.

However, it can be seen in Figure 1²⁻⁷ that the spectral absorbance of the rutile titanium dioxide screens out most of the ultraviolet and visible range, leaving only a small area through which the ultraviolet radiation can penetrate. TiO_2 pigmented formulations could not be cured by the 200 W/in medium pressure mercury lamps used for clear coatings, requiring instead 300 W/in lamps, indicating that TiO_2 has significant effects on cure.

Other pigments were also investigated. A pigmentation study was conducted to establish typical pigment loadings for white, black and gray paints with complete hiding power.

Although the loading has not been optimized, 40% by weight of TiO_2 gives complete hiding in a white pigmented coating. The 40% by weight level of Mapico black gives complete hiding for a black pigmented coating and the 40% level of a mixture of 95% TiO_2 and 5% Mapico black yields a pigmented coating whose color closely approximates ANSI 61 light gray machinery enamel.

Paints of several different colors were formulated based on white paint P28. The coatings are listed in Table 4. These paints cured in 3 to 30 seconds, with faster curing paints usually resulting in a smooth coating. Yellows and reds are particularly slow to cure. The colors were clear and bright, with little sign of yellowing.

Primers were pigmented with red iron oxide alone, as a corrosion inhibiting pigment; red iron oxide, Halox BW-111, as corrosion inhibitors, and Fibrene C-400 as a pigment extender. The results are shown in Tables 5 and 6.

TABLE 3- PROPERTIES OF COATINGS CONTAINING TITANIUM DIOXIDE PIGMENT AND 1.00%
2-ETHYLANTHRQUINONE (EAQ) CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P254								
TS3577-11	30.00g	5 Passes						
TPGDA	5.00	12.5 ft/min	100%	100%	96%	4B	3B	4B
EEA	5.00	300 w/in						
EAQ	0.20	D Bulb	100%	100%	96%	3B	3B	4B
Tioxide								
R-HD6X	20.00							2/4/81
P255								
TS3577-11	30.00g	4 Passes						
TPGDA	5.00	12.5 ft/min	100%	100%	11%	F	HB	4B
EEA	5.00	300 w/in						
EAQ	0.20	D Bulb	100%	100%	20%	H	F	4B
Unitane								
O-310	20.00							2/4/81
P258								
TS3577-11	30.00g	5 Passes						
TPGDA	5.00	12.5 ft/min	100%	100%	10%	F	HB	3B
EEA	5.00	300 w/in						
EAQ	0.20	D Bulb	100%	100%	100%	2H	F	2B
Titanox								
2010	26.60							2/5/81

Fusion Systems Corp. D. Bulb
 Lamp Width (Irradiation Zone) 8.25 in.
 Effective Radiation Time is 3.3 sec/ pass.

TABLE 4 - PROPERTIES OF COATINGS PHOTONITIATED WITH 2-CHLOROTHIOXANTHONE (CTX)
AND N-METHYL DIETHANOLAMINE (MDEA) , CONTAINING VARIOUS PIGMENTS

Based On
P282:
TS 3577-11 30.00 g
HDDA 10.00
EEA 5.00
CTX 0.18
MDEA 0.45
TiO₂ 2010 30.00

Formulation	Number of Passes at 12.5 ft/min and D Bulb, 300 W/in	Date	Comments About Coating
P310 8.00 g P282 2.00 Thalo Green	1 Pass	5/6/81	Smooth
P311 8.00 g P282 2.00 Thalo Blue	1 Pass	5/6/81	Smooth
P312 8.00 g P282 2.00 Brown Fe Oxide	2 Passes	5/6/81	Wrinkled
P313 8.00 g P282 2.00 Fast Red	1 Pass	5/6/81	Smooth
P314 8.00 g P282 2.00 Red Fe Oxide	3 Passes	5/6/81	Wrinkled
P315 8.00 g P282 2.00 Chrome Yellow	3 Passes	5/6/81	Wrinkled
P316 8.00 g P282 2.00 Carbizol Violet	1 Pass	5/6/81	Smooth
P317 8.00 g P282 2.00 Raw Umber	3 Passes	5/6/81	Wrinkled
P318 8.00 g P282 2.00 Chromium Oxide	2 Passes	5/6/81	Smooth
P319 8.00 g P282 2.00 Hansa Yellow	4 Passes	5/6/81	Smooth
P320 8.00 g P282 2.00 Yellow Oxide	3 Passes	5/6/81	Wrinkled
P321 8.00 g P282 2.00 Black	2 Passes	5/6/81	Smooth

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TABLE 5 - PROPERTIES OF PRIMERS PIGMENTED WITH VARIOUS PIGMENTS CONTAINING 2-ETHYLANTHRAQUINONE (EAQ) CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P.P. 206								
TS3577-11	28.20 g	5 Passes	22%	100%	100%	H	6B	3B
Iron Oxide	22.50	12.5 ft/min						12/31/80
Zinc Chromate	4.50	300 w/in						
EAQ	0.14	D Bulb	100%	100%	100%	H	<6B	2B
P.P. 207 I								
TS3577-11	28.20 g	5 Passes	100%	100%	100%	HB	2B	3B
Iron Oxide	22.50	12.5 ft/min						1/9/81
Chemlink Z	0.56	300 w/in	100%	100%	100%	8	2B	2B
EAQ	0.14	D Bulb						1/12/81
P.P. 207 II								
TS3577-11	28.20 g	5 Passes	100%	100%	22%	HB	HB	4B
Iron Oxide	22.50	12.5 ft/min						1/14/81
Chemlink Z	0.56	300 w/in	100%	100%	100%	HB	2B	4B
EAQ	0.14	D Bulb						1/16/81
P.P. 207 III								
TS3577-11	28.20 g	5 Passes	100%	100%	100%	8	2B	2B
Iron Oxide	22.50	12.5 ft/min						1/15/81
Chemlink Z	0.56	300 w/in	100%	100%	100%	HB	8	2B
EAQ	0.14	D Bulb						1/19/81

TABLE 6 - THE PROPERTIES OF UV CURABLE EPOXY PRIMER

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
PCP 288								
Red Iron Oxide	7.14	D Bulb	100 %	100 %	4H	3H	3H	3/12/81
Halox BW-111	11.25	300 w/in						
Fibrene C-400	21.51	12.5 ft/min						
Bentonite 34	0.61	3 Passes for Steel						
ERL 4221	49.74	5 Passes for						
Limonene Dioxide	9.95	Fe Phos.						
FC-509	3.00	and Zn Phos.						
FC-510	1.00							
PCP 289								
Red Iron Oxide	7.14	D Bulb	100 %	100 %	H	2H	H	3/12/81
Halox BW-111	11.25	300 w/in						
Fibrene C-400	21.51	12.5 ft/min						
Bentonite 34	0.61	3 Passes for Steel						
ERL 4221	49.74	5 Passes for						
Limonene Dioxide	9.95	Fe Phosph &						
FC-508	4.00	Zn Phos.						
FC-510	1.00							
PCP 290								
Red Iron Oxide	7.14	D Bulb	100 %	100 %	3H	3H	H	3/12/81
Halox BW-111	11.25	300 w/in						
Fibrene C-400	21.51	12.5 ft/min						
Bentonite 34	0.61	2 Passes for Steel						
ERL 4221	29.00	3 Passes for						
Limonene Dioxide	30.69	Zn Phos. &						
FC-509	3.00	Fe Phos.						
FC-510	1.00							
PCP 291								
Red Iron Oxide	7.14	D Bulb	100 %	100 %	4H	4H	F	3/12/81
Halox BW-111	11.25	300 w/in						
Fibrene C-400	21.51	12.5 ft/min						
Bentonite 34	0.61	5 Passes for						
ERL 4221	29.00	Zn Phos.						
Limonene Dioxide	30.69	3 Passes for Steel						
FC-508	4.00							
FC-510	1.00							

5.1.2 Photoinitiator Systems

In the study of photoinitiator systems, several compounds listed in Table 7 were tested in clear coatings for UV photosensitivity. Highly photoactive compounds were then examined in pigmented coatings. Paints formulated for the photoinitiator study were applied on zinc phosphated, iron phosphated and untreated cold rolled steel panels. To aid in the selection of photoinitiators suitable for titanium dioxide-pigmented UV curing paints, the spectral properties of selected compounds listed in Table 8 were studied further. The most promising photoinitiators, 2-chlorothioxanthone (CTX), and benzil, absorb in the spectral range which is not screened by TiO_2 , Figure 3.²⁻⁷

In any given system, free radical production is dependent upon the chemical characteristics and reaction pathways of the individual components. The effects of concentration on cure time and physical properties of films photoinitiated with 2-chlorothioxanthone (CTX), benzil, and 2-ethylanthraquinone (EAQ)²⁻²⁰ were examined. In each case, cure times and physical properties of films which were cured using Fusion Systems Corporation D bulbs⁹ reached an optimum and then declined with increasing photoinitiator concentration.

Photoinitiator studies using 2-chlorothioxanthone in the 0.2 to 1.0% levels gave harder, more adherent films, though yellowing occurred at concentrations greater than 0.4% as shown in Table 9. On the other hand, benzil-photoinitiated films in concentrations of 0.2 to 3.0% were hard and adherent with no yellowing, as shown in Table 10. Table 11 shows that the optimum concentration for CTX was 0.4%, benzil was 1.0% and EAQ was 0.5%.

Co-initiators such as tertiary amines, arylchloromethyl and chlorosulfonyl compounds were added for H-transfer, electron transfer and energy transfer. The following compounds exhibited co-reactivity in both clear

TABLE 7
EFFECT OF PHOTONINITIATOR CONCENTRATION ON CURING HDDA

Photo Initiator	Wt. % Photoinitiator								Reaction
	2	1	0.5	0.2	0.1	0.05	0.02	0.01	
V10	Thick Skin	Thick Skin	Thick Skin	Thru Cure	Thru Cure	Thru Cure	NR (No Reaction)	NR	Reaction Passes
	1*	1	1	1	2	4			
V30	"	"	"	Thick Skin	"	"	Bottom Cure	NR	Reaction Passes
	1	1	1	1	2	4	6		
DEAP	90% Cure	90% Cure	90% Cure	90% Cure	"	"	NR	NR	Reaction Passes
	1	1	1	1	2	5			
1116 DAROCUR	90% Cure	--	Thru Cure	Thru Cure	90% Cure	"	Partial Cure	--	Reaction Passes
	1	1	1	1	2	3	6		
1173 DAROCUR	Thick Skin	Thick Skin	Thick Skin	Thick Skin	Thru Cure	"	Thru Cure	--	Reaction Passes
	1	1	1	1	2	3	9		
651 IRGACURE	Thick Skin	Thick Skin	Thick Skin	Thru Cure	Thru Cure	"	Thru Cure	Thru* Cure	Reaction Passes
	1	1	1	1	1	1	2	5	
Benzo- phenone	Trace Reaction	NR	NR	NR	NR	NR	NR	NR	Reaction Passes
	10								
Mischler's Ketone	--	--	Thin Skin	Thin Skin	Thin Skin	Thin Skin	Thin Skin	--	Reaction Passes
			1	1	1	1	1		
Chlorothio- xanthone	--	--	Partial Cure	Thick Skin	Thick Skin	Thick Skin	Thru Cure	--	Reaction Passes
			10	10	10	10	6		

Ten gram samples of HDDA in 2.5 in. diameter aluminum dishes RPC Processor with 2 lamps, 200 W/in. each, 40 ft/min. Irradiation zone 8 ft. long. Effective irradiation time is 2.5 sec/pass. If did not react, given 10 passes.

TABLE 7 (continued)

<u>Photo Initiator</u>	<u>2</u>	<u>1</u>	<u>0.5</u>	<u>0.2</u>	<u>0.1</u>	<u>0.05</u>	<u>0.02</u>	<u>0.01</u>	
BZ/MK	Thick Skin 10	Thick Skin 10	90% Cure 10	90% Cure 10	Thick Skin 10	Thick Skin 10	Thin Skin 10	--	Reaction Passes
BZ/MK Chloroform	Thru Cure 5	--	--	Thru Cure 5	--	--	--	--	Reaction Passes
BZ/MDEA**	Thru Cure 2	Thru Cure 1	Thru Cure 1	Thru Cure 2	Thru Cure 6	Thru Cure 9	NR	--	Reaction Passes
BZ/MDEA Acetone	Thru Cure 1	--	--	Thru Cure 2	--	--	--	--	Reaction Passes
MK/MDEA	Thin Skin 1	Thin Skin 1	Thin Skin 1	Thin Skin 1	Thin Skin 1	Thin Skin 1	Thru Cure 10	--	Reaction Passes
CTX/MDEA	--	Thru Cure 6	Thru Cure 2	Thru Cure 2	Thru Cure 2	Thru Cure 1	--	--	Reaction Passes
Acetone	NR								
Chloroform									

All initiators dissolved in HDDA and also in solvent. The solvent was acetone except for Mischler's ketone combinations where chloroform was used. The effects of solvents were negligible except where noted.

*The numbers refer to the number of passes through the UV processor at 40 ft/min.; each pass provides 2.5 seconds irradiation.

**MDEA = methyl diethanolamine.

TABLE 8-SPECTROSCOPIC PROPERTIES OF PHOTONITIATORS

Solvent	Compound	E_S (kcal/mole)	E_S (nm)	ϵ_f	τ_E (nsec)	τ_{sec}	E_T (kcal/mole)	E_T (nm)	λ_{max} (nm)	τ_T (usec)	ϵ_{254}	ϵ_{313}	ϵ_{366}	ϵ_{380}	Favored Reaction Pathway	Reference	
n	Acetophenone	78.7	363	$< 10^{-6}$		1.00	73.7	388		3.5	10^3	4×10^1	5			11	
p				0.00			74.1	386		0.41	6×10^3	6×10^1	1				
n	Anthracene	76.3	375	0.27	4.9	0.75	42.0	681			1.2×10^5	1.2×10^3	1.7×10^3			11	
p		76.3	375	0.27	5.3	0.72	42.7	670			1.5×10^5	1.2×10^3	1.9×10^3				
n	Anthraquinone						62.4	458								11	
p							62.7	456									
n	Benzil	59.0	485	10^{-3}		0.92	53.4	535			1.5×10^4	5×10^2	7×10^1			11	
p							54.3	526			1.6×10^4	1.2×10^2	2×10^2				
n	Benzoin Ethers						~ 73									Intramolecular	
p	(e.g. Benzoin Me Ether)															42	
n	Benzophenone	75.4	379	4×10^{-6}	0.005	1.00	68.6	417		12	1.7×10^4	5×10^1	7×10^1			11	
p		74.4	384	0.00			69.2	413			1.8×10^4	1.4×10^2	5×10^1				
n	CTX						62		~ 385				4.0×10^3	4.7×10^3		Intermolecular	41, 42
p																	
n	MK					1.00				27						Intermolecular	11
p																	
n	$\text{Ph}_2\text{I}^+\text{AsF}_5$	95				0.21	68		~ 461		1.3×10^4	1.1×10^4	2.8×10^4			Cat.	30, 31
p									~ 280								
n	$\text{Ph}_3\text{S}^+\text{AsF}_6$	99				0.17	74		~ 350							Cat.	30, 31
p																	
n	OSC						~ 63		~ 456							Intramolecular	3, 11
p																	
n	TX						65.5	437								Intermolecular	11, 41
p																	
n	Xanthone	77.6	368				74.0	386		50	1×10^4	3×10^3	2×10^2	6×10^1		Intermolecular	11
p							74.0	386			9×10^3	2.5×10^3					

SYMBOLS FOR TABLE 8

E_s	Origin of lowest excited singlet state.
ϕ_f	Quantum yield of fluorescence (in solution at room temperature).
τ_s	Lifetime of lowest excited singlet state (in solution at room temperature).
ϕ_{ics}	Quantum yield of intersystem crossing or triplet formation (in solution at room temperature).
E_T	Origin of lowest excited triplet state (generally in a glass at 77°K).
τ_T	Lifetime of lowest excited triplet state (in solution at room temperature).
ϵ_{254}	Extinction coefficient at 254 nm (in solution at room temperature).
ϵ_{313}	Extinction coefficient at 313 nm (in solution at room temperature).
ϵ_{366}	Extinction coefficient at 366 nm (in solution at room temperature).
ϵ_{385}	Extinction coefficient at 385 nm (in solution at room temperature).
n	Nonpolar solvent, generally saturated hydrocarbon or benzene.
p	Polar solvent, generally alcohols or EPA (5:5:2 ether:isopentane:ethanol).
Cationic	Cationic reaction mechanism.
Intramolecular	Photocleavage reaction mechanism
Intermolecular	Intermolecular reaction mechanism

Figure 3

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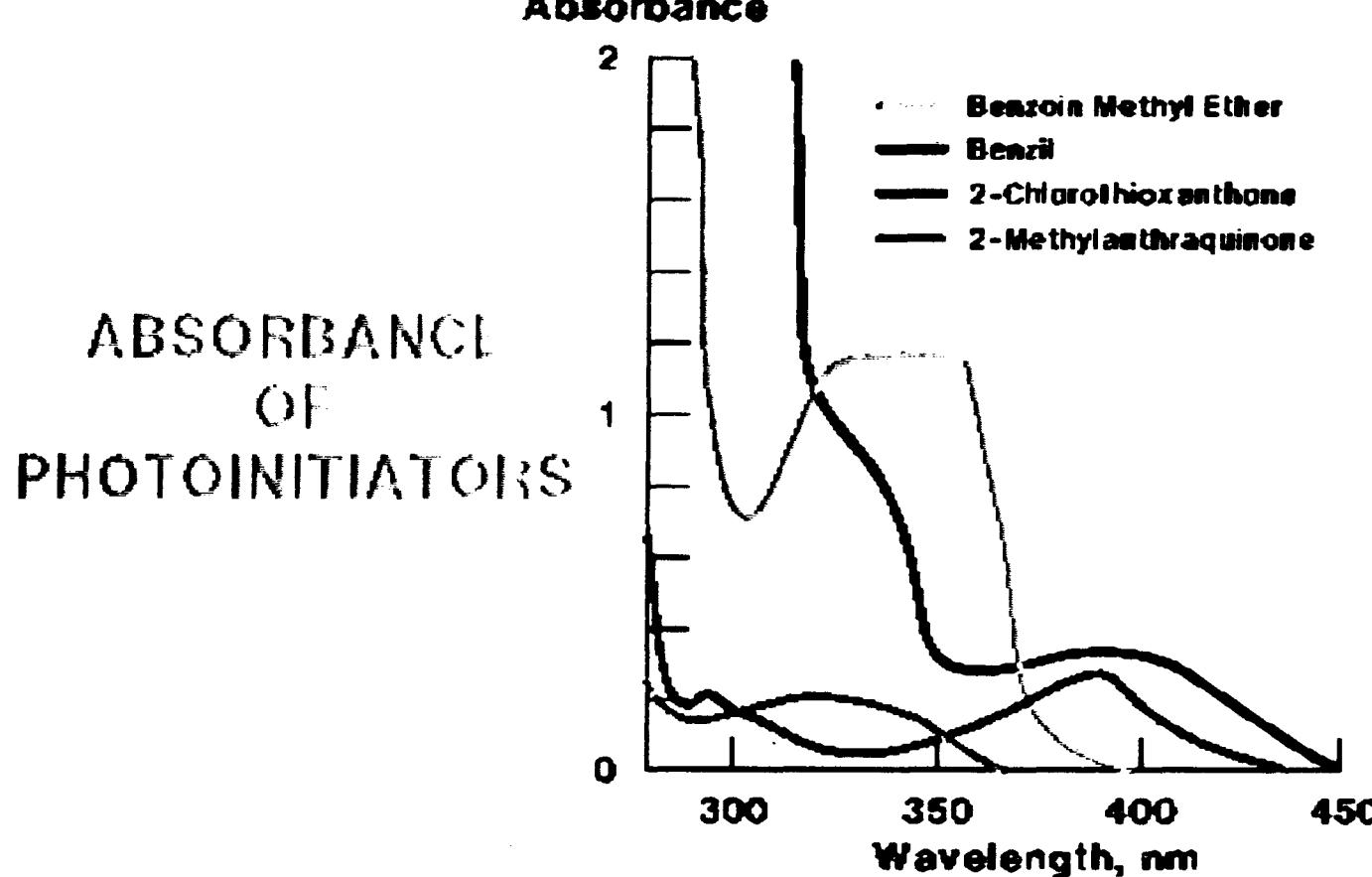


TABLE 9—PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.20% TO 1.00%
2-CHLOROTHIOXANTHONE (CTX) CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test	Comments
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P 140A (0.20% CTX) TS 3577-11 30.00 g	6 Passes 12.5 ft/min 300 W/in	99% 100%	100% 100%	40% 93%	F 2H	H HB	3B B	9/4/80 9/8/80	
HDDA 10.00 EEA 5.00 CTX 0.90 TiO_2 20.00									
P 140E (0.30% CTX) TS 3577-11 30.00 g	5 Passes 12.5 ft/min 300 W/in	70% 100%	97% 100%	32% 21%	6B < 6B	6B < 6B	< 6B < 6B	9/23/80 9/25/80	
HDDA 10.00 EEA 5.00 CTX 0.14 TiO_2 20.00									
P 140B (0.40% CTX) TS 3577-11 30.00 g	3 Passes 12.5 ft/min 300 W/in	98% 100%	74% 100%	12% 10%	2B 2B	4B B	< 6B < 4B	9/4/80 9/8/80	
HDDA 10.00 EEA 5.00 CTX 0.18 TiO_2 20.00									
P 140F (0.50% CTX) TS 3577-11 30.00 g	3 Passes 12.5 ft/min 300 W/in	100% 100%	100% 100%	12% 10%	HB HB	3B < 6B	< 6B < 6B	9/23/80 9/25/80	
HDDA 10.00 EEA 5.00 CTX 0.18 TiO_2 20.00									
P 140C (0.60% CTX) TS 3577-11 30.00 g	3 Passes 12.5 ft/min 300 W/in	93% 93%	53% 98%	0% 7%	HB HB	2H H	3B 4B	9/4/80 9/8/80	Yellowing
HDDA 10.00 EEA 5.00 CTX 0.27 TiO_2 20.00									
P 140D (0.80% CTX) TS 3577-11 30.00 g	2 Passes 12.5 ft/min 300 W/in	45% 100%	60% 100%	1% 6%	3B 3B	3B 3B	< 6B 5B	9/4/80 9/8/80	Yellowing
HDDA 10.00 EEA 5.00 CTX 0.36 TiO_2 20.00									
P 140 (1.00% CTX) TS 3577-11 30.00 g	2 Passes 12.5 ft/min 300 W/in	95% 100%	88% 100%	11% 45%	B F	B HB	< 6B 6B	9/9/80 9/11/80	Yellowing
HDDA 10.00 EEA 5.00 CTX 0.45 TiO_2 20.00									

TABLE 10- PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.20% TO 3.00% BENZIL CURED WITH D BULB

A. Grosset
e.s. 12-12-80

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P 148A (0.20% Benzil) TS 3577-11 30.00g HDDA 10.00 EEA 5.00 Benzil 0.90 TiO ₂ 20.00	7 Passes 12.5 ft/ 300 W/in	98% 100%	100% 100%	21% 98%	H 2H	H 3H	B 2B	9/8/80 9/10/80
P 148B (0.60% Benzil) TS 3577-11 30.00g HDDA 10.00 EEA 5.00 Benzil 0.27 TiO ₂ 20.00	3 Passes 12.5 ft/min 300 W/in	76% 97%	83% 100%	2% 20%	H H	H H	3B B	9/8/80 9/10/80
P 148 (1.00% Benzil) TS 3577-11 30.00g HDDA 10.00 EEA 5.00 Benzil 0.45 TiO ₂ 20.00	2 Passes 12.5 ft/min W/in	84% 70%	98% 100%	30% 83%	H H	F 2H	4B HB	9/9/80 9/11/80
P 148 (1.00% Benzil) TS 3577-11 30.00g HDDA 10.00 EEA 5.00 Benzil 0.45 TiO ₂ 20.00	3 Passes 12.5 ft/min 300 W/in	62% 96%	100% 100%	39% 37%	F HB	F F	6B 2B	11/4/80 11/6/80
P 148C (2.00% Benzil) TS 3577-11 30.00g HDDA 10.00 EEA 5.00 Benzil 0.90 TiO ₂ 20.00	2 Passes 12.5 ft/min 300 W/in	97% 100%	100% 100%	46% 79%	3B F	F 2H	< 6B 2B	9/9/80 9/11/80
P 148D (3.00% Benzil) TS 3577-11 30.00g HDDA 10.00 EEA 5.00 Benzil 1.35 TiO ₂ 20.00	2 Passes 12.5 ft/min 300 W/in	100% 100%	100% 100%	55% 75%	F H	B HB	2B 2B	9/9/80 9/11/80

TABLE 11—PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.50% to 4.00% 2-ETHYLANTHRAQUINONE (EAQ) CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P 160A (0.50% EAQ) TS 3577-11 30.00 g HDDA 10.00 EEA 5.00 EAQ 0.23 TiO ₂ 20.00	7 Passes 12.5 ft/min 300 W/in	100%	97%	100%	6H	HB	2B	11/12/80
P 160B (1.00% EAQ) TS 3577-11 30.00 g HDDA 10.00 EEA 5.00 EAQ 0.45 TiO ₂ 20.00	5 Passes (steel) 8 Passes (Zn Phos) 8 Passes (Fe Phos) 12.5 ft/min 300 W/in	100%	100%	100%	8H	3H	B	11/12/80
P 160C (2.00% EAQ) TS 3577-11 30.00 g HDDA 10.00 EEA 5.00 EAQ 0.90 TiO ₂ 20.00	7 Passes 12.5 ft/min 300 W/in	100%	100%	100%	HB	4B	2B	11/12/80
P 160D (3.00% EAQ) TS 3577-11 30.00 g HDDA 10.00 EEA 5.00 EAQ 1.35 TiO ₂ 20.00	7 Passes 12.5 ft/min 300 W/in	100%	100%	100%	3B	2B	3B	11/12/80
P 160E (4.00% EAQ) TS 3577-11 30.00 g HDDA 10.00 EEA 5.00 EAQ 1.80 TiO ₂ 20.00	7 Passes 12.5 ft/min 300 W/in	100%	100%	100%	<6B	<6B	3B	11/13/80
		100%	100%	100%	5B	5B	4B	11/17/80

and pigmented coatings:

N-methyldiethanolamine (MDEA) ^{13,21}
diethylenetriamine (DETA)
monoethanolamine (MEOA)
diethanolamine (DEOA)
triethanolamine (TEOA)
N,N-dimethylaminoethylmethacrylate (DMEMA)
phenylethylamine (PEA)
triethylamine (TEA)
dimethylethanolamine (DMEOA)
morpholine (MORPH)
ethyl-para-dimethylaminobenzoate (EPDMAB)
quinoline sulfonyl chloride (QSC)
Virginia Chemical Co. Uvecryl P101
Virginia Chemical Co. Uvecryl P102
Virginia Chemical Co. Uvecryl P104
Stauffer Chemical Co. Vicure 30 (benzoin isobutyl ether, V-30)
Michler's Ketone (4,4-bis(N,N-dimethylamine benzophenone)

Tables 12, 13, 14, 15, 16, 17, and 18 summarize the results.
MEOA, MDEA and DETA were particularly reactive in clear coatings.

Thermal initiators were also studied for combination ultraviolet and thermal curing in clear coatings (Tables 19 and 20). Benzoyl peroxide and DTBP were among the most effective. Combinations of photoinitiators and photosensitizers were examined in clear coatings to determine photosensitivity. The results are summarized in Table 21.

Tables 22 and 23 show that a mixture of isobutyl benzoin either DTBP and MDEA can cure TMPTA by a combination of UV and thermal cure.

TABLE 12

AMINE ACCELERATORS EXAMINED FOR UV CURING OF HDDA

Materials	Concentration wt. %	Comment
i-Propylamine	0.5	NR*
Phenethylene	0.5	NR
Aniline	0.5	NR, yellows
Diphenylamine	0.5	NR, yellows
Triphenylamine	0.5	NR
Triethylamine	0.5	Partial cure
Diethanolamine	0.5	NR
Triethanolamine	0.5	Gel
Triethylene diamine	0.5	NR
Triethylamine	0.05	NR
Triethanolamine	0.05	NR

*NR = No reaction.

TABLE 13

EFFECTIVENESS OF AMINES AT 0.1% CONCENTRATION IN CURING 1,6-HEXANEDIOL DIACRYLATE WHEN USED IN CONJUNCTION WITH 0.03% V10 AND 0.1% DTBP

Amine	Result
i-Propylamine	17.5 sec. 90% cure.
Phenethylamine	12.5 sec. through cure.
Aniline	25 sec. no reaction; yellow.
Di-i-propylamine	Not available.
Di-phenylamine	25 sec. no reaction; yellow.
Triphenylamine	Insoluble in HDDA.
N,N-Dimethyl m-toluidine	17.5 sec. through cure.
N,N-Dimethyl p-toluidine	25 sec. gel.
Triethylamine	12.5 sec. gel.
Monoethanolamine	10 sec. through cure.
Diethanolamine	10 sec. through cure; dissolved in ethyl alcohol.
Triethanolamine	10 sec. through cure; dissolved in ethyl alcohol.
N-Methyl diethanolamine	10 sec. through cure.
N,N-dimethyl formamide	25 sec. slight gel.
Triethylene diamine	Insoluble in HDDA.
Di-ethylene triamine	10 sec. through cure.
N,N-Dimethylaminoethyl methacrylate	12.5 sec. through cure.

Reactivity of HDDA tested with 0.03 wt. % V10, 0.1 wt. % dtpb, 0.1 wt. % amine.

Solutions irradiated at 40 ft/min with D bulb.

TABLE 14
REACTIVITY OF AMINE ACCELERATORS IN UV AND THERMAL CURING

	Primary	Secondary		Tertiary
Aliphatic	Diethylenetriamine	3 sec.	Diethanolamine	3 sec.
	Triethylenediamine	NR	Di-i-propylamine	4.5 sec.
	Monoethanolamine	4.5 sec.		N,N-Dimethylaminoethyl-methacrylate
	i-Propylamine	7.5 sec.		N,N-Dimethyl Formamide
Aliphatic/ Aromatic	Triethylamine			4.5 sec.
	Phenethylamine	3 sec.	N-Methyl Aniline	NR
Aromatic				N,N-Dimethyl m-Toluidine
	Aniline	NR	Diphenylamine	4.5 sec.
				N,N-Dimethyl p-Toluidine
				15 sec.
				Triphenylamine

Numbers refer to irradiation time required to polymerize HDDA.

Solutions consisted of HDDA, amine 0.1%, Di-T-butyl perbenzoate 0.1% and isobutyl benzoin ether 0.03%.

TABLE 15

REACTIVITY OF AMINE ACCELERATORS IN UV AND THERMAL CURING

	Primary	Secondary	Tertiary			
Aliphatic	Diethylenetriamine	3 sec	Diethanolamine	3 sec.	Triethanolamine	3 sec.
	Triethylenediamine	NR	Di-i-propylamine	7.5 sec.	N-Methyl Diethanolamine	3 sec.
	Monoethanolamine	4.5 sec.			N,N-Dimethylaminoethyl-methacrylate	3 sec.
	i-Propylamine	7.5 sec.			N,N-Dimethyl Formamide	4.5 sec.
29 Aliphatic/ Aromatic					Triethylamine	3 sec.
	Phenethylamine	4.5 sec.	N-Methyl Aniline	12 sec.	N,N-Dimethyl m-Toluidine	7.5 sec.
Aromatic					N,N-Dimethyl p-Toluidine	3 sec.
	Aniline	NR	Diphenylamine	NR	Triphenylamine	13.5 sec.

Numbers refer to irradiation time required to polymerize HDDA.

Solutions consisted of HDDA, amine 0.1%, Di-T-butyl perbenzoate 0.075% and isobutyl benzoin ether 0.03%.

TABLE 16
ANALYSIS OF REACTIVITY OF AMINE ACCELERATORS

	Primary	Secondary	Tertiary
Aromatic Amines	Aniline: no reaction	Di-phenylamine: no reaction	Triphenylamine: insoluble in HDDA
Aliphatic/ Aromatic Amines	Phenethylamine: 12.5 sec.	----	N,N-Dimethyl m-toluidine: 17.5 sec. N,N-Dimethyl p-toluidine: 25 sec. to gel.
Aliphatic Amines	i-propylamine: 17.5 sec. Monoethanolamine: 10 sec. Triethylenediamine: insoluble in HDDA Di-ethylene triamine: 10 sec.	Di-i-propyl amine: 10 sec. Diethanolamine: 10 sec.	Triethanolamine: 10 sec. N-Methyldiethanolamine: 10 sec. N,N-Dimethyl formamide 25 sec. to gel. N,N-Dimethyl aminoethyl methacrylate 12.5 sec.

TABLE 17

EFFECT OF COMBINING AMINES WITH BENZOYL PEROXIDE
ON CURING HDDA

<u>Amine</u>	<u>Comment</u>
i-Propylamine	NR*
Phenethylene	NR
Aniline	NR, yellows
Diphenylamine	NR, yellows
Triphenylamine	NR, yellows
Triethylamine	NR
Diethanolamine	Cures
Triethanolamine	NR
Triethylene diamine	NR

Amine concentration was 0.05 wt. %, benzoyl peroxide
was 0.05 wt. % in HDDA.

*NR = No reaction.

TABLE 18

SCREENING TEST FOR MOST EFFECTIVE AMINE ACCELERATORS

Reaction	Accelerator			Di-ethylene triamine
	None	Monoethanol- amine	N-Methyl- diethanol- amine	
Time to skin (seconds)	5	6	4	2.5
Time to gel (seconds)	6	7.5	5	3
Time to through cure (seconds)	7.5	9	6	5

Solution prepared as follows:

Solution A: HDDA 9 g, V10 1 g.

Solution B: Solution A 0.3 g, Di-t-butyl perbenzoate (DTBP)
0.1 g, make up to 100 g with HDDA.

Solution C: Amine 0.5 g, HDDA 9.5 g.

Solution D: Solution B 9 g, Solution C 1 g.

Solution D contains 0.03% V10, 0.1% DTBP, 0.05% amine.

The amine solutions were irradiated at 80 ft./min.

TABLE 19
EFFECTIVENESS OF THERMAL INITIATORS IN CURING HDDA

Thermal Initiator*	Concentration Wt. %	Comment
	0.5	NR*
t-Butyl hydroperoxide	0.5	
Hydrogen peroxide (30%)	0.5	CURES
2,5-Dimethyl 2,5-bis (t-butyl peroxy) hexane	0.5	CURES
Lauroyl peroxide	0.5	NR
Dicumyl peroxide	0.5	CURES
2,5-Dimethyl 2,5-bis (hydroperoxy) hexane	0.5	NR
Azobis (isobutyronitrile)	0.5	CURES evolves N ₂
Bis (t-butyl peroxy i-propyl benzene	0.5	CURES
2,5-Dimethyl 2,5-bis (benzoyl peroxy) hexane	0.5	NR
Cumene hydroperoxide	0.5	NR
Benzoyl peroxide	0.5	CURES
Methylethyl ketone (60%)	0.5	NR
Di-t-butyl perbenzoate	0.5	CURES
Hydrogen peroxide (30%)	0.1	NR
2,5-Dimethyl-2,5-bis (t-butyl peroxy) hexane	0.1	NR
Dicumyl peroxide	0.1	NR
Azobis (isobutyronitrile)	0.1	CURES
Bis(t-butyl peroxy i-propyl) benzene	0.1	CURES
Benzoyl peroxide	0.1	CURES
Di-t-butyl perbenzoate	0.1	NR
Azobis (isobutyronitrile)	0.05	NR
Bis(t-butyl peroxy i-propyl) benzene	0.05	NR
Benzoyl peroxide	0.05	NR

*NR = No Reaction

TABLE 20

THERMAL INITIATORS EXAMINED FOR UV AND THERMAL CURING OF HDDA

Material	Concentration,* 10^{-5} moles	Time to Polymerize HDDA, Seconds
Methyl ethyl ketone peroxide (60%)	6.1	6
2,5 Dimethyl 2,5-bis (hydroperoxy) hexane	4.3	6
Cumene hydroperoxide	4.9	6
Lauroyl peroxide	1.9	4.5
Benzoyl peroxide	3.1	3
Di t-butyl perbenzoate	5.1	4.5
Bis (t-butyl peroxy 1-propyl) benzene	2.2	6
2.5-Dimethyl 2,5-bis (benzoyl peroxy) hexane	2.0	6
Dicumyl peroxide	2.8	6
2,5-Dimethyl 2,5-bis (t-butyl peroxy) hexane	2.6	6
Hydrogen peroxide (30%)	22.1	9
Azo bis (i-butyronitrile)	4.7	3

*Initiator consisted of BE 0.02 wt.%, MDEA 0.05 wt.%, peroxide 0.075 wt.%; molar concentration is based on number of moles of peroxide in 10 g. of resin.

TABLE 21

THE PHOTOSENSITIVITY OF COMBINATIONS OF PHOTONINITIATORS AND PHOTOSENSITIZERS

Conc. % Photo Initiator \	2%	1%	0.5%	0.2%	0.1%	0.05%	0.02%	0.01%
BZ/MK	Thick Skin 10 passes*	Thick Skin 10 passes	90% Cure 10 passes	90% Cure 10 passes	Thick Skin 10 passes	Thick Skin 10 passes	Thin Skin 10 passes	---
BZ/MK Chloroform	Thru Cure 5 passes	---	---	Thru Cure 5 passes	---	---	---	---
BZ/ MDEA **	Thru Cure 2 passes	Thru Cure 1 pass	Thru Cure 1 pass	Thru Cure 2 passes	Thru Cure 6 passes	Thru Cure 9 passes	NR	---
BZ/MDEA Acetone	Thru Cure 1 pass	---	---	Thru Cure 2 passes	---	---	---	---
MK/MDEA	Thin Skin 1 pass	Thin Skin 1 pass	Thin Skin 1 pass	Thin Skin 1 pass	Thin Skin 1 pass	Thin Skin 1 pass	Thru Cure 10 passes	
CTX/ MDEA	---	Thru Cure 6 passes	Thru Cure 2 passes	Thru Cure 2 passes	Thru Cure 2 passes	Thru Cure 1 pass	---	---
Acetone	NR							
Chloroform								

All initiators dissolved in HDDA and also in solvent. The solvent was acetone except for Mischler's ketone combinations where chloroform was used. The effects of solvents were negligible except where noted.

*The numbers refer to the number of passes through the UV processor at 40 ft/min.; each pass provides 2.5 seconds irradiation.

**MDEA = methyl diethanolamine.

TABLE 22
EFFECT OF REACTANT COMBINATIONS ON UV PLUS
THERMAL CURING OF TMPTA

<u>Initiator</u>	<u>Result</u>
None	No reaction.
MDEA 1.5%	No reaction.
DTBP 1.5%	Surface skin.
BE 1.5%	Surface skin.
BE 0.75% + MDEA 0.75%	Surface skin.
BE 0.75% + DTBP 0.75%	Surface skin.
DBP 0.75% + MDEA 0.75%	Surface skin.
BE 0.5% + DTBP 0.5% + MDEA 0.5%	Through cure.

Ten gram samples of TMPTA in aluminum dishes irradiated for 1.5 secs.
BE-isobutyl benzoin ether.
DTBP-di t-butyl perbenzoate.
MDEA-methyl diethanolamine.

TABLE 23

RADIATION TIME REQUIRED TO ACHIEVE UV AND THERMAL CURING* OF
 TMPTA WITH ISOBUTYL BENZOIN ETHER, DI-T-BUTYL PERBENZOATE,
 AND N-METHYL DIETHANOLAMINE

Conveyor Speed, fpm	Number of Passes to Cure	Cumulative Exposure Time to Cure Resin, Secs.
100	2	0.2
200	3	0.15
250	4	0.16

*Resin consisted of TMPTA, 0.5 wt. % isobutyl benzoin ether, 0.1 wt. % di-t-butyl perbenzoate and 0.1 wt. % methyl diethanolamine.

Table 24 shows the adhesion tests of some clear coatings. Table 25 shows that thermal initiators and accelerators increase the rate of cure of pigmented systems. Certain of these paints were formulated using photo-initiator systems consisting of 2-chlorothioxanthone (CTX) and various amines and thermal initiators (Tables 26, 27, and 28). CTX-photoinitiated films also exhibited yellowing. Of the coatings listed in Tables 26, 27 and 28, cure times depend upon the relative concentrations of CTX and amine (Fig. 4). The most promising formulation was P146, whose co-initiator was N-methyldiethanolamine (MDEA). Results of further studies of P146 are detailed in Table 27. The formulations containing CTX and MDEA cured rapidly (1 pass) and formed hard films, but adhesion was unsatisfactory. The addition of surfactants such as Ganex and FC170 to this formulation resulted in consistent adhesion to zinc phosphated panels. The formulations containing CTX and morpholine, Table 28, cured more slowly, were softer and had better adhesion than CTX and MDEA. Isopropylthioxanthone exhibited properties similar to those of CTX. Benzil and tertiary amine photoinitiator systems were evaluated (Table 29). Benzil amine systems gave fast curing, soft, white films having poor adhesion. Cure times depend upon the ratio of the concentrations of benzil and amine (Figure 5). The benzil/morpholine system described in Table 30 had good properties, but was extremely sensitive to thickness. Cured films containing benzil and morpholine had greater adhesion than films containing CTX. Their hardness and adhesion were better in lower film thicknesses (0.5 mils). Combinations of CTX and benzil with tertiary amines resulted in increased adhesion with no decrease in cure speed.

The ratio of photoinitiator to amine can significantly affect cure times as shown in Figures 4 and 5.⁶ Although EAQ and MEAQ-initiated films were yellow and slower curing than CTX and benzil containing films, they were hard and adherent. In general, it appears that the addition of tertiary amines to EAQ-photoinitiated formulations had little effect on properties as shown in Table 31, although the photocleavage of 8-quinoline

TABLE 24

ADHESION TESTS OF CLEAR COATINGS CURED BY 200 WATTS/INCH UV RADIATION AT 40 FT./MIN.

Formulation	No Photoinitiator				V10 Photoinitiator				Irgacure Photoinitiator			
	No. of Passes	ZnPhos	FePhos	Steel	No. of Passes	ZnPhos	FePhos	Steel	No. of Passes	ZnPhos	FePhos	Steel
P67 & P68 TS3577-11	20	100	100	100	5	100	100	71	4	100	100	85
P69 & P75 TS3577-11 DTBP	6	83	91	0	5	100	100	3	3	100	100	100
P70 & P71 TS3577-11 DTBP MDEA	6	98	97	7	4	99	95	94	3	100	100	92
P72A & P74 TS3577-11 HDDA EEA DTBP MDEA	5	0	0	0	6	0	0	0	4	0	0	0
P73 & P76 TS3577-11 HDDA EEA DTBP	11	0	0	0	9	100	100	-	-	-	-	-
P77 TS3577-11 HDDA EEA	-	-	-	-	11	17	0	0	8	2	2	0

TABLE 25

EFFECT OF THERMAL INITIATOR AND ACCELERATOR ON CURE OF PIGMENTED COATING
300 WATTS/INCH UV RADIATION AT 6.25 FT./MIN.

Formulation	V10 Photoinitiator				Irgacure Photoinitiator			
	No. of Passes To Cure	ZnPhos	FePhos	Steel	No. of Passes To Cure	ZnPhos	FePhos	Steel
P72 TS3577-11 HDDA EEA DTBP* MDEA** TiO ₂	6	100	100	100	3	100	100	85
P77 TS3577-11 HDDA EEA TiO ₂	11	100	100	100	6	100	100	100

*Thermal initiator.

**Thermal accelerator.

TABLE 26—PROPERTIES OF PIGMENTED COATINGS CONTAINING 2-CHLOROTHIOXANTHONE (CTX)
(Sheet 1 of 4)

Cured With H Bulb									
P96									
TS3577-11	20.00 g	2 Passes 6.25 ft/min	6%	23%	0%	2B	H	6B	7/3/80
HDDA	5.00								
EEA	2.50	300 w/in	7%	100%	0%	F	F	3B	7/7/80
DTBP	0.28								
MDEA	0.28								
Chlorothioxanthone	0.28								
TiO ₂	18.25								
Cured With H & D Bulbs									
P96									
TS3577-11	20.00 g	2 Passes 12.5 ft/min 2 Lamps @ 300 w/in	4%	25%	0%	3H	3H	3B	7/10/80
HDDA	5.00								
EEA	2.50								
DTBP	0.28								
MDEA	0.28								
Chlorothioxanthone	0.28								
TiO ₂	18.25								

TABLE 26 - PROPERTIES OF PIGMENTED COATINGS CONTAINING D.40% 2-CHLOROTHIOXANTHONE (CTX)
AND 1.00% AMINE CURED WITH D BULB (SHEET 2 of 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P 141 TS 3577-11 30.00g HDDA 10.00 EEA 5.00 CTX 0.18 DMEA 0.45 TiO ₂ 20.00	3 Passes 12.5 ft/min 300 W/in	95% 85%	89% 60%	0% 0%	4B 4B	B B	6B 5B	9/26/80 9/29/80
P 142 TS 3577-11 30.00g HDDA 10.00 EEA 5.00 CTX 0.18 TEA 0.45 TiO ₂ 20.00	2 Passes 12.5 ft/min 300 W/in	82% 74%	59% 97%	0% 0%	F F	2B B	< 6B 6B	9/26/80 9/29/80
P 143 TS 3577-11 30.00g HDDA 10.00 EEA 5.00 CTX 0.18 Morpholine 0.45 TiO ₂ 20.00	2 Passes 12.5 ft/min 300 W/in	89% 98%	83% 81%	29% 19%	2B HB	HB HB	6B 6B	9/29/80 10/1/80
P 144 TS 3577-11 30.00g HDDA 10.00 EEA 5.00 CTX 0.18 EPDMAB 0.45 TiO ₂ 20.00	2 Passes 12.5 ft/min 300 W/in	91% 89%	11% 66%	1% 0%	H 2H	F F	4B HB	9/29/80 10/1/80

TABLE 26 - PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.40% 2-CHLOROTHOXANTHONE (CTX)
AND 1.00% AMINE CURED WITH D BULB (SHEET 3 of 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P 145								
TS 3577 - 11	30.00 g	2 Passes 12.5 ft/min	90%	2%	6%	3H	B	2B
HDDA	10.00							9/29/80
EEA	5.00	300 W/in	100%	4%	2%	4H	F	HB
CTX	0.18							10/1/80
QSC	0.45							
TiO ₂	20.00							
P 146								
TS 3577 - 11	30.00 g	1 Pass 12.5 ft/min	29%	51%	0%	2H	B	2B
HDDA	10.00							9/29/80
EEA	5.00	300 W/in	77%	69%	12%	2H	HB	3B
CTX	0.18							10/1/80
MDEA	0.45							
TiO ₂	20.00							
P 147								
TS 3577 - 11	30.00 g	1 Pass 12.5 ft/min	88%	73%	0%	F	B	3B
HDDA	10.00							9/29/80
EEA	5.00	300 W/in	86%	59%	0%	HB	F	HB
CTX	0.18							10/1/80
MDEA	0.45							
Hexachloro- ethane	0.45							
TiO ₂	20.00							

TABLE 26 PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.40% 2-CHLOROTHIOXANTHONE (CTX)
CURED WITH D BULB (Sheet 4 of 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates	
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P181									
TS3577-11	30.00g	1 Pass 12.5 ft/min 300 w/in D Bulb	2%	0%	0%	<6B	<6B	<6B	12/4/80
HDDA	10.00								
EEA	5.00								
CTX	0.18								
MK	0.45								
Titanox 2010	20.00								
P182									
TS3577-11	30.00g	2 Passes 12.5 ft/min 300 w/in D Bulb	78%	8%	0%	6B	<6B	<6B	12/4/80
HDDA	10.00								
EEA	5.00								
CTX	0.18								
V10	0.45								
Titanox 2010	20.00								
P216									
TS3577-11	30.00g	2 Passes 12.5 ft/min 300 w/in D Bulb	100%	49%	0%	3H	HB	4B	1/5/81
HDDA	10.00								
EEA	5.00								
CTX	0.18								
Benzop.	0.68								
MDEA	0.45								
Titanox 2010	20.00								

TABLE 27-PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.40% 2-CHLOROTHIOXANTHONE (CTX)
AND 1.00% N-METHYLDIETHANOLAMINE (MDEA) CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P146 I								
TS3577-11 30.00g								
HDDA 10.00	1 Pass 12/5 ft/min	29%	51%	0%	2H	B	2B	9/29/80
EEA 5.00	300 w/in	77%	69%	12%	2H	HB	3B	10/1/80
CTX 0.18	D Bulb							
MDEA 0.45								
Titanox 20.00								
P146 II								
TS3577-11 30.00g								
HDDA 10.00	1 Pass 12.5 ft/min	83%	34%	0%	HB	HB	<6B	12/15/80
EEA 5.00	300 w/in	26%	90%	0%	H	F	6B	12/17/80
CTX 0.18	D Bulb							
MDEA 0.45								
Titanox 20.00								
P146 III								
TS3577-11 30.00g								
HDDA 10.00	1 Pass 12.5 ft/min	89%	4%	0%	3H	5B	6B	12/30/80
EEA 5.00	300 w/in	97%	0%	0%	3H	2B	<6B	1/2/81
CTX 0.18	D Bulb							
MDEA 0.45								
Titanox 20.00								
P235								
TS3577-11 30.00g								
HDDA 10.00	1 Pass 12.5 ft/min	91%	55%	0%	F	2B	<6B	1/6/81
EEA 5.00	300 w/in	98%	78%	0%	H	H	<6B	1/8/81
Ganex 1.35	D Bulb							
CTX 0.18								
MDEA 0.45								
Titanox 20.00								
P250								
TS3577-11 30.00g								
HDDA 10.00	1 Pass 12.5 ft/min	69%	0%	0%	HB	2B	<6B	1/6/81
EEA 5.00	300 w/in	100%	54%	0%	H	4B	4B	1/8/81
FC 171 0.45	D Bulb							
CTX 0.18								
MDEA 0.45								
Titanox 20.00								
P267								
TS3577-11 30.00g								
TPCDA 5.00	1 Pass 12.5 ft/min	99%	17%	0%	HB	2B	5B	1/22/81
EEA 5.00	300 w/in	100%	100%	1%	F	F	4B	1/26/81
CTX 0.16	D Bulb							
MDEA 0.40								
Titanox 20.00								

TABLE 28-PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.40% 2-CHLOROTHIOXANTHONE (CTX)
AND 1.00% MORPHOLINE (MORPH.) CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P143 I								
TS3577-11 30.00g	2 Passes 12.5 ft/min	89%	83%	23%	2B	HB	6B	9/29/80
HDDA 10.00								
EEA 5.00	300 w/in D Bulb	98%	81%	19%	HB	HB	6B	10/1/80
CTX 0.18								
Morph. 0.45								
Titanox 20.00 2010								
P143 II								
TS3577-11 30.00g	2 Passes 12.5 ft/min	92%	3%	0%	HB	<6B	4B	12/15/80
HDDA 10.00								
EEA 5.00	300 w/in D Bulb	60%	0%	8%	F	4B	3B	12/17/80
CTX 0.18								
Morph. 0.45								
Titanox 20.00 2010								
P143 III								
TS3577-11 30.00g	1 Pass 12.5 ft/min	81%	10%	6%	B	<6B	<6B	12/30/80
HDDA 10.00								
EEA 5.00	300 w/in D Bulb	78%	0%	0%	HB	<6B	4B	1/2/81
CTX 0.18								
Morph. 0.45								
Titanox 20.00 2010								

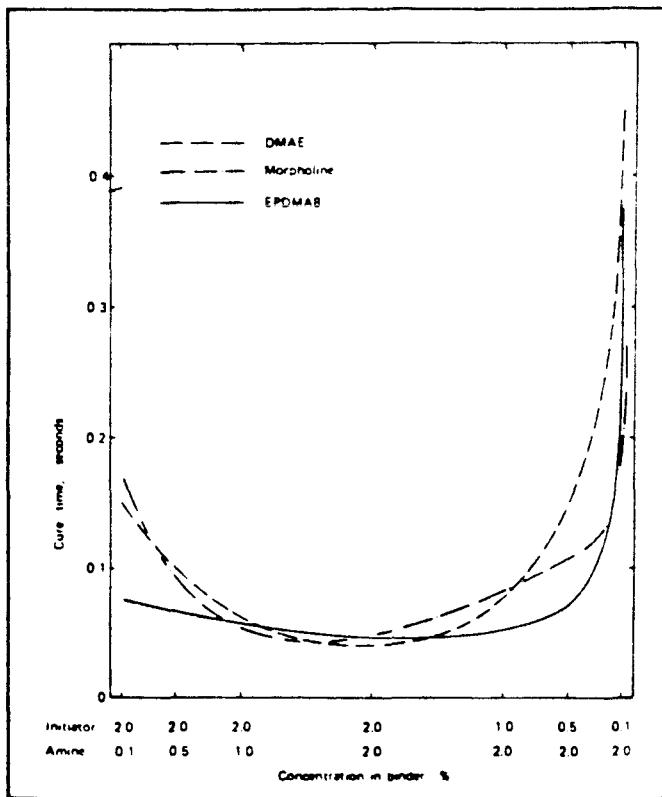


Figure 4 - Cure rates with 2-chlorothioxanthone+amine (Ref. 9)

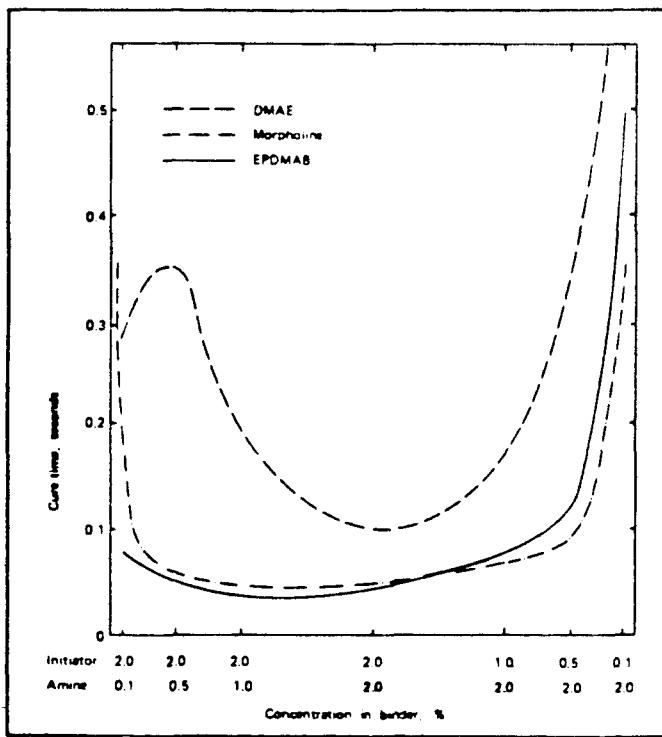


Figure 5 - Cure rates with benzil+amine (Ref. 9)

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TABLE 29—PROPERTIES OF PIGMENTED COATINGS CONTAINING 1.00% BENZIL AND 1.00% AMINE CURED WITH D BULB (SHEET 1 of 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P149								
TS 3577 - 11	30.00 g	2 Passes	93%	100%	2B	HB	5B	10/7/80
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 W/in	76%	97%	23%	HB	2H	10/9/80
Benzil	0.45							
DMEA	0.45							
TiO ₂	20.00							
P150								
TS 3577 - 11	30.00 g	1 Pass	99%	100%	20%	< 6B	< 6B	11/6/80
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 W/in	86%	100%	9%	4B	HB	11/10/80
Benzil	0.45							
TEA	0.45							
TiO ₂	20.00							
P151								
TS 3577 - 11	30.00 g	1 Pass	100%	100%	100%	2H	HB	3B
HDDA	10.00	12.5 ft/min						10/7/80
EEA	5.00	300 W/in	100%	100%	99%	2H	F	2B
Benzil	0.45							10/9/80
Morpholine	0.45							
TiO ₂	20.00							

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TABLE 29 - PROPERTIES OF PIGMENTED COATINGS CONTAINING 1.00% BENZIL AND 1.00% AMINE
CURED WITH D BULB (SHEET 2 OF 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P 152 TS 3577 - 11 30.00 g HDDA 10.00 EEA 5.00 Benzil 0.45 EPDMA 0.45 TiO ₂ 20.00	2 Passes 12.5 ft/min 300 W/in	79%	99%	41%	F	F	6B	11/4/80
P 153 TS 3577 - 11 30.00 g HDDA 10.00 EEA 5.00 Benzil 0.45 QSC 0.45 TiO ₂ 20.00	2 Passes 12.5 ft/min 300 W/in	94%	95%	43%	F	F	4B	11/6/80
P 154 TS 3577 - 11 30.00 g HDDA 10.00 EEA 5.00 Benzil 0.45 MDEA 0.45 TiO ₂ 20.00	2 Passes 12.5 ft/min 300 W/in	99%	100%	2%	HB	2B	<6B	10/7/80
		92%	100%	1%	F	2B	4B	10/9/80
		14%	88%	46%	2B	F	3B	11/7/80
		100%	91%	10%	H	F	2B	11/10/80

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TABLE 30-PROPERTIES OF PIGMENTED COATINGS CONTAINING 1.00% BENZIL AND 1.00% MORPHOLINE (MORPH.) CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P151 I								
TS3577-11	30.00g	1 Pass	100%	100%	100%	2H	HB	3B
HDDA	10.00	12.5 ft/min						10/7/80
EEA	5.00	300 w/in						
Benzil	0.45	D Bulb						
Morph.	0.45							10/9/80
Titanox	20.00							
2010								
P151 II								
TS3577-11	30.00g	2 Passes	100%	19%	25%	HB	2B	5B
HDDA	10.00	12.5 ft/min						12/15/80
EEA	5.00	300 w/in						
Benzil	0.45	D Bulb						
Morph.	0.45							12/17/80
Titanox	20.00							
2010								
P151 III								
TS3577-11	30.00g	2 Passes	95%	100%	59%	HB	B	5B
HDDA	10.00	12.5 ft/min						12/30/80
EEA	5.00	300 w/in						
Benzil	0.45	D Bulb						
Morph.	0.45							1/2/81
Titanox	20.00							
2010								

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TABLE 31—PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.50% 2-ETHYLANTHRAQUINONE (EAQ)
AND 1.00% AMINE CURED WITH D BULB (SHEET 1 OF 3)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P161 TS 3577-11 30.00g HDDA 10.00 EEA 5.00 EAQ 0.23 DMEA 0.45 TiO ₂ 20.00	5 Passes 12.5 ft/min 300 W/in	100%	100%	100%	B	H	2B	11/19/80
P162 TS 3577-11 30.00g HDDA 10.00 EEA 5.00 EAQ 0.45 TEA 0.45 TiO ₂ 20.00	5 Passes 12.5 ft/min 300 W/in	100%	100%	100%	HB	5B	<6B	11/19/80
P163 TS 3577-11 30.00g HDDA 10.00 EEA 5.00 EAQ 0.90 Morpholine 0.45 TiO ₂ 20.00	6 Passes 12.5 ft/min 300 W/in	100%	100%	100%	4H	<6B	<6B	11/19/80
P164 TS 3577-11 30.00g HDDA 10.00 EEA 5.00 EAQ 1.35 EPDMAB 0.45 TiO ₂ 20.00	7 Passes 12.5 ft/min 300 W/in	100%	100%	98%	2H	H	HB	11/19/80
		100%	100%	98%	3H	2H	HB	11/21/80

TABLE 31— PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.50% 2-ETHYLANTHRAQUINONE (EAQ)
AND 1.00% AMINE CURED WITH D BULB (SHEET 2 of 3)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P165 TS3577 - 11 30.00g HDDA 10.00 EEA 5.00 EAQ 0.23 QSC 0.45 TiO ₂ 20.00	3 Passes 12.5 ft/min 300 W/in	100%	94%	0%	< 6B	< 6B	< 6B	12/2/80
P166 TS 3577 - 11 30.00g HDDA 10.00 EEA 5.00 EAQ 0.23 MDEA 0.45 TiO ₂ 20.00	6 Passes 12.5 ft/min 300 W/in	100%	100%	99%	F	HB	4B	11/19/80
P170 TS3577 - 11 30.00g HDDA 10.00 EEA 5.00 EAQ 0.23 UVEC 0.45 P-101 TiO ₂ 20.00	6 Passes 12.5 ft/min 300 W/in	100%	100%	100%	3H	2B	HB	11/24/80
					2H	2B	B	11/26/80

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TABLE 31 - PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.50% 2-ETHYLANTHRAQUINONE (EAQ) AND 1.00% AMINE CURED WITH D BULB (SHEET 3 of 3)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P171 TS 3577 - 11 30.00 g HDDA 10.00 EEA 5.00 EAQ 0.23 UVEC P-102 0.45 TiO ₂ 20.00	6 Passes 12.5 ft/min 300 W/in	100%	100%	100%	2H	4B	B	11/24/80
P172 TS 3577 - 11 30.00 g HDDA 10.00 EEA 5.00 EAQ 0.23 UVEC P-104 0.45 TiO ₂ 20.00	6 Passes 12.5 ft/min 300 W/in	100%	100%	100%	F	2B	HB	11/24/80

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sufonyl chloride decreased cure time. On the other hand, coatings containing Irgacure, DTBP and MDEA were slower curing and softer. Irgacure 651 is more effective than V10 in pigmented coatings (Table 32). DEAP (Table 33) was ineffective in curing TiO_2 -pigmented coatings.

The properties of films containing various combinations of photoinitiators are summarized in Tables 34 through 40. Pigmented coatings containing CGI 1184 (Table 34) showed no improvement in whiteness over coatings containing CTX. Coatings using combinations of photoinitiators which included benzil (Table 35) exhibited inconsistent adhesion, i.e., were particularly sensitive to film thickness. Coatings containing benzil, CTX and amine (Table 36) were similar to coatings containing benzil and morpholine. EAQ was tested in conjunction with the other photoinitiators shown in Table 37. A more rapid cure and good adhesion were achieved with the addition of Michler's Ketone and ethyl para-dimethylaminobenzoate (EPDMAB). Combinations of EAQ with other photoinitiators such as benzil might accelerate cure speed while retaining adhesion. Formulations containing EAQ appeared to most consistently produce satisfactory adhesion. As expected, an increase in length of exposure to UV radiation led to harder coatings (Table 37). Photoinitiator systems containing MEAQ (Table 38) and EAQ (Table 37) had consistently good adhesion, except in combination with MK or BP. The cure time of MEAQ and EAQ containing coatings was usually 5 to 7 passes at 12.5 ft. per minute and 300 watts per inch. Coatings containing Irgacure 651 and other photoinitiators (Table 39), cured at a moderate rate and were soft. Coatings containing various combinations of photoinitiators are listed in Table 40. Primers which were pigmented with combinations of iron oxide and zinc chromate (Table 57 on page 103) and iron oxide alone (Table 58 on page 105) were evaluated in formulations using several free radical mechanism photoinitiators.

These tests indicated that Irgacure was effectively screened by the pigments, a CTX/MDEA combination had poor adhesion, and an EAQ system was too viscous to handle.

TABLE 32

ADHESION TESTS OF PIGMENTED COATINGS CURED BY 300 WATTS/INCH UV RADIATION AT 6.25 FT./MIN.

Formulation	H Bulb				V10 Photoinitiator				Irgacure Photoinitiator			
	No. of Passes	ZnPhos	No Photoinitiator % Adhesion To FePhos	Steel	No. of Passes	ZnPhos	No Photoinitiator % Adhesion To FePhos	Steel	No. of Passes	ZnPhos	No Photoinitiator % Adhesion To FePhos	Steel
P68 TS3577-11 TiO ₂	3	100	100	100	4	67	97	0	10	100	100	11
P69 & P75 TS3577-11 DTBP TiO ₂	4	100	100	53	3	93	95	0	3	94	100	2
P70 & P71 TS3577-11 DTBP MDEA TiO ₂	7	100	100	52	3	100	100	1	3	74	100	8
P72 & P74 TS3577-11 HDDA EEA DTBP MDEA TiO ₂	11	100	100	100	6	100	100	13	3	100	100	73
P73 & P76 TS3577-11 HDDA EEA DTBP TiO ₂	10	100	100	100	9	100	100	65	5	100	100	99
P77 TS3577-11 HDDA EEA TiO ₂	-	-	-	-	11	100	100	100	6	100	100	100

TABLE 33-PROPERTIES OF PIGMENTED COATINGS CONTAINING 1.00% DIETHOXYACETOPHENONE(DEAP)
CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P173								
TS3577-11	30.00g	10 Passes						12/4/80
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 w/in						
DEAP	0.45	D Bulb						
Tioxide	20.00	*Sample would not cure in 10 passes						
P175								
TS3577-11	30.00g	1 Pass	56%	1%	0%	5B	<6B	12/4/80
HDDA	10.00	12.5 ft/min	43%	14%	0%	6B	6B	12/8/80
EEA	5.00	300 w/in						
DEAP	0.45	D Bulb						
MK	0.45							
Tioxide	20.00							
P176								
TS3577-11	30.00g	10 Passes	100%	100%	0%	2B	3B	12/4/80
HDDA	10.00	12.5 ft/min	100%	100%	24%	3B	2B	12/8/80
EEA	5.00	300 w/in						
DEAP	0.45	D Bulb						
EPDMAB	0.45							
Tioxide	20.00							
P177								
TS3577-11	30.00g	10 Passes	100%	100%	9%	5B	<6B	5B
HDDA	10.00	12.5 ft/min	100%	100%	77%	<6B	<6B	5B
EEA	5.00	300 w/in						
DEAP	0.45	D Bulb						
VIO	0.45							
Tioxide	20.00							

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TABLE 34-PROPERTIES OF PIGMENTED COATINGS CONTAINING CGI 1184 CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P202								
TS3577-11	30.00g	3 Passes						
HDDA	10.00	12.5 ft/min	59%	42%	14%	<6B	<6B	12/23/80
EEA	5.00	300 w/in						
CGI 1184	1.80	D Bulb	0%	0%	0%	<6B	<6B	12/29/80
PAQ	0.11							
Titanox 2010	20.00							
P203								
TS3577-11	30.00g	3 Passes	100%	99%	53%	<6B	<6B	12/19/80
HDDA	10.00	12.5 ft/min	99%	97%	81%	<6B	<6B	12/22/80
EEA	5.00	300 w/in						
CGI 1184	0.45	D Bulb						
Benzop	0.68							
MDEA	0.90							
Titanox 2010	20.00							

TABLE 35-PROPERTIES OF PIGMENTED COATINGS CONTAINING 1.00% BENZIL CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P183								
TS3577-11	30.00g	1 Pass						
HDDA	10.00	12.5 ft/min	52%	66%	0%	<6B	<6B	12/4/80
EEA	5.00	300 w/in	30%	45%	0%	6B	6B	12/8/80
Benzil	0.45	D Bulb						
MK	0.45							
Titanox	20.00							
2010								
P184								
TS3577-11	30.00g	2 Passes						
HDDA	10.00	12.5 ft/min	87%	16%	0%	HB	HB	12/4/80
EEA	5.00	300 w/in	4%	0%	0%	F	F	12/8/80
Benzil	0.45	D Bulb						
EPDMAB	0.45							
Titanox	20.00							
2010								
P185								
TS3577-11	30.00g	2 Passes						
HDDA	10.00	12.5 ft/min	100%	96%	0%	<6B	<6B	12/4/80
EEA	5.00	300 w/in	100%	100%	45%	5B	<6B	12/8/80
Benzil	0.45	D Bulb						
V10	0.45							
Titanox	20.00							
2010								
P208								
TS3577-11	30.00g	2 Passes						
HDDA	10.00	12.5 ft/min	95%	10%	5%	2H	HB	12/30/80
EEA	5.00	300 w/in	100%	0%	0%	2H	F	1/2/81
Benzil	0.23	D Bulb						
MDEA	0.23							
Titanox	20.00							
2010								

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TABLE 36 - PROPERTIES OF PIGMENTED COATINGS CONTAINING BENZIL 2-CHLOROTHIOXANTHONE (CTX), AND AMINE CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P155								
TS 3577 - 11	30.00 g	1 Pass	91%	96%	16%	6B	< 6B	< 6B
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 W/in	98%	100%	43%	< 6B	< 6B	HB
Benzil	0.45							
CTX	0.18							
EPDMAB	0.45							
TiO ₂	20.00							
P157								
TS 3577 - 11	30.00 g	1 Pass	97%	100%	58%	2B	4B	6B
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 W/in	100%	100%	79%	2B	2B	2B
Benzil	0.90							
CTX	0.09							
MDEA	0.90							
TiO ₂	20.00							
P158								
TS 3577 - 11	30.00 g	2 Passes	100%	100%	64%	H	4B	3B
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 W/in	100%	100%	29%	H	3B	3B
Benzil	1.35							
CTX	0.09							
MDEA	1.35							
TiO ₂	20.00							

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TABLE 37 - PROPERTIES OF PIGMENTED COATINGS CONTAINING 2-ETHYLANTHRAQUINONE (EAQ)
AND OTHER PHOTOMONITIATORS CURED WITH D BULB (Sheet 1 of 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test	
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P167 TS 3577 - 11 HDDA EEA EAQ CTX TiO ₂	30.00 g 10.00 5.00 0.23 0.09 20.00	6 Passes 12.5 ft/min 300 W/in	100% 100% 100%	100% 100% 100%	98% 100% 100%	3H 2H	H H	3B 2B	11/18/80 11/21/80
P168 TS 3577 - 11 HDDA EEA EAQ EPDMAB V30 TiO ₂	30.00 g 10.00 5.00 0.23 0.23 0.23 20.00	6 Passes 12.5 ft/min 300 W/in	100% 100% 100%	100% 100% 85%	97% 85%	< 6B 6B	HB 2H	4B 4B	11/24/80 11/26/80
P169 TS 3577 - 11 HDDA EEA EAQ MK EPDMAB TiO ₂	30.00 g 10.00 5.00 0.23 0.23 0.23 20.00	3 Passes 12.5 ft/min 300 W/in	97% 100% 100%	100% 100% 100%	100% 100% 100%	< 6B < 6B < 6B	< 6B < 6B < 6B	< 6B < 6B < 6B	11/18/80 11/21/80

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TABLE 37-PROPERTIES OF PIGMENTED COATINGS CONTAINING 2-ETHYLANTHRAQUINONE (EAQ) AND OTHER PHOTONIATORS CURED WITH D BULB (Sheet 2 of 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P197								
TS3577-11	30.00g	3 Passes						
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 w/in						
EAQ	0.45	D Bulb						
MK	0.23							
EPDMAB	0.23							
Titanox	20.00							
2010								
P217								
TS3577-11	30.00g	4 Passes						
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 w/in						
EAQ	0.45	D Bulb						
MK	0.23							
Titanox	20.00							
2010								
P219								
TS3577-11	30.00g	4 Passes						
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 w/in						
EAQ	0.23	D Bulb						
BP	0.45							
QSC	0.45							
Titanox	20.00							
2010								

TABLE 38-PROPERTIES OF PIGMENTED COATINGS CONTAINING 0.50% 2-METHYLANTHRAQUINONE (MeAQ)
CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P174								
TS3577-11 30.00g	6 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	100%	4H	H	F	12/4/80
HDDA 10.00								
EEA 5.00								
2 MeAQ 0.23								
Tioxide 2010 20.00								
P178								
TS3577-11 30.00g	3 Passes 12.5 ft/min 300 w/in D Bulb	75%	3%	54%	<6B	<6B	<6B	12/4/80
HDDA 10.00								
EEA 5.00								
2 MeAQ 0.23								
MK 0.45								
Tioxide 2010 20.00								
P179								
TS3577-11 30.00g	5 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	100%	4B	<6B	6B	12/4/80
HDDA 10.00								
EEA 5.00								
2 MeAQ 0.23								
EPDMAB 0.45								
Tioxide 2010 20.00								
P180								
TS3577-11 30.00g	5 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	95%	3B	HB	HB	12/4/80
HDDA 10.00								
EEA 5.00								
2 MeAQ 0.23								
VIO 0.23								
Tioxide 2010 20.00								

TABLE 39-PROPERTIES OF PIGMENTED COATINGS CONTAINING IRGACURE 651 (Irg 651)
CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P199								
TS3577-11	30.00g	3 Passes	91%	92%	3%	<6B	<6B	12/23/80
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 w/in						
Irg 651	1.80	D Bulb	99%	91%	45%	4B	<6B	12/29/80
PAQ	0.11							
Titanox	20.00							
2010								
P200								
TS3577-11	30.00g	2 Passes	98%	96%	92%	<6B	<6B	12/22/80
HDDA	10.00	12.5 ft/min						
EEA	5.00	300 w/in						
Irg 651	0.45	D Bulb	99%	100%	60%	<6B	<6B	12/29/80
Benzop.	0.68							
MDEA	0.90							
Titanox	20.00							
2010								

TABLE 40—PROPERTIES OF PIGMENTED COATINGS CONTAINING VARIOUS PHOTONINITIATORS CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates	
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P195									
TS3577-11	30.00g	1 Pass 12.5 ft/min 300 w/in D Bulb	46%	2%	0%	F	2B	4B	12/19/80
HDDA	10.00								
EEA	5.00								
Irg 651	0.90								
CTX	0.11								
MDEA	0.90								
Titanox	20.00								
2010									
P201									
TS3577-11	30.00g	1 Pass 12.5 ft/min 300 w/in D Bulb	7%	12%	0%	F	5B	<6B	12/19/80
HDDA	10.00								
EEA	5.00								
CGI 1184	0.90								
CTX	0.11								
MDEA	0.90								
Titanox	20.00								
2010									
P209									
TS3577-11	30.00g	4 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	100%	F	F	3B	12/30/80
HDDA	10.00								
EEA	5.00								
Benzil	0.23								
EAQ	0.23								
MDEA	0.23								
Titanox	20.00								
2010									
P218									
TS3577-11	30.00g	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	99%	98%	<6B	<6B	<6B	1/5/81
HDDA	10.00								
EEA	5.00								
EAQ	0.45								
MK	0.23								
CTX	0.45								
MDEA	0.23								
Titanox	20.00								
2010									

Newly developed UV sensitive cationic photoinitiators, the so-called "onium salts", aryldiazonium, diaryliodonium, and triarylsulfonium salts of complex metal halide anions such as hexafluoroarsenate, hexa-fluorophosphate, and hexafluoroantimonate, were used to polymerize epoxy resin. Cationic photoinitiators, FC-508 and FC-509, are available from 3M Company. Since the spectral sensitivity of cationic photoinitiators is relatively low^{23,28} (Figures 6,7 and 8), and the white pigment, rutile TiO₂, screened out the major absorption region of the photoinitiators (see Figures 1 and 3), a photosensitizer is necessary in the formulation to have an effective photoinduced reaction. A Fusion System Corp. non-mercury D bulb or V bulb was used to cure the epoxy paints because the bulbs have strong emission spectra at the edge of or outside the range of the rutile TiO₂ absorption spectra (see Figure 2, the emission spectra of fusion bulbs).

A summary of test results of paints, a formulation using FC-508 and FC-509 photoinitiators, is shown in Table 41. The cure rate of a coating photoinitiated with FC-508 is slower than with FC-509 at same concentration (see formulations CP190 and CP211). The cure rate is usually increased by increasing FC-508 concentration as in formulation CP211 and CP216II. However, coatings photoinitiated using FC-508 have a longer shelf life than those using FC-509. Formulation CP304 which contains FC-508 is not gelled after four months at room temperature, whereas CP305 containing FC-509 gelled after three months at room temperature.

The following photosensitizers were used:

FC-510 (3M Co.), 2-chlorothioxanthone, thioxanthone, benzanthrone, 1,3-diphenyl-2-propane, triphenyl acetophenone, fluorenone, biphenyl, flavone, 4-acetyl biphenyl, diacetyl, fluorene, 9,10-phenanthrene-quinone, benzophenone and benzil. 2-Chlorothioxanthone, thioxanthone and FC-510 had the same degree of effectiveness, whereas all the others were less effective.

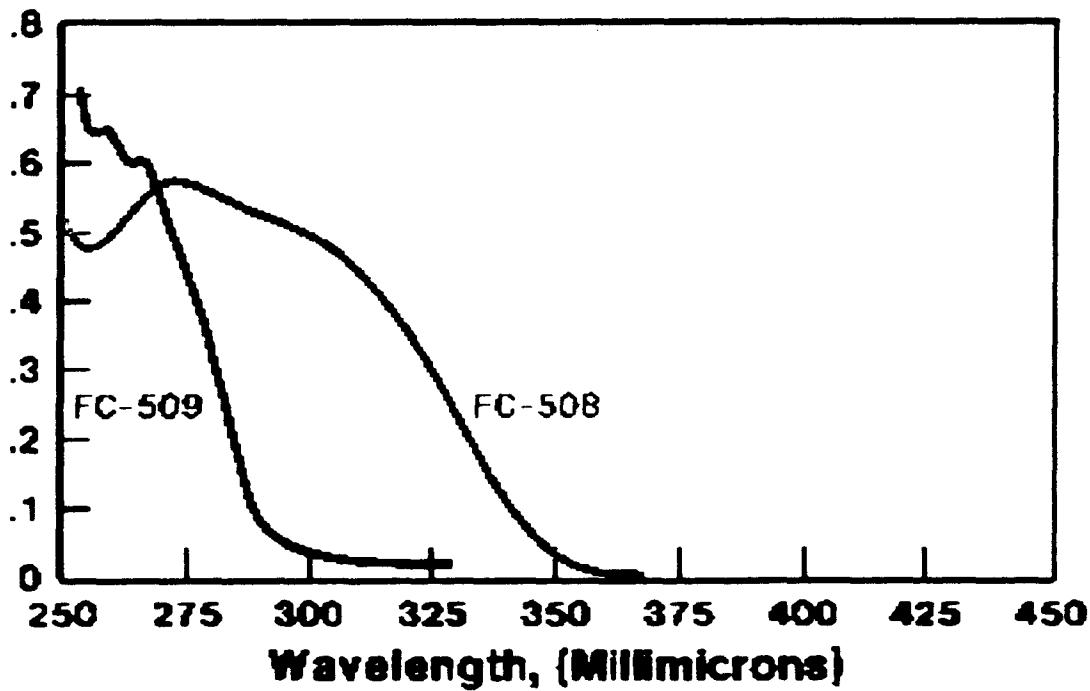
Figure 6

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ULTRAVIOLET ABSORPTION SPECTRA 3M Brand UV-Activated Epoxy Curatives

O.D. (Abs.)



99

Slide Masters, Inc. Pgh. Pa. (412) 366-8313

WR A

3M Brand UV Activated Epoxy Curatives

Optically Sensitized Absorption Spectra

FC-509: 2-Chlorothioxanthone

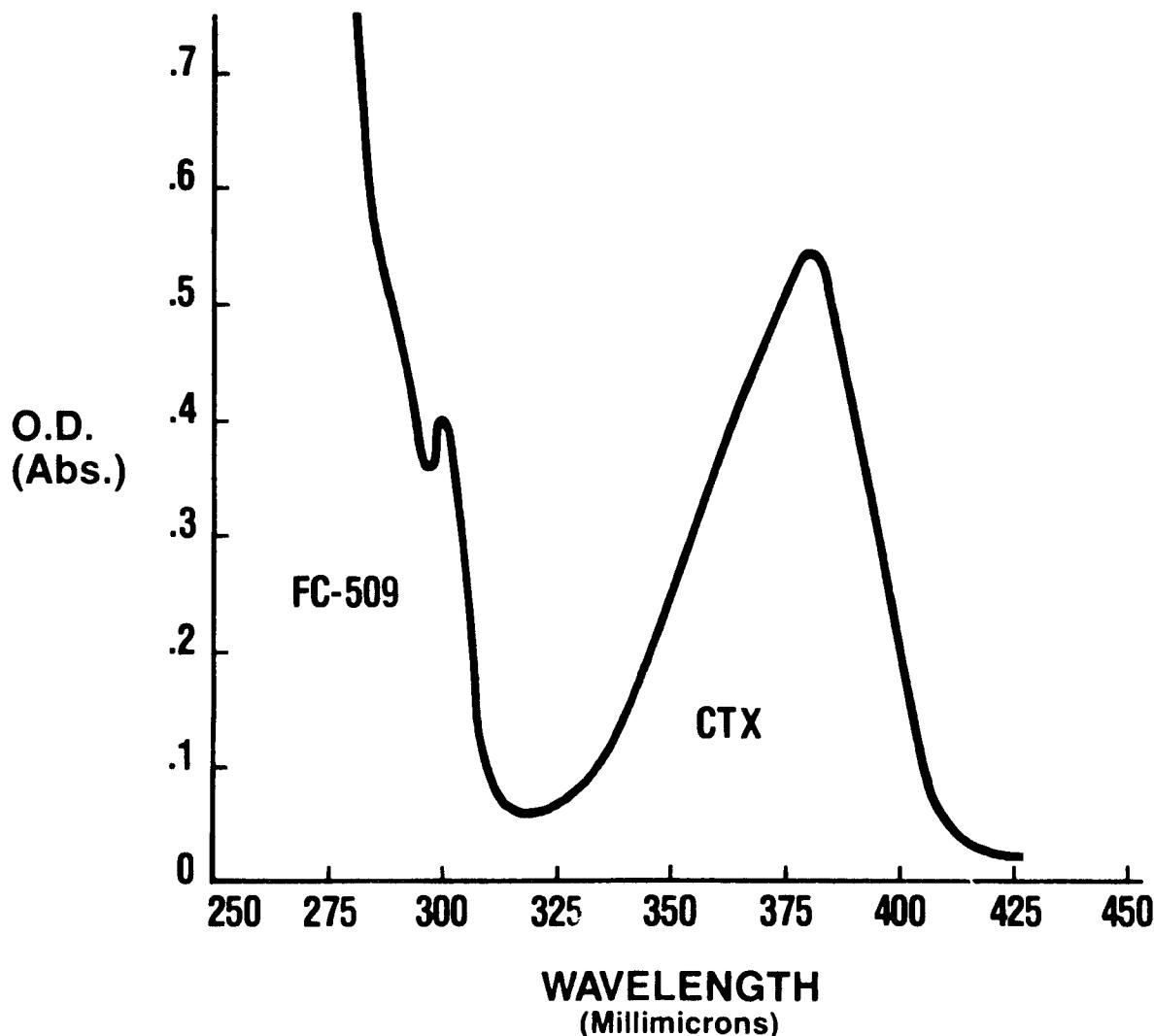


FIGURE 7

3M Brand UV Activated Epoxy Curatives Optically Sensitized Absorption Spectrum

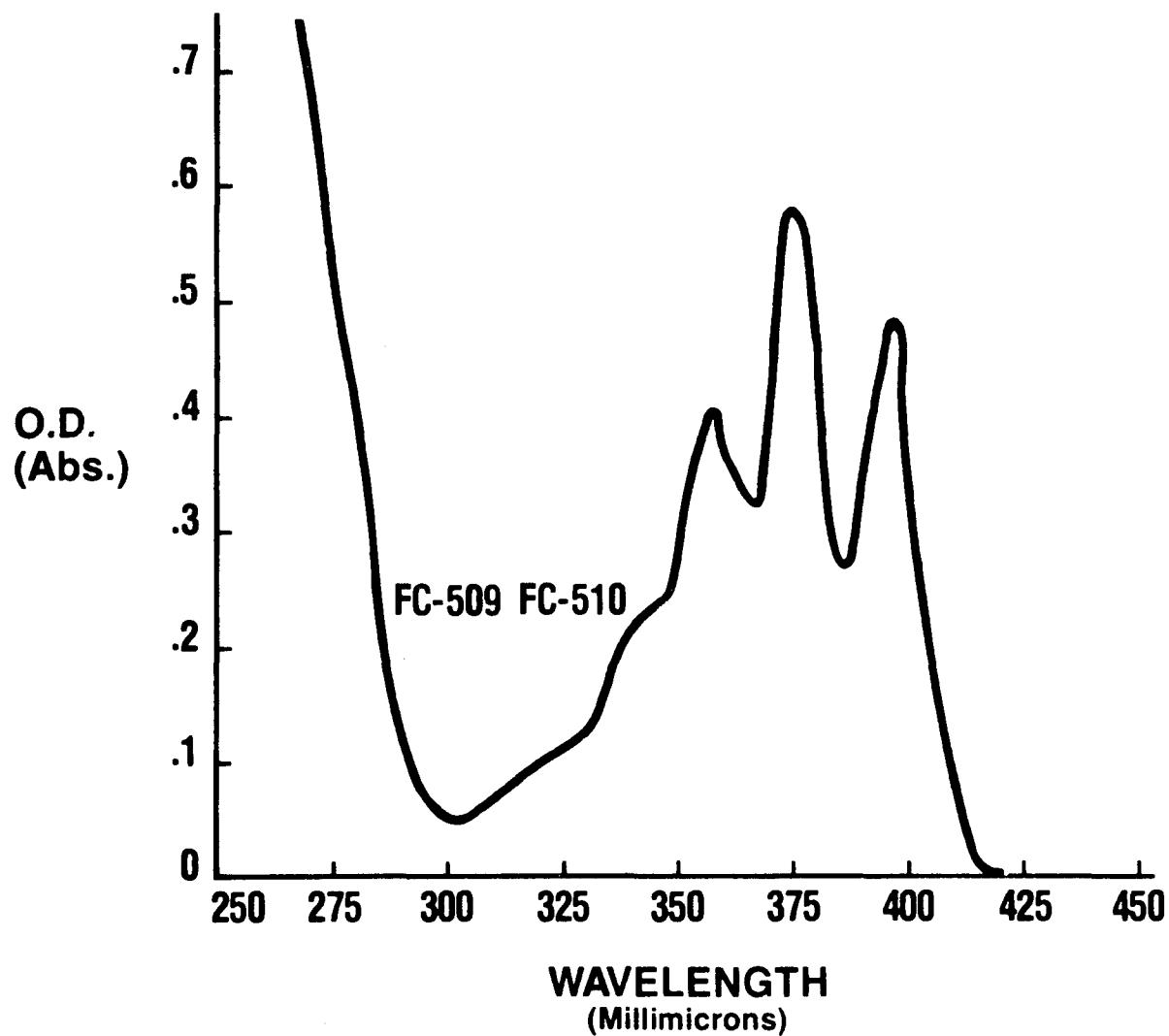


FIGURE 8

TABLE 41 - THE PHOTO INITIATOR STUDY OF UV CURABLE EPOXY PAINT

Formulation		Cure Time	Crosshatch Test			Pencil Hardness Test			Date
			Zn Phos.	Fe Phos.	Steel	Zn Phos.	Fe Phos.	Steel	
CP 304		D Bulb							
FC-508	4.00g	300 w/in							
FC-510	1.00	12.5 ft/min							
Titanox 2020	35.00	3 Passes For							
ERL4221	45.20	Fe-Phos.							
n-Butyl Alcohol	10.00	6 Passes For							
RD-1	7.00	Steel							
CP 305		D Bulb							
FC-509	3.30g	300 w/in							
FC-510	1.00	12.5 ft/min							
Titanox 2020	35.00	3 Passes							
ERL4221	43.20								
n-Butyl Alcohol	10.00								
RD-1	7.00								
CP 190		D Bulb							
ERL4221	25.00g	300 w/in	100%	100%	64%	F	HB	38	12-5-80
Epoxide 8	5.50	12.5 ft/min	100%	100%	0%	2H	2H	28	12-8-80
FC-509	1.50	3 Passes	-	-	11%	-	-	38	12-30-80
FC-510	0.50		-	-	100%	-	-	38	1-2-81
FC-171	0.25								
Titanox 2020	17.50	V Bulb	100%	100%	2%	HB	B	48	12-5-80
		300 w/in	100%	100%	0%	H	F	28	12-8-80
		12.5 ft/min							
		3 Passes	-	-	22%	-	-	38	12-30-80
					100%	-	-	HB	1-2-81
CP 211		D Bulb							
ERL4221	25.00g	300 w/in							
Epoxide 8	5.50	12.5 ft/min							
FC-508	1.50	3 Passes	-	-	100%	-	-	< 68	12-30-80
FC-510	0.50				100%			< 68	1-2-81
FC-171	0.25								
Titanox 2020	17.50	V Bulb							
		300 w/in							
		12.5 ft/min							
		3 Passes	-	-	100%	-	-	< 68	12-30-80
								< 68	1-2-81
CP 216II		D Bulb							
ERL4221	25.00g	300 w/in							
Epoxide 8	5.50	12.5 ft/min							
FC-508	2.00	3 Passes	100%	100%	100%	5B	6B	< 68	12-31-80
FC-510	0.50				100%	HB	3B	5B	1-2-81
FC-171	0.25								
Titanox 2020	17.50	V Bulb							
		300 w/in							
		12.5 ft/min							
		3 Passes	-	-	100%	-	-	3B	12-30-81
								3B	1-2-81

The cure rate is optimized at a 3 to 1 ratio of FC-509 to photosensitizer and a 4 to 1 ratio of FC-508 to photosensitizer. 2-Chlorothioxanthone and thioxanthone usually gave an off-white, yellowing coating, which may, due to the visible absorption of the photosensitizer. FC-510 also gave a yellowing coating right after the UV curing. However, the yellowing diminished through time. Figures 6 and 7 show the absorption spectra of FC-509 sensitized by 2-chlorothioxanthone and FC-510, respectively. The addition of photosensitizers can extend the absorption spectra of photoinitiators to the outside range of the TiO_2 pigment's absorption spectra. The photosensitizer absorbs the light first, transfers the absorbed energy to the photoinitiator, then photoinitiation and polymerization follows.

5.1.3 Oligomers and Monomers

To develop a suitable paint vehicle, it was first necessary to select monomer diluents and base resins that are sensitive to the UV curing process. The reactive monomeric diluents tested included multi-functional acrylates, monofunctional acrylates and non-acrylic unsaturated esters,^{22,23} (Tables 42, 43, 44, 45, 46, and 47) which showed high reactivity when photoinitiated using isopropyl benzoin ether (Stauffer Chemical Co., Vicure V-10) are listed below:

- pentaerythritol tetracrylate (PETA)
- pentaerythritol triacrylate (PETA)
- trimethylolpropane triacrylate (TMPTA)
- tetraethyleneglycol diacrylate (TEGDA)
- diethyleneglycol diacrylate (DEGDA)
- 1,6-hexanediol diacrylate (HDDA)
- triethylene glycol dimethacrylate (TEGDMA)
- 1,3-butylene dimethacrylate (BDMA)

Highly reactive monomers such as 1,6-hexanediol diacrylate (HDDA), 2-ethoxyethyl acrylate (EEA), tripropyleneglycol diacrylate (TPGDA), triethyleneglycol diacrylate (TEGDA) were then investigated to control viscosity and flow of the paint vehicle.

TABLE 42
PHOTOSENSITIVITY OF MULTIFUNCTIONAL MONOMERS

Monomer	Photoinitiator* Concentration, wt. %	Comment
Pentaerythritol Tetra-acrylate	0.02	5 sec. slight gel; 7.5 sec. through cure.
Pentaerythritol Triacrylate	0.02	5 sec. thin skin; 7.5 sec. 90% cure.
Trimethylolpropane Triacrylate	0.02	12.5 sec. 90% cure.
Neopentyl Glycol Diacrylate	0.02	25 sec. no reaction; carcinogenic.
Hexanediol Diacrylate	0.02	17.5 sec. slight gel; 22.5 sec. 50% cure.
Tetraethylene Glycol Diacrylate	0.02	5 sec. thin skin; 7.5 sec. 90% cure, straw color.
Diethylene Glycol Diacrylate	0.02	10 sec. slight gel; 20 sec. through cure, yellow
Trimethylol Propane Trimethacrylate	0.02	25 sec. no reaction, yellow.
Neopentyl Glycol Dimethacrylate	0.02	25 sec. no reaction, yellow.
Triethylene Glycol Dimethacrylate	0.02	20 sec. slight gel, straw.
1,3-Butylene Dimethacrylate	0.02	25 sec. no reaction, yellow.
Ethoxylated Bis-Phenol A Dimethacrylate	0.02	2.5 sec. slight gel, brown.

CONTINUED

TABLE 42 (Cont.)

Monomer	Photoinitiator* Concentration, wt. %	Comment
Pentaerythritol Tetra-acrylate	0.5	2.5 sec. thick skin.
Pentaerythritol Triacrylate	0.5	2.5 sec. thick skin.
Trimethylol Propane Trimethacrylate	0.5	2.5 sec. thick skin.
Neopentyl Glycol Diacrylate	0.5	2.5 sec. thick skin.
Hexanediol Diacrylate	0.5	2.5 sec. 90% cure.
Tetraethylene Glycol Diacrylate	0.5	2.5 sec. 90% cure.
Diethylene Glycol Diacrylate	0.5	2.5 sec. thick skin.
Trimethylol Propane Trimethacrylate	0.5	2.5 sec. thick skin.
Neopentyl Glycol Dimethacrylate	0.5	12.5 sec. thin skin.
Triethylene Glycol Dimethacrylate	0.5	7.5 sec. through cure.
1,3-Butylene Dimethacrylate	0.5	7.5 sec. through cure.
Ethoxylated Bis-Phenol A Dimethacrylate	0.5	2.5 sec. thick skin.

*Photoinitiator was V10.

TABLE 43
PHOTOSENSITIVITY* OF ACRYLIC MONOMERS

Monomer	Results
2-Hydroxyethyl acrylate	2.5 sec. soft gel.
2-Hydroxyethyl methacrylate	7.5 sec. increase in viscosity.
Hydroxypropyl methacrylate	10 sec. increase in viscosity.
Ethyl acrylate	22.5 sec. no reaction.
Butyl Acrylate	22.5 sec. no reaction.
n-hexyl acrylate	20 sec. increase in viscosity.
2-Ethyl hexylacrylate	7.5 sec. increase in viscosity.
Crotyl acrylate	25 sec. gel.
Furfuryl acrylate	25 sec. no reaction.
Di-cyclopentadiene acrylate	2.5 sec. skin.
Isobornyl acrylate	7.5 sec. increase in viscosity.
Methoxyethyl acrylate	10 sec. gel.
2-Ethoxyethyl acrylate	10 sec. gel.
Butoxyethyl acrylate	10 sec. gel.
Phenoxyethyl acrylate	17.5 sec. slight gel.
Methyl methacrylate	25 sec. no reaction.
Ethyl methacrylate	25 sec. no reaction.
N-hexyl methacrylate	25 sec. no reaction.
Glycidyl methacrylate	25 sec. no reaction.

*0.5 wt. % V10 in monomer.

TABLE 44
PHOTOSENSITIVITY OF NONACRYLIC ESTER MONOMERS

Monomer	Result			
Vinyl Acetate	25 second no reaction.			
N-Vinyl Pyrrolidone	"	"	"	"
Vinyl Toluene	"	"	"	"
Diallyl Phthalate	"	"	"	"
Styrene	"	"	"	"
Divinyl Benzene	"	"	"	"
1,5-Cyclo-Octadiene	"	"	"	"
Acrylic Acid	2.5 sec. gel, polymer precipitates.			
Methacrylic Acid	2.5 sec. gel, polymer precipitates.			

Photoinitiator was V10, 0.5 wt. %.

TABLE 45

REACTIVITY OF MULTIFUNCTIONAL MONOMERS WITH ISOBUTYL BENZOIN ETHER,
N-METHYLDIETHANOLAMINE, AND DI-T-BUTYL PERBENZOATE

Monomer	Comment
Pentaerythritol Tetracrylate	2.5 sec. 50% cure.
Pentaerythritol Triacrylate	7.5 sec. through cure.
Trimethylol Propane Triacrylate	2.5 sec. 90% cure.
Neopentyl Glycol Diacrylate	20 sec. slight gel.
Hexanediol Diacrylate	7.5 sec. through cure.
Tetraethylene Glycol Diacrylate	20 sec. through cure.
Diethylene Glycol Diacrylate	7.5 sec. through cure.
Trimethylol Propane Trimethacrylate	22.5 sec. through cure.
Neopentyl Glycol Dimethacrylate	25 sec. no reaction.
Triethylene Glycol Dimethacrylate	17.5 sec. through cure.
1,3-Butylene Dimethacrylate	25 sec. no reaction.
Ethoxylated Bis-Phenol A Dimethacrylate	2.5 sec. thick skin.

Activator Concentration for the above is: V10 0.02 wt. %,
MDEA 0.1 wt. %, DTBP 0.1 wt. %.

Pentaerythritol Tetracrylate	2.5 sec. through cure.
Pentaerythritol Triacrylate	2.5 sec. through cure.
Trimethylol Propane Triacrylate	2.5 sec. through cure.
Neopentyl Glycol Diacrylate	2.5 sec. through cure.
Hexanediol Diacrylate	2.5 sec. through cure.
Tetraethylene Glycol Diacrylate	2.5 sec. through cure.
Diethylene Glycol Diacrylate	12.5 sec. through cure.
Trimethylol Propane Trimethacrylate	7.5 sec. 90% cure.
Neopentyl Glycol Dimethacrylate	17.5 sec. through cure.
Triethylene Glycol Dimethacrylate	10 sec. through cure.
1,3-Butylene Dimethacrylate	--
Ethoxylated Bis-Phenol A Dimethacrylate	2.5 sec. thick skin.

Activator Concentration for the above is: V10 0.5 wt. %,
MDEA 0.1 wt. %, DTBP 0.1 wt. %.

V10, isobutyl benzoin ether
MDEA, methyldiethanolamine
DTBP, di-t-butyl perbenzoate.

TABLE 46

REACTIVITY OF ACRYLIC MONOMERS PHOTONINITIATED WITH ISOBUTYL BENZOIN ETHER,
N-METHYLDIETHANOLAMINE, AND DI-T-BUTYL PERBENZOATE

Monomer*	Result
2-Hydroxyethyl acrylate	2.5 sec. gel.
2-hydroxyethyl methacrylate	15 sec. increase in viscosity.
Hydroxypropyl methacrylate	25 sec. no reaction.
Ethyl Acrylate	25 sec. no reaction.
Butyl Acrylate	7.5 sec. increase in viscosity.
N-hexyl acrylate	20 sec. increase in viscosity.
2-ethyl hexacrylate	7.5 sec. increase in viscosity.
Crotyl Acrylate	25 sec. gel.
Furfuryl Acrylate	25 sec. no reaction.
Di-cyclopentadiene acrylate	5 sec. gel.
Isobornylacrylate	7.5 sec. gel.
Methoxyethyl acrylate	7.5 sec. gel.
2-ethoxyethyl acrylate	7.5 sec. gel.
Butoxyethyl acrylate	2.5 sec. no reaction
Phenoxyethyl acrylate	25 sec. no reaction.
Methyl methacrylate	17.5 sec. slight gel.
Ethyl methacrylate	25 sec. no reaction.
N-hexyl methacrylate	25 sec. no reaction.
Glycidyl methacrylate	25 sec. no reaction.

The photoinitiator system consisted of 0.5 wt. % V10, 0.1 wt. % DTBP, 0.1 wt. % MDEA.

TABLE 47

REACTIVITY OF NONACRYLIC ESTER MONOMERS WITH ISOBUTYL BENZOIN ETHER,
AMINE, DI-T-BUTYL PERBENZOATE, N-METHYL DIETHANOL

Monomer	Result				
Vinyl Acetate	25 second no reaction, yellow				
N-Vinyl Pyrrolidone	"	"	"	"	"
Vinyl Toluene	"	"	"	"	"
Diallyl Phthalate	"	"	"	"	"
Styrene	"	"	"	"	"
Divinylbenzene	"	"	"	"	"
1,5-Cyclo-Octadiene	"	"	"	"	"
Acrylic Acid	2.5 second cure.				
Methacrylic Acid	7.5 second cure.				

Photoinitiator and accelerator concentrations are:
V10 0.5 wt. %, DTBP 0.1 wt. %, MDEA 0.1 wt. %.

The viscosity and cure times of several systems which combined various resins and reactive diluents, were determined and the properties of their films were evaluated by conical mandrel bend, impact, abrasion and hardness tests (Tables 48, 49 and 50). Because of its high reactivity in these systems, HDDA was chosen as a diluent to reduce viscosity when added to base resins. The simple acrylates, methoxyethyl acrylate (MEA), 2-ethoxyethyl acrylate (EEA), butoxyethyl acrylate (BEA) showed only a moderate degree of reactivity when photoinitiated using V-10. However, when EEA was added to the base resin and photoinitiated using V-10, the resultant films had increased flexibility. On the other hand, the non-acrylic ester monomers were generally unreactive when photoinitiated with V-10, although acrylic acid and methacrylic acid formed polymeric gels which precipitated. Clear and pigmented coatings compounded with UV curing resins from Hughson Chemical Co. which were irradiated with the Fusion Systems Corp. H bulb and H and D bulbs in tandem, failed to give good film properties (Tables 51, 52, 53 and 54). Coatings compounded with UV curing resins from Armstrong Cork Co., which were modified with Reichold Chemical Co. Alpolit (unsaturated polyester resin) gave good film properties when irradiated with the combined H and D bulbs. Eleven UV curing resins from Westinghouse were irradiated using the H bulb, D bulb and H and D bulbs in tandem. Although unpigmented resins gave hard and adherent films, none of these materials cured well after pigmentation. These resins which were supplied photoinitiated with Irgacure 651, were evaluated using additional photoinitiators specifically tailored for pigmentation.

In a system photoinitiated using V-10 which was accelerated with N-methyldiethanolamine (MDEA) and di-tertiarybutyl perbenzoate (DTBP), the most promising resin for free-radical curing was the Hughson Chemical Co. acrylated urethane TS3577-11, diluted with HDDA and EEA. The effects of the addition of various monomeric reactive diluents on this base resin, which was photoinitiated with EAQ or CTX/MDEA or IRG are shown in Tables 55, 56, 57 and 58. The different monomers appear

TABLE 48
EVALUATION OF OLIGOMERS AS RESIN BINDERS

Test System Number	Resin* Composition	Viscosity, cp	Comments
RP1	Alpolit V81 50 g, HDDA 30 g	2,700	Cake cures in 5 sec. Panel cures in 2.5 sec. 1.25" mandrel cracks film.
RP2	Actomer X80 50 g, HDDA 20 g	1,290	Cake cures in 6 sec. Panel cures in 6 sec. 1.25" mandrel cracks film.
RP3	RD2970-1 5 g, HDDA 5 g	620	Cake cures in 5 sec. Panel cures in 2.5 sec. No cracks on conical mandrel.
RP4	RD2970-5 50 g, HDDA 5 g	470	Cake cures in 5 sec. Panel cures in 5 sec. 0.5" mandrel cracks film.
RP5	RD3073-35 50 g, HDDA 5 g	2,270	Cake cures in 5 sec. Panel cures in 2.5 sec. No cracks on conical mandrel
RP13	Aronix 50 g, HDDA 5 g	1,760	Cake cures in 2.5 sec. Panel cures in 10 sec. 1.25" mandrel cracks film.

*Photoinitiator and accelerator concentration in each resin was 1% V10, 1% DTBP, 1% MDEA.

NOMENCLATURE FOR TABLE 48

REACTIVE OLIGOMERS EXAMINED AS PAINT BINDERS

Material	Resin Type	Supplier
Alpolit V81	polyester	Hoechst
Actomer X80	acrylated fatty acid	Union Carbide
RD2970-1	acrylated urethane	Hughson
RD2970-5	acrylated urethane	Hughson
RD3073-35	acrylated urethane	Hughson
Celrad 3700	acrylated epoxy	Celanese
Celrad 3701	acrylated epoxy	Celanese
RR0383	acrylated epoxy	Celanese
RR27482	acrylated epoxy	Celanese
XD9002	acrylated epoxy	Dow
301	acrylated epoxy	Shell
303	acrylated epoxy	Shell
Aronix	not identified	Mitsui

TABLE 49

EVALUATION OF OLIGOMERS AS PAINT BINDERS

Test System Number, Material	Viscosity cp	Cure Time Secs.	Conical Mandrel	Impact Test, inch-pounds		Taber Abrasion, g/1000 rev.		
				Direct	Reverse			
R1 Alpolit 81 HDDA	50.0 g. 30.0 g.	1,290	4.5	Failed at 1"	3	< 1	0.012	
R2 Actomer X-80 HDDA	50.0 g. 20.0 g.	970	7.5	Failed at 1"	30	15	0.016	
R3 RD2970-1 HDDA	50.0 g. 50.0 g.	627	4.5	Failed at 0.625"	20	2	0.005	
R4 RD2970-5 HDDA	50.0 g. 5.0 g.	320	4.5	Failed at 1"	5	3	0.006	
T8	R5 RD3073-35 HDDA	50.0 g. 50.0 g.	2700	6	No failure	100	20	0.007
	R6 Celrad 3700 HDDA	50.0 g. 50.0 g.	435	3	Failed at 1"	0	0	0.003
	R7 Celrad 3701 HDDA	50.0 g. 50.0 g.	140	3	Failed at 1"	20	2	0.011
	R8 RR0383 HDDA	50.0 g. 30.0 g.	275	3	Failed at 1"	30	3	0.001
	R9 RR27482 HDDA	50.0 g. 50.0 g.	1,290	6	Failed at 1"	4	1	0.002
	R10 XD9002 HDDA	50.0 g. 50.0 g.	125	9	Failed at 1"	5	0	0.001
	R11 Shell 301 HDDA	50.0 g. 50.0 g.	125	4.5	Failed at 1"	3	< 1	0.018
	R12 Shell 303 HDDA	50.0 g. 50.0 g.	140	6	Failed at 1"	5	1	0.019
	R13 Aronix HDDA	50.0 g. 5.0 g.	1,760	9	Failed at 1"	5	< 1	0.006

TABLE 49 (Cont.)

Test	System Number, Material	Viscosity cp	Cure Time Secs.	Conical Mandrel	Impact Test, inch-pounds		Taber Atrasion, g/1000 rev.
					Direct	Reverse	
R14	TS3577-11 HDDA	80.0 g. 20.0 g.	140	3	Failed at 1"	< 1	< 1
R15	TS3577-12 HDDA	80.0 g. 20.0 g.	500	3	Failed at 0.125"	10	10
R16	10,037 HDDA	70.0 g. 30.0 g.	550	9	Failed at 1"	1	1
R17	2000X164 HDDA	70.0 g. 30.0 g.	1,290	10.5	Failed at 0.25"	70	20
R18	2216X2 HDDA	50.0 g. 50.0 g.	500	12	Failed at 0.875"	15	3
R19	G1000-AC HDDA	30.0 g. 15.0 g.	470	4.5	Fails at 0.75"	40	10
R20	1300X17-AC HDDA	30.0 g. 25.0 g.	2,700	6	Fails at 0.75"	15	6

82

Initiator system consisted of V10 1%, DTBP 1% and MDEA 1%.

TABLE 49 (Cont.)

EVALUATION OF OLIGOMERS AS PAINT BINDERS

Test System Number, Material	Viscosity cp	Conical Mandrel		Impact Tests		Abrasion, Cure	
		Steel Panels	Zn Phosphate Panels	inch-pounds	g/1000	Time, rev.	Secs.
R21 TS3577-12	80.0 g.						
HDDA	20.0 g.					< 1	
TMPTA	5.0 g.						
R22 2% V10							
TS3577-12	80.0 g.					< 1	
HDDA	20.0 g.						
TMPTA	5.0 g.						
R24 1300X17AC	30.0 g.	1,760	Fails at 1"		40	17	0.035 10.5
HDDA	25.0 g.						
TMPTA	5.0 g.						

C8

Initiator consisted of V10 1%, DTBP 1% and MDEA 1% except for R22 which contained V10 2%.

TABLE 49 (Cont.)

Test System Number, Material		Viscosity cp	EVALUATION OF OLIGOMERS AS PAINT BINDERS										Taber Abrasion, g/1000 rev.	Cure Time, Secs.		
			Conical Mandrel		Impact Tests, inch-pounds				Steel Panels	Reverse	Zn Phosphate Panels	Reverse				
			Steel Panels	Zn Phosphate Panels	Steel Panels	Reverse	Zn Phosphate Panels	Reverse								
R25	Alpolit	50.0 g.														
	HDDA	30.0 g.							10		10					
	EEA	10.0 g.														
R26	Actomer X-80	50.0 g.														
	HDDA	20.0 g.	820	Failed at 0.875"	Failed at 0.875"				30	3	50	10	0.016	4.5		
	EEA	10.0 g.														
R27	RD3073-35	50.0 g.	65	Failed at 0.875"	No Failure				40	1	80	20	0.001	15		
	HDDA	50.0 g.														
	EEA	10.0 g.														
R28	RD3073-35	50.0 g.							40		60					
	HDDA	50.0 g.														
	EEA	25.0 g.														
R29	TS3577-11	80.0 g.	85	No Failure	No Failure				10	10	130	40	0.008	3		
	HDDA	20.0 g.														
	EEA	10.0 g.														
R30	TS3577-12	80.0 g.							1							
	HDDA	20.0 g.														
	EEA	5.0 g.														
R31	TS3577-12	80.0 g.	225	No Failure	No Failure				40	30	110	30	0.007	3		
	HDDA	20.0 g.														
	EEA	10.0 g.														
R32	C1000AC	30.0 g.												Would not cure		
	HDDA	15.0 g.														
	EEA	10.0 g.														

Continued

TABLE 49 (Cont.)

Test System Number, Material	Viscosity cp	Impact Tests, inch-pounds								Taber Abrasion, g/1000 rev.	Cure Time, Secs.
		Conical Mandrel		Zn Phosphate Panels		Steel Panels		Zn Phosphate Panels			
Panels		Panels		Direct	Reverse	Direct	Reverse				
R33 1300X17AC	30.0 g.	470	No Failure	No Failure	80	50	100	50	0.025	10.5	
HDDA	25.0 g.										(tacky)
EEA	12.5 g.										
R34 1300X17AC	30.0 g.	370	Failed at 0.5"	Failed at 0.25"	100	20	100	30	0.030	10.5	
HDDA	25.0 g.										
TMPTA	5.0 g.										
EEA	12.5 g.										
A35 RR0883	50.0 g.	85	Failed at 1"	Failed at 0.5"	30	3	50	5	0.005	3	
HDDA	30.0 g.										
EEA	10.0 g.										
C8 R36 2000X164	70.0 g.	1000	No	No	140	40	160	50	0.003	9	
	HDDA	30.0 g.									
	EEA	10.0 g.									

Initiator consisted of V10 1%, DTBP 1%, and MDEA 1%.

TABLE 50

PENCIL HARDNESS TEST

<u>Test System Number</u>	<u>Resin</u>	<u>Hardness</u>
R1	Alpolit V81/HDDA	6H
R2	Actomer X80/HDDA	6H
R3	RD2970-1/HDDA	7H
R4	RD2970-5/HDDA	6H
R5	RD3073-35/HDDA	--
R6	Celrad 3700/HDDA	5H
R7	Celrad 3701/HDDA	6H
R8	RR0383/HDDA	--
R9	RR27482/HDDA	6H
R10	XD9002/HDDA	1H
R11	Shell 301/HDDA	6H
R12	Shell 303/HDDA	5H
R13	Aronix/HDDA	6H
R14	TS3577-11/HDDA	--
R15	TS3577-12/HDDA	5H
R16	FS10, 037/HDDA	5H
R17	2000X164/HDDA	4H
R18	2216XZ/HDDA	4B
R19	G1000-AC/HDDA	3B
R20	1300X17-AC/HDDA	3B
R21	TS3577-12/HBBA/TMPTA	4H
R22	TS3577-12/HDDA/TMPTA	5H
R23	LX10-AC/HDDA	--
R24	1300X17AC/HDDA/TMPTA	3B
R25	Alpolit/HDDA/EEA	7H
R26	Actomer X80/HDDA/EEA	7H
R27	RD3073-35/HDDA/EEA	6H
R28	RD3073-35/HDDA/EEA	6H
R29	TS3577-11/HDDA/EEA	6H
R30	TS3577-12/HDDA/EEA	6H
R31	TS3577-12/HDDA/EEA	7H
R32	G1000AC/HDDA/EEA	not cured
R33	1300X17AC/HDDA/EEA	6H
R34	1300X17AC/HDDA/TMPTA/EEA	H
R35	RR0883/HDDA/EEA	6H
R36	2000X164/HDDA/EEA	6B

TABLE 51 - PROPERTIES OF CLEAR COATINGS CURED WITH FUSION SYSTEMS H BULB (SHEET 1 OF 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Tests
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P44								
Alpolit	50.00 g							
HDDA	30.00	1 Pass	32%	0%	HB	F	HB	7/3/80
EEA	10.00	6.25 ft/min						
DTBP	0.90	30 w/in	100%	52%	2H	F	HB	7/7/80
VIO	0.90							
MDEA	0.90							
P67A								
TS3577-11		20 Passes						
		40 ft/min						
		200 w/in						
P68A								
TS3577-11	30.00 g	5 Passes						
VIO	0.30	40 ft/min						
		200 w/in						
P68C								
TS3577-11	30.00 g	4 Passes						
Irg.	0.30	40 ft/min						
		200 w/in						
P69A								
TS3577-11	30.00 g	5 Passes						
DTBP	0.30	40 ft/min						
VIO	0.30	200 w/in						
P69C								
TS3577-11	30.00 g	3 Passes						
DTBP	0.30	40 ft/min						
Irg.	0.30	200 w/in						
P70A								
TS3577-11	30.00 g	6 Passes						
DTBP	0.30	40 ft/min						
MDEA	0.30	200 w/in						
P71A								
TS3577-11	30.00 g	4 Passes						
DTBP	0.30	40 ft/min						
MDEA	0.30	200 w/in						
VIO	0.30							
P71C								
TS3577-11	40.00 g	3 Passes						
DTBP	0.40	40 ft/min						
MDEA	0.40	200 w/in						
Irg.	0.40							

Fusion System Corp. D Bulb
 Lamp Width (Irradiation Zone) 8.25 in.
 Effective Radiation Time is 1.0 sec/pass.

TABLE 51 - (CON'T) - CLEAR COATINGS - USING H BULB (SHEET 2 OF 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Tests
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P72A TS3577-11 HDDA 40.00 g EEA 10.00 DTBP 5.00 MDEA 0.55 VIO 0.55	6 Passes 40 ft/min 200 w/in	0%	0%	0%	-	-	-	5/16/80
P72C TS3577-11 HDDA 40.00 g EEA 10.00 DTBP 5.00 MDEA 0.55 Irg. 0.55	4 Passes 40 ft/min 200 w/in	0%	0%	0%	-	-	-	5/16/80
P73A TS3577-11 HDDA 40.00 g EEA 10.00 DTBP 5.00 VIO 0.55	9 Passes 40 ft/min 200 w/in	100%	100%	0%	-	-	-	5/20/80
P73C TS3577-11 HDDA 40.00 g EEA 10.00 DTBP 5.00 Irg. 0.55	8 Passes 40 ft/min 200 w/in	0%	0%	0%	-	-	-	5/20/80
P74A TS3577-11 HDDA 40.00 g EEA 10.00 DTBP 5.00 MDEA 0.55	5 Passes 40 ft/min 200 w/in	0%	0%	0%	-	-	-	5/16/80
P75A TS3577-11 DTBP 30.00 g 0.30	6 Passes 40 ft/min 200 w/in	83%	91%	0%	-	-	-	5/16/80
P76A TS3577-11 HDDA 40.00 g EEA 10.00 DTBP 5.00	11 Passes 40 ft/min 200 w/in	0%	0%	0%	-	-	-	5/19/80

TABLE 51 - (CON'T) CLEAR COATINGS - USING H BULB (SHEET 3 OF 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Tests
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P77A TS3577-11 40.00 g HDDA 10.00 EEA 5.00 VIO 0.55	11 Passes 40 ft/min 200 w/in	17%	0%	0%	-	-	-	5/20/80
P77C TS3577-11 40.00 g HDDA 10.00 EEA 5.00 Irg. 0.55	8 Passes 40 ft/min 200 w/in	2%	2%	0%	-	-	-	5/20/80
P94 TS3401-IF TS3577-11 HDDA EEA DTBP Irg. MDEA	P72C 1 Pass 6.25 ft/min 300 w/in	14%	0%	1%	F	F	3B	7/3/80
P95 (Same as P87) TS3401-IF	1 Pass ft/min 300 w/in	100% 100%	100% 100%	98% 72%	HB F	F F	HB F	6/30/80 7/2/80
P97 P72C 4.00 g P44 4.00	1 Pass 6.25 ft/min 300 w/in	38% 63%	13% 0%	0% 0%	3H 2H	2H F	F F	7/3/80 7/7/80
P98 P72C 6.00 g P44 3.00	1 Pass 6.25 ft/min 300 w/in	10% 90%	0% 0%	0% 1 1%	2H 2H	H HB	HB HB	7/3/80 7/7/80
P99 P72C 6.00 g P44 3.00	1 Pass 6.25 ft/min 300 w/in	100%	9%	0%	7H	2H	HB	7/7/80
P100 P72C 16.0 g P44 2.00	1 Pass 6.25 ft/min 300 w/in	15% 75%	12% 33%	0% 0%	3H 2H	3H 2H	HB 2B	7/7/80 7/9/80

Dwg. 4249835

TABLE 51-(CON'T) USING H BULB -300 w/in 6.25 ft/min (SHEET 4 of 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Tests
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P101 Alpolit 50.00 g HDDA 30.00 EEA 10.00 DTBP 0.90 Irg. 0.90 MDEA 0.90	1 Pass 6.25 ft/min 300 w/in	100%	100%	98%	4H	3H	F	7/7/80
P102 P72C 4.00 g P101 4.00	1 Pass 12.5 ft/min 300 w/in	99%	1%	0%	3H	H	B	7/8/80
P103 P72C 6.00 g P101 3.00	1 Pass 12.5 ft/min 300 w/in	8%	3%	0%	2H	4H	3B	7/8/80
P104 P72C 16.00 g P101 4.00	1 Pass 12.5 ft/min 300 w/in	14%	30%	0%	H	3H	3B	7/8/80
P105 P72C 16.00 g P101 2.00	12.5 ft/min 1 Pass 300 w/in	17%	7%	0%	3H	3H	3H	7/8/80
		15%	1%	0%	H	3H	3B	7/10/80

TABLE 52-PROPERTIES OF PIGMENTED COATINGS CURED WITH H BULB
(SHEET 1 of 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Tests	Comments
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P106 P44 TiO ₂	24.00 g 16.00	2 Passes 6.25 ft/min 300 w/in	100%	100%	100%	3H	F	F	7/7/80
P67B TS3577-11 TiO ₂	20.00 g 18.25	3 Passes 6.25 ft/min 300 w/in	100%	100%	100%	-	-	-	8/27/80
P68E TS3577-11 V10 TiO ₂	20.00 g 0.20 18.25	4 Passes 6.25 ft/min 300 w/in	67%	97%	0%	-	-	-	4/15/80
P68D TS3577-11 Irg. TiO ₂	20.00 g 0.20 18.25	10 Passes 6.25 ft/min 300 w/in	100%	100%	11%	-	-	-	4/15/80
P75B TS3577-11 DTBP TiO ₂	18.25 g 0.18 12.17	4 Passes 6.25 ft/min 300 w/in	100%	100%	53%	-	-	-	5/16/80
P69E TS3577-11 DTBP V10 TiO ₂	18.25 g 0.18 0.18 12.17	3 Passes 6.25 ft/min 300 w/in	93%	95%	0%	-	-	-	5/16/80
P69D TS3577-11 DTBP Irg. TiO ₂	18.25 g 0.18 0.18 12.17	3 Passes 6.25 ft/min 300 w/in	94%	100%	2%	-	-	-	5/16/80
P70E TS3577-11 DTBP MDEA TiO ₂	18.25 g 0.18 0.18 12.17	7 Passes 6.25 ft/min 300 w/in	100%	100%	52%	-	-	-	5/16/80
P71E TS3577-11 DTBP MDEA V10 TiO ₂	18.25 g 0.18 0.18 0.18 12.17	3 Passes 6.25 ft/min 300 w/in	100%	100%	1%	-	-	-	5/16/80

Fusion System Corp. H Bulb
Lamp Width (Irradiation Zone) 8.25 in.
Effective Radiation Time is 6.6 sec/pass.

TABLE 52 - (CON'T) USING H BULB - PIGMENTED COATINGS (SHEET 2 OF 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Tests	Comments
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P71D TS3577-11 18.25 g DTBP 0.18 MDEA 0.18 Irg. 0.18 TiO ₂ 12.17	3 Passes 6.25 ft/min 300 w/in	74%	100%	8%	-	-	-	5/16/80	
P72B TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.275 MDEA 0.275 VIO 0.275 TiO ₂ 18.25	6 Passes 6.25 ft/min 300 w/in	100%	100%	13%	-	-	-	5/16/80	
P72D (P72C + 40% Rutile TiO ₂) TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.2 Irg. 0.28 MDEA 0.28 TiO ₂ 18.25	3 Passes 6.25 ft/min 300 w/in	100%	100%	85% 73%	HB F	HB 3B	3B 2B	6/5/80 6/10/80	
P73B TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.275 VIO 0.275 TiO ₂ 18.25	9 Passes 6.25 ft/min 300 w/in	100%	100%	65%	-	-	-	5/20/80	
P73D TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.28 Irg. 0.28 TiO ₂ 18.25	5 Passes 6.25 ft/min 300 w/in	100%	100%	99%	-	-	-	5/20/80	
P74B TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.275 MDEA 0.275 TiO ₂ 18.25	11 Passes 6.25 ft/min 300 w/in	100%	100%	100%	-	-	-	5/16/80	
P75B TS3577-11 18.25 g DTBP 0.18 TiO ₂ 12.17	4 Passes 6.25 ft/min 300 w/in	100%	100%	53%	-	-	-	5/20/80	
P76B TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.275 TiO ₂ 18.25	10 Passes 6.25 ft/min 300 w/in	100%	100%	100%	-	-	-	5/20/80	

TABLE 52 - (CON'T) PIGMENTED COATINGS - USING H BULB (SHEET 3 OF 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test	Comments
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P77B TS3577-11 HDDA EEA VIO TiO ₂	20.00 g 5.00 2.50 0.28 18.25	11 Passes 6.25 ft/min 300 w/in	100%	100%	100%	-	-	-	5/20/80
P77C TS3577-11 HDDA EEA Irg. TiO ₂	20.00 g 5.00 2.50 0.28 18.25	6 Passes 6.25 ft/min 300 w/in	100%	100%	100%	-	-	-	5/20/80
P91 TS3401-1F DTBP MDEA TiO ₂	22.50 g 6.23 6.23 15.00	4 Passes 6.25 ft/min 300 w/in	100%	100%	5% 37%	B HB	HB F	5B 5B	7/1/80 7/7/80
P93 TS3401-1F HDDA EEA DTBP Irg. MDEA TiO ₂	20.00 g 5.00 2.50 0.2 0.28 18.25	3 Passes 6.25 ft/min 300 w/in	8% 57%	93% 100%	43% 0%	<6B <6B	<6B <6B	<6B <6B	7/3/80 7/7/80
P10 ^a P72C P97 P44 TiO ₂	12.00 g 12.00 16.00	3 Passes 6.25 ft/min 300 w/in	100%	100%	100%	4H	2H	F	7/7/80
P108 P72C P98 P44 TiO ₂	16.00 g 8.00 16.00	6 Passes 12.5 ft/min 300 w/in	100%	100%	100%	B	<6B	<6B	7/8/80
P109 P72C P99 P44 TiO ₂	32.00 g 6.00 20.00	7 Passes 12.5 ft/min 300 w/in	100%	100%	100%	<6B	<6B	<6B	7/8/80
P110 P72C P100 P44 TiO ₂	32.00 g 4.00 24.00	7 Passes 12.5 ft/min 300 w/in	100%	100%	100%	<6B	<6B	<6B	7/8/80
P111 P101 TiO ₂	24.00 g 16.00	8 Passes 12.5 ft/min 300 w/in	100% 100%	100% 100%	100% 100%	2B 2B	4B <6B	2B 6B	7/11/80 7/14/80
P112 P72C P102 P101 TiO ₂	12.00 g 12.00 16.00	8 Passes 12.5 ft/min 300 w/in	100%	100%	100%	3H	HB	F	7/11/80
							HB	H	7/14/80

TABLE 52- PIGMENTED COATINGS - USING H BULB
(SHEET 4 OF 4)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Tests	Comments
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P113 P103 P101 TiO ₂	P72C 16.00 g 8.00 16.00 4 Passes 12.5 ft/min 300 w/in	100%	100%	100%	<6B	<6B	<6B	7/9/80	
P114 P104 P101 TiO ₂	P72C 24.00 g 6.00 20.00 4 Passes 12.5 ft/min 300 w/in	100%	100%	100%	<6B	<6B	<6B	7/9/80	
P115 P105 P101 TiO ₂	P72C 24.00 g 3.00 18.00 8 Passes 12.5 ft/min 300 w/in	100%	100%	100%	6B	<6B	<6B	7/11/80	

TABLE 53-PROPERTIES OF COATINGS CURED WITH H & D BULBS
(SHEET 1 of 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Test	Comments
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P44 Alpolit 50.00 g HDDA 30.00 EEA 10.00 DTBP 0.90 VIO 0.90 MDEA 0.90	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	100%	100%	100% H. Lines	2H	3H	F	7/15/80	
P720 TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.28 Irg. 0.28 MDEA 0.28 TiO ₂ 18.25	7 Passes 12.5 ft/min 2 Lamps @ 300 w/in	100%	100%	100%	5B	2B	5B	7/10/80	Uneven Coating On Fe Phos
P91 TS3401-1F 22.30 g DTBP 6.23 MDEA 6.23 TiO ₂ 15.00	5 Passes 12.5 ft/min 2 Lamps @ 300 w/in	100%	60%	92%	F	H	2B	7/10/80	
P93 TS3401-1F 40.00 g HDDA 10.00 EEA 5.00 DTBP 0.55 Irg. 0.55 MDEA 0.55 TiO ₂ 22.00	4 Passes 12.5 ft/min 2 Lamps @ 300 w/in	0%	3%	0%	<6B	<6B	<6B	7/10/80	
P94 TS3401-1F TS3577-11 40.00 MDDA 10.00 EEA 5.00 DTBP 0.55 Irg. 0.55 MDEA 0.55	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	1%	11%	1%	F	2H	2B	7/17/80	
P95 (P87) TS3401-1F	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	100%	100%	30%	HB	F	F	7/17/80	
		100%	100%	6%	B	F	B	7/21/80	

TABLE 53 (CON'T) USING H & D BULBS
(SHEET 2 OF 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness			Date of Tests	Comments
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P97 P72C P44	5.00 g 5.00	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	47% 77%	7% 3%	0% 0%	3H 3H	3H 2H	HB HB	7/15/80 7/17/80
P98 P72C P44	8.00 g 4.00	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	1% 5%	1% 0%	0% 0%	3H 3H	2H 3H	HB HB	7/15/80 7/17/80
P99 P72C P44	16.00 g 4.00	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	0% 0%	100% 100%	0% 0%	H H	2H 3H	HB 2B	7/15/80 7/17/80
P100 P72C P44	16.00 g 2.00	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	0% 0%	2% 0%	0% 0%	3H 3H	3H 3H	4B 4B	7/15/80 7/17/80
P101 Apolit HDDA EEA DTBP Irg. N'DEA	50.00 g 30.00 10.00 0.90 0.90 0.90	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	100% 100%	61% 100%	100% 100%	3H H	4H >9H	HB F	7/17/80 7/21/80
P102 P72C P101	4.00 g 4.00	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	24% 45%	76% 0%	0% 0%	2H F	5H 2H	F HB	7/10/80 7/14/80
P103 P72C P101	6.00 g 3.00	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	58% 49%	85% 51%	0% 0%	3H 3H	7H 5H	2B 4B	7/10/80 7/14/80
P104 P72C P101	6.00 g 3.00	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	4% 1%	63% 33%	0% 0%	H H	5H 3H	2B 2B	7/10/80 7/14/80
P105 P72 P101	16.00 g 2.00	1 Pass 12.5 ft/min 2 Lamps @ 300 w/in	0% 18%	0% 0%	0% 0%	2H 3H	2H 3H	2B 3B	7/10/80 7/14/80
P106 P44 TiO ₂	24.00 g 16.00	4 Passes 12.5 ft/min 2 Lamps @ 300 w/in	100% 100%	100% 100%	100% 100%	H HB	H 2B	2H F	7/14/80 7/16/80
P107 P72C P44 TiO ₂	12.00 g 12.00 16.00	5 Passes 12.5 ft/min 2 Lamps @ 300 w/in	100% 100%	100% 100%	100% 100%	3H 2H	2H F	H F	7/14/80 7/16/80
P108 P72C P44 TiO ₂	16.00 g 8.00 16.00	5 Passes 12.5 ft/min 2 Lamps @ 300 w/in	100% 100%	100% 97%	100% 100%	H H	3B 4B	3B 3B	7/14/80 7/16/80
P109 P72C P44 TiO ₂	24.00 g 6.00 20.00	6 Passes 12.5 ft/min 2 Lamps @ 300 w/in	100% 100%	100% 100%	100% 100%	H F	2H F	H HB	7/14/80 7/16/80
P110 P72C P44 TiO ₂	32.00 g 4.00 24.00	6 Passes 12.5 ft/min 2 Lamps @ 300 w/in	100% 100%	100% 100%	100% 100%	HB HB	2B HB	2B 4B	7/14/80 7/16/80

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10-1-80

Dwg. 7732A07

TABLE 54-PROPERTIES OF COATINGS CURED WITH D BULB

Using D Bulb - 300 w/in										
P44	Alpolit	50.00 g	1 Pass	100%	89%	100%	3H	5H	F	7/17/80
	HDDA	30.00	12.5 ft/min			100%	2H	5H	HB	7/21/80
	EEA	10.00	2 Lamps	100%	100%					
	DTBP	0.90								
	VIO	0.90								
	MDEA	0.90								

97

Dwg. 7732A07

TABLE 55-PROPERTIES OF PIGMENTED COATINGS CONTAINING ACRYLIC MONOMERS AND 1.00%
2-ETHYLANTHRAQUINONE (EAQ) CURED WITH D BULB (SHEET 1 of 3)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P160A I								
TS3577-11	30.00g	7 Passes	100%	97%	100%	6H	HB	2B
HDDA	10.00	12.5 ft/min				6H	H	3B
EEA	5.00	300 w/in						11/14/80
EAQ	0.23	D Bulb						
Titanox	20.00							
2010								
P160A II								
TS3577-11	30.00g	5 Passes	100%	100%	100%	3B	<6B	F
HDDA	10.00	12.5 ft/min				3B	<6B	12/15/80
EEA	5.00	300 w/in						
EAQ	0.23	D Bulb						
Titanox	20.00							12/17/80
2010								
P160A III								
TS3577-11	30.00g	6 Passes	100%	100%	100%	3H	H	H
HDDA	10.00	12.5 ft/min				3H	2H	3H
EEA	5.00	300 w/in						12/30/80
EAQ	0.23	D Bulb						
Titanox	20.00							1/2/81
2010								
P251								
TS3577-11	30.00g	5 Passes	100%	100%	64%	HB	B	<6B
TPGDA	10.00	12.5 ft/min				HB	HB	6B
EEA	5.00	300 w/in						1/14/81
EAQ	0.23	D Bulb						1/16/81
Titanox	20.00							
2010								
P252								
TS3577-11	30.00g	5 Passes	100%	100%	0%	F	2B	3B
TTHFA	10.00	12.5 ft/min				HB	2B	2B
EEA	5.00	300 w/in						1/14/81
EAQ	0.23	D Bulb						1/16/81
Titanox	20.00							
2010								

TABLE 55-PROPERTIES OF PIGMENTED COATINGS CONTAINING ACRYLIC MONOMERS AND 1.00% 2-ETHYLANTHRAQUINONE (EAQ) CURED WITH D BULB (SHEET 2 of 3)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P253 I								
TS3577-11 30.00g	5 Passes	100%	100%	79%	5B	B	4B	1/15/81
TPGDA 5.00	12.5 ft/min							
EEA 5.00	300 w/in	100%	100%	100%	3B	B	2B	1/19/81
EAQ 0.20	D Bulb							
Titanox 2010 20.00		100%	100%	100%	2B	2B	2B	1/21/81
P253 II								
TS3577-11 30.00g	5 Passes	100%	100%	100%	2B	2B	2B	1/20/81
TPGDA 5.00	12.5 ft/min							
EEA 5.00	300 w/in	100%	100%	100%	HB	2B	2B	1/22/81
EAQ 0.20	D Bulb							
Titanox 2010 20.00								
P253 III								
TS3577-11 30.00g	5 Passes	100%	100%	100%	2B	6B	3B	1/20/81
TPGDA 5.00	12.5 ft/min							
EEA 5.00	300 w/in	100%	100%	100%	HB	2B	2B	1/22/81
EAQ 0.20	D Bulb							
Titanox 2010 20.00								
P256								
TS3577-11 30.00g	5 Passes	100%	100%	10%	F	HB	3B	2/3/81
TPGDA 5.00	12.5 ft/min							
EEA 5.00	300 w/in	100%	100%	100%	2H	F	2B	2/5/81
EAQ 0.20	D Bulb							
Titanox 2010 26.60								
P262								
TS3577-11 30.00g	5 Passes	100%	100%	64%	<6B	<6B	<6B	1/16/81
D.S. 4028 10.00	12.5 ft/min							
EEA 5.00	300 w/in	100%	100%	100%	<6B	<6B	6B	1/19/81
EAQ 0.23	D Bulb							
Titanox 2010 20.00								
P263								
TS3577-11 30.00g	5 Passes	100%	100%	16%	6B	<6B	<6B	1/16/81
D.S. 4028 10.00	12.5 ft/min							
D.S. 4039 5.00	300 w/in	100%	100%	40%	6B	<6B	<6B	1/19/81
EAQ 0.23	D Bulb							
Titanox 2010 20.00								

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3-23-81

Dwg. 1719504

TABLE 55-PROPERTIES OF PIGMENTED COATINGS CONTAINING ACRYLIC MONOMERS AND 1.00% 2-ETHYLANTHRAQUINONE (EAQ) CURED WITH D BULB (SHEET 3 of 3)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P264								
TS3577-11	30.00g	5 Passes	99%	100%	24%	<6B	6B	<6B
D.S. 4072	10.00	12.5 ft/min						1/16/81
D.S. 4039	5.00	300 w/in						
EAQ	0.23	D Bulb	100%	100%	92%	5B	5B	6B
Titanox	20.00							1/19/81
2010								
P265								
TS3577-11	30.00g	5 Passes	100%	100%	6%	F	F	4B
D.S. 4072	10.00	12.5 ft/min						1/16/81
EEA	5.00	300 w/in						
EAQ	0.23	D Bulb	100%	100%	32%	F	HB	3B
Titanox	20.00							1/19/81
2010								
P280								
TS3577-11	30.00g	5 Passes						
BGDA	5.00	12.5 ft/min	100%	100%	100%	H	H	HB
EEA	5.00	300 w/in						2/25/81
EAQ	0.20	D Bulb	100%	100%	100%	F	H	F
Titanox	26.60							2/27/81
2010								
P281								
TS3577-11	30.00g	5 Passes	100%	100%	100%	H	3H	HB
BDAA	5.00	12.5 ft/min						2/25/81
EEA	5.00	300 w/in	100%	100%	100%	H	3H	HB
EAQ	0.20	D Bulb						2/27/81
Titanox	26.60							
2010								

Dwg. 1719804

TABLE 56 - PRIMERS CONTAINING VARIOUS MONOMER DILUENTS AND FLOW CONTROL AGENTS (1 OF 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P. P. 236								
TS3577-11	28.20 g	4 Passes	100%	100%	5B	4B	<6B	1/9/81
Iron Oxide	22.50	12.5 ft/min		0%				
Zinc Chromate	4.50	300 w/in						
FC 430	0.84	D Bulb	100%	100%	4B	3B	<6B	1/12/81
EAQ	0.14							
P. P. 237								
TS3577-11	28.20 g	5 Passes	100%	86%	2B	5B	6B	1/12/81
Iron Oxide	22.50	12.5 ft/min		0%				
Zinc Chromate	4.50	300 w/in						
Ganex	1.68	D Bulb	100%	88%	HB	5B	<6B	1/14/81
EAQ	0.14							
P. P. 240								
TS3577-11	28.20 g	6 Passes	100%	100%	4B	<6B	<6B	1/12/81
Iron Oxide	22.50	12.5 ft/min		1%				
Zinc Chromate	4.50	300 w/in						
FC 430	0.84	D Bulb	100%	100%	6B	<6B	<6B	1/14/81
Aerosil 200	0.28							
EAQ	0.14							
P. P. 238								
TS3577-11	28.20 g	7 Passes	100%	100%	13%	<6B	<4B	6B
Iron Oxide	22.50	12.5 ft/min	100%	100%	63%	4B	4B	5B
Zinc Chromate	4.50	300 w/in						
Ebec 170H	0.28	D Bulb						
EBEC 358	0.28							
EAQ	0.14							
P. P. 268								
TS3577-11	28.20 g	6 Passes	100%	100%	47%	5B	<6B	1/23/81
Iron Oxide	22.50	12.5 ft/min		47%				
Zinc Chromate	4.50	300 w/in						
TPGDA	7.50 g	D Bulb	100%	100%	82%	4B	<6B	1/26/81
EEA	7.50 g							
EAQ	0.20							

A. Grossel
d. m. s. - μ d 10-27-81

Dwg. 424986-

TABLE 56 - (2 OF 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P. P. Z39								
TS3577-11	28.20 g	8 Passes						
Iron Oxide	22.50	12.5 ft/min	100%	100%	100%	2B	5B	4B
Chemlink Z	4.50	D Bulb	100%	100%	100%	B	4B	<6B
EAQ	0.14							
P. P. 266								
TS3577-11	28.20 g	6 Passes						
Iron Oxide	22.50	12.5 ft/min	100%	100%	97%	2B	3B	3B
Chemlink Z	0.56	300 w/in				2B	3B	4B
TPGDA	5.0	D Bulb	100%	100%	100%			
EEA	5.0							
EAQ	0.14							

Dwg. 4249864

TABLE 57-PROPERTIES OF PRIMERS PIGMENTED WITH IRON OXIDE AND ZINC CHROMATE AND CONTAINING DIFFERENT PHOTONINITIATORS CURED WITH D BULB (SHEET 1 of 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
P. P. 204								
TS3577-11	28.20g	10 Passes	100%	100%	31%	B	B	6B
Iron Oxide	22.50	12.5 ft/min						12/17/80
Zinc Chromate	4.50	300 w/in						
Irg. 651	0.85	D Bulb						12/19/80
P. P. 205								
TS3577-11	28.20g	6 Passes	100%	100%	35%	B	<6B	6B
Iron Oxide	22.50	12.5 ft/min						12/18/80
Zinc Chromate	4.50	300 w/in						
CTX	0.11	D Bulb						12/22/80
MDEA	0.28							
P. P. 270								
TS3577-11	28.20g	4 Passes	100%	100%	20%	3B	<6B	4B
Iron Oxide	22.50	12.5 ft/min						1/26/81
Zinc Chromate	4.50	300 w/in						
TPGDA	12.50	D Bulb						
EEA	7.50							
CTX	0.17							
MDEA	0.40							

TABLE 57-PROPERTIES OF PRIMERS PIGMENTED WITH IRON OXIDE AND ZINC CHROMATE AND
CONTAINING DIFFERENT PHOTONIATORS CURED WITH D BULB (SHEET 2 of 2)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates	
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P.P.206									
TS3577-11	28.20g	5 Passes	22%	100%	100%	H	6B	3B	12/31/80
Iron Oxide	22.50	12.5 ft/min							
Zinc Chromate	4.50	300 w/in							
EAQ	0.14	D Bulb	100%	100%	100%	H	<6B	2B	1/2/81
P.P.236									
TS3577-11	28.20g	4 Passes	100%	100%	0%	5B	4B	<6B	1/9/81
Iron Oxide	22.50	12.5 ft/min							
Zinc Chromate	4.50	300 w/in							
FC 430	0.84	D Bulb	100%	100%	0%	4B	3B	<6B	1/12/81
EAQ	0.14								
P.P.237									
TS3577-11	28.20g	5 Passes	100%	86%	0%	2B	5B	6B	1/12/81
Iron Oxide	22.50	12.5 ft/min							
Zinc Chromate	4.50	300 w/in							
Ganex	1.68	D Bulb	100%	88%	0%	HB	5B	<6B	1/14/81
EAQ	0.14								
P.P.240		6 Passes							
TS3577-11	28.20g	12.5 ft/min	100%	100%	1%	4B	<6B	<6B	1/12/81
Iron Oxide	22.50	300 w/in							
Zinc Chromate	4.50	D Bulb	100%	100%	0%	6B	<6B	<6B	1/14/81
FC 430	0.84								
Aerosil	0.28								
200									
EAQ	0.14								
P.P.238		7 Passes							
TS3577-11	28.20g	12.5 ft/min	100%	100%	13%	<6B	<4B	6B	1/12/81
Iron Oxide	22.50	300 w/in							
Zinc Chromate	4.50	D Bulb	100%	100%	63%	4B	4B	5B	1/14/81
EBEC 170H	0.28								
EBEC 358	0.28								
EAQ	0.14								
P.P.268		6 Passes							
TS3577-11	28.20g	12.5 ft/min	100%	100%	47%	5B	<6B	<6B	1/23/81
Iron Oxide	22.50	300 w/in							
Zinc Chromate	4.50	D Bulb	100%	100%	82%	4B	<6B	<6B	1/26/81
TPGDA	7.50g								
EEA	7.50g								
EAQ	0.20								

TABLE 58-PROPERTIES OF PRIMERS PIGMENTED WITH IRON OXIDE AND CONTAINING 2-ETHYLANTHRAQUINONE (EAQ) AND A ZINC DIACRYLATE (CHEMLINK Z) CURED WITH D BULB

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates	
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
P.P.207 I									
TS3577-11	28.20g	5 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	100%	HB	2B	3B	1/9/81
Iron Oxide	22.50								
Chemlink Z	0.56								
EAQ	0.14								
P.P.207 II									
TS3577-11	28.20g	5 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	22%	HB	HB	4B	1/14/81
Iron Oxide	22.50								
Chemlink Z	0.56								
EAQ	0.14								
P.P.207 III									
TS3577-11	28.20g	5 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	100%	B	2B	2B	1/15/81
Iron Oxide	22.50								
Chemlink Z	0.56								
EAQ	0.14								
P.P.239									
TS3577-11	28.20g	8 Passes 12.5 ft/min D Bulb	100%	100%	100%	2B	5B 4B	4B <6B	1/12/81 1/14/81
Iron Oxide	22.50								
Chemlink Z	4.50								
EAQ	0.14								
P.P.266									
TS3577-11	28.20g	6 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	97%	2B	3B	3B	1/22/81
Iron Oxide	22.50								
Chemlink Z	0.56								
TPGDA	5.0								
EEA	5.0								
EAQ	0.14								

to exert little effect on cure time. Adherent films were obtained using the following monomer combinations added to 30 parts of the base resin: 10 parts HDDA to 5 parts EEA, 5 parts TPGDA to 5 parts EEA, 5 parts BGDA to 5 parts EEA, and 5 parts BDDA to 5 parts EEA. The addition of surface active agents or monomers reduced adhesion and increased cure times. The addition of small amounts of zinc diacrylate, Chemlink Z, to iron oxide pigmented films only increased the gloss.

An interesting alternative to photoinduced free radical polymerization is at the photoinduced cationic polymerization.²³ Unlike free radical chain reactions, cationic polymerizations have few termination reactions.²⁴

Epoxy compounds and resins are of particular interest in UV curable paint applications. In general, these materials are readily available as commodity items, and the resulting cured polymers possess excellent dimensional and thermal stability as well as superior mechanical strength and chemical resistance. These UV curable epoxy resin based paints also have the following advantages over UV cured acrylated resin paints: good adhesion to substrate, safer raw materials which have less skin sensitivity and odor, as well as lower volatility. With these advantages in mind, the cationic polymerization of epoxy resins in UV curable paint applications was carefully examined.

An epoxy resin ERL 4221 (Union Carbide) was used as a paint vehicle in the preliminary experiments because of its rapid cure speed^{25,26} and excellent film adhesion.²⁷ Reactive diluents, butyl glycidyl ether, 1,4-butanediol diglycidyl ether (Ciba Geigy RD-2), n-butanol and limonene dioxide were used to lower viscosity. A surfactant, 3M Co. FC-171, was used in the formulations to improve the wetting properties of the coating. Table 59 shows the results of the preliminary study. They have a good initial adhesion and pencil hardness. It is significant to note the

TABLE 59—PROPERTIES OF UV CURABLE EPOXY PAINT CONTAINING FC-509 AND FC-510

Formulation	Cure Time	Cross Hatch Test			Pencil Hardness			Date of Test	
		Zn Phos	Fe-Phos	Steel	Zn Phos	Fe Phos	Steel		
CP 186 18 W 286	35.00g	CP 186A D Bulb 3 Passes	100%	97%	0%	3H	F	2B	11/24/80
ERL4221	7.50	12.5 ft/min							
FC-509	1.50	300 W/in	100%	7%	100%	5H	H	H	11/26/80
FC-510	0.50	CP 186B V Bulb 3 Passes	100%	100%	85%	3H	H	2B	11/24/80
FC-171	0.25	12.5 ft/min							
Butyl Glycidyl Ether	3.00	300 W/in	100%	100%	100%	3H	2H	F	11/26/80
n-Butanol	2.50								
CP 187		CP 187A D Bulb 3 Passes							
ERL4221	25.00g	100%	100%	0%	3H	F	B	11/24/80	
Titanox 2020	17.50	12.5 ft/min							
FC-509	1.50	300 W/in	100%	100%	100%	3H	3H	H	11/26/80
FC-510	0.50	CP 187B V Bulb 3 Passes	100%	100%	0%	6H	2H	2B	11/24/80
FC-171	0.25	12.5 ft/min							
Limonene Dioxide	3.00	300 W/min	100%	100%	97%	5H	2H	H	11/26/80
RD-2	2.50								
CP 189A		CP 189A D Bulb 3 Passes							
ERL4221	25.00g	100%	100%	100%	4H	HB	HB	11/24/80	
Titanox 2020	17.50	12.5 ft/min							
FC-509	1.50	200 W/in	100%	100%*	100%*	3H	F	HB	11/26/80
FC-510	0.50	CP 189B V Bulb 3 Passes	100%	100%	100%	3H	F	F	11/24/80
FC-171	0.25	12.5 ft/min							
Limonene Dioxide	5.50	300 W/in	100%	100%*	100%	3H	H	2H	11/26/80

*Very brittle, coating is almost off the square

increase in adhesion and hardness properties two days after exposure to UV light. This may be due to the "living" characteristic^{23,28} of cationic polymerization which causes the film to set and continue to cure after UV radiation.

The cure rate of UV curable epoxy paint depends on the structure and concentration of the epoxy resins and diluents as well as the photo-initiator, photosensitizer, and light source. Table 60 shows the dependence of curing speed of UV curable epoxy paint on epoxy resin. The epoxidized olefins are expected to have a high rate of cure^{25,26} because of their low epoxy equivalent and their lack of a reaction retarding electron donor group in the epoxy structure. Diepoxy octane was used in formulation CP217 to replace part of ERL 4221 epoxy resin, as used in formulation CP211. The lack of a cure rate increase may be due to the low amount of diepoxy octane used. All the epoxy cannot be replaced with diepoxy octane because of its high volatility and cost. Formulation CP218 which contains more ERL 4221 and less Epoxide 8 than formulation CP211, has a lower epoxy equivalent weight and gave harder films. This may indicate CP218 cures faster than CP211.

The flexibility of ERL 4221 cycloaliphatic epoxide based coatings can be modified by using a long chain epoxy resin, long chain reactive diluent or polyol. Formulations containing ERL 4299 and Celanese Epi-Rez 502, Epoxide 8 reactive diluent and polyol LHT240 were evaluated. Limonene dioxide, RD-1, RD-2 and diepoxy octane were added to maintain the fast cure rate and low viscosity. Table 61 shows the results of this feasibility study. The addition of the flexibilizers causes a deterioration of physical properties of the cured films. For example, while formulation CP246 has 98% adhesion and a HB pencil hardness, formulation CP240 has 100% adhesion and only B pencil hardness owing to its slower reaction rate.

UV curable epoxy primers were formulated using a base resin, ERL4221, and a reactive diluent, limonene dioxide, for viscosity reduction. The results are shown in Table 62. The cure rates of 1 mil thick films ranged from 30 sec. to 150 sec. Film hardnesses of H (formulation PCP289 and 290)

TABLE 60-DEPENDENCE OF CURING SPEED OF UV CURABLE EPOXY PAINT ON EPOXY COMPONENTS

Formulation	Cure Time	Crosshatch Test Cold Rolled Steel	Pencil Hardness Test Cold Rolled Steel	Dates
CP211				
ERL 4221 25.00g Epoxide 8 5.50	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	<6B	12/30/80
FC-508 1.50 FC-510 0.50 FC-171 0.25		100%	<6B	1/2/81
Titanox 2020 17.50	3 Passes 12.5 ft/min 300 w/in V Bulb	100%	<6B	12/30/80
		100%	<6B	1/2/81
CP218				
ERL 4221 27.50g Epoxide 8 3.00	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	<6B	12/31/80
FC-508 1.50 FC-510 0.50 FC-171 0.25		100%	3B	1/2/81
Titanox 2020 17.50	3 Passes 12.5 ft/min 300 w/in V Bulb	100%	<6B	12/31/80
		100%	4B	1/2/81
CP217				
ERL 4221 20.00g Epoxide 8 5.50	2 Passes 12.5 ft/min 300 w/in D Bulb	0%	<6B	12/31/80
Diepoxy Octane 5.00		0%	<6B	1/2/81
FC-508 1.50 FC-510 0.50 FC-171 0.25	2 Passes 12.5 ft/min 300 w/in V Bulb	0%	<6B	12/31/80
Titanox 2020 17.50	3 Passes 12.5 ft/min 300 w/in V Bulb	0%	4B	1/2/81
		0%	<6B	12/31/80
		0%	<6B	1/2/81

TABLE 61 - FLEXIBILITY STUDY OF UV CURABLE EPOXY PAINT (PAGE 1 OF 3)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates	
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
CP246 ERL 4221 RD-1 LHT-240	15.00g 10.00 5.50	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	0%	B	3B	4B	1/30/81
			100%	100%	28%	HB	F	3B	2/2/81
	17.50	3 Passes 12.5 ft/min 300 w/in V Bulb	100%	30%	92%	F	3B	2B	1/30/81
			100%	100%	98%	F	F	HB	2/2/81
CP 244 ERL 4299 RD-1 FC-508 FC-510 FC-171	25.00g 5.50	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	0%	F	2B	3B	1/14/81
			100%	100%	90%	2H	HB	B	1/16/81
		3 Passes 12.5 ft/min 300 w/in	100%	84%	0%	F	2B	3B	1/14/81
	2.00 0.50 0.25		100%	100%	82%	2H	HB	B	1/16/81
		3 Passes 12.5 ft/min 300 w/in D Bulb	100%	20%	0%	HB	4B	3B	1/15/81
			100%	90%	25%	F	2B	2B	1/19/81
CP247 ERL 4299 Limonene Dioxide	25.00g 5.50	3 Passes 12.5 ft/min 300 w/in V Bulb	35%	0%	0%	HB	3B	2B	1/15/81
			100%	85%	85%	F	B	HB	1/19/81
		3 Passes 12.5 ft/min 300 w/in D Bulb	100%	90%	0%	HB	3B	2B	1/15/81
	2.00 0.50 0.25		100%	85%	85%	F	B	HB	1/19/81
		3 Passes 12.5 ft/min 300 w/in V Bulb	100%	90%	0%	HB	2B	<6B	1/15/81
			100%	100%	0%	F	HB	3B	1/19/81
CP 248 ERL 4299 Diepoxy Octane	25.00g 5.00	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	90%	0%	B	2B	3B	1/15/81
			100%	100%	0%	F	HB	3B	1/19/81
	2.00 0.50 0.25	3 Passes 12.5 ft/min 300 w/in V Bulb	100%	86%	0%	HB	HB	3B	1/15/81
			100%	100%	0%	2H	F	3B	1/19/81

TABLE 61-FLEXIBILITY STUDY OF UV CURABLE EPOXY PAINT (PAGE 2 OF 3)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates	
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel		
CP245									
ERL 4221	15.00g	3 Passes 12.5 ft/min 200 w/in D Bulb	100%	100%	100%	3B	3B	3B	1/14/81
Epi-rez-502	5.00								
Titanox 2020	17.50								
FC-508	2.00								
FC-510	0.50								
FC-171	0.25								
Limonene	10.00	3 Passes 12.5 ft/min 200 w/in V Bulb	100%	100%	100%	B	2B	B	1/16/81
Dioxide									
CP273									
ERL 4221	20.00g	3 Passes 12.5 ft/min 200 w/in D Bulb	100%	66%	0%	2B	6B	4B	1/30/81
LHT 240	2.26								
RD-1	5.00								
RD-2	2.50								
FC-508	2.00								
FC-510	0.50								
FC-171	0.25								
Limonene	3.00	3 Passes 12.5 ft/min 200 w/in V Bulb	100%	100%	0%	2B	H	4B	1/30/81
Dioxide									
CP272									
ERL 4221	16.00g	3 Passes 12.5 ft/min 200 w/in D Bulb	100%	95%	0%	HB	HB	4B	1/30/81
LHT 240	4.50								
RD-1	10.00								
Titanox 2020	17.50								
FC-508	2.00								
FC-510	0.50								
FC-171	0.25								
CP243									
ERL 4221	25.00g	3 Passes 12.5 ft/min 200 w/in	100%	100%	12%	2B	HB	4B	1/14/81
RD-2	5.00								
Titanox 2020	17.50								
FC-508	2.00	D Bulb							
FC-510	0.50	3 Passes 12.5 ft/min 200 w/in	100%	100%	78%	HB	HB	4B	1/14/81
FC-171	0.25	V Bulb	100%	100%	100%	F	F	2B	1/16/81

TABLE 61 -FLEXIBILITY STUDY OF UV CURABLE EPOXY PAINT (PAGE 3 OF 3)

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
CP216 II ERL 4221 25.00g Epoxide 8 5.50 FC-508 2.00 FC-510 0.50 FC-171 0.25	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	100%	5B	6B	<6B	12/31/80
		100%	100%	100%	HB	3B	5B	1/2/81
Titanox 2020 17.50	3 Passes 12.5 ft/min 300 w/in V Bulb			100%			3B	12/30/80
				100%			3B	1/2/81
CP249 ERL 4221 20.00g Epoxide 8 5.50 Titanox 2020 17.50	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	100%	<6B	<6B	6B	1/15/81
		100%	100%	100%	4B	4B	3B	1/19/81
FC-508 2.00 FC-510 0.50 FC-171 0.25 Limonene Dioxide 5.00	3 Passes 12.5 ft/min 300 w/in V Bulb	100%	100%	28%	<6B	2B	4B	1/15/81
		100%	100%	100%	4B	HB	2B	1/19/81
CP275 ERL 4221 25.00g Epoxide 8 6.00 Titanox 2020 17.50	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	32%	0%	3B	6B	<6B	2/23/81
		99%	100%	80%	B	4B	2B	2/25/81
FC-508 2.00 FC-510 0.5 FC-171 0.25 RD-2 5.0	3 Passes 12.5 ft/min 300 w/in V Bulb	100%	100%	0%	HB	3B	4B	2/23/81
		100%	100%	14%	F	HB	3B	2/25/81
CP277 ERL 4221 25.00g Epoxide 8 5.50 Titanox 2020 17.50	3 Passes 12.5 ft/min 300 w/in D Bulb	100%	100%	88%	3B	5B	<6B	2/23/81
		100%	97%	100%	2B	3B	6B	2/25/81
FC-508 2.00 FC-510 0.50 FC-171 0.25 A-186 0.50	3 Passes 12.5 ft/min 300 w/in V Bulb	100%	98%	25%	3B	5B	6B	2/23/81
		100%	100%	92%	2B	3B	5B	2/25/81

TABLE 62 - THE PROPERTIES OF UV CURABLE EPOXY PRIMER

Formulation	Cure Time	Crosshatch Test			Pencil Hardness Test			Dates
		Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	
PCP 288								
Red Iron Oxide	7.14	D Bulb 300 w/in 12.5 ft/min	100 %	100 %	100 %	4H	3H	3H
Halox BW-111	11.25	3 Passes for Steel						
Fibrene C-400	21.51	5 Passes for Fe-Phos. and Zn-Phos.	100 %	100 %	100 %	5H	4H	4H
Bentone 34	0.61							
ERL 4221	49.74							
Limonene Dioxide	9.95							
FC-509	3.00							
FC-510	1.00							
PCP 289								
Red Iron Oxide	7.14	D Bulb 300 w/in 12.5 ft/min	100 %	100 %	100 %	H	2H	H
Halox BW-111	11.25	3 Passes for Steel						
Fibrene C-400	21.51	5 Passes for Fe-Phos- & Zn-Phos.	100 %	100 %	100 %	H	2H	H
Bentone 34	0.61							
ERL 4221	49.74							
Limonene Dioxide	9.95							
FC-508	4.00							
FC-510	1.00							
PCP 290								
Red Iron Oxide	7.14	D Bulb 300 w/in 12.5 ft/min	100 %	100 %	100 %	3H	3H	H
Halox BW-111	11.25	2 Passes for Steel						
Fibrene C-400	21.51	3 Passes for Zn-Phos. & Fe-Phos.	100 %	100 %	100 %	4H	4H	3H
Bentone 34	0.61							
ERL 4221	29.00							
Limonene Dioxide	30.69							
FC-509	3.00							
FC-510	1.00							
PCP 291								
Red Iron Oxide	7.14	D Bulb 300 w/in 12.5 ft/min	100 %	100 %	100 %	4H	4H	F
Halox BW-111	11.25	5 Passes for Zn-Phos. 3 Passes for Steel	100 %	100 %	100 %	4H	4H	F
Fibrene C-400	21.51							
Bentone 34	0.61							
ERL 4221	29.00							
Limonene Dioxide	30.69							
FC-508	4.00							
FC-510	1.00							

are dependent on the formulation as well as the film's substrate. All formulations had excellent adhesion to all substrates.

5.2 Evaluation of Paints

The performance characteristics of the paints were determined by ASTM testing methods and were compared with standard conventional paints presently being used at Westinghouse plants.

In the preliminary screening, paints were applied on three different substrates, iron phosphated, zinc phosphated and untreated cold rolled steel. The paints were applied to a thickness of 1 mil with a Bird applicator. Then the coated panels were passed through a UV processor and were irradiated.

Several tests were run to evaluate the paint. First, the adhesion was determined using the crosshatch method which consists of scoring the surface to bare metal using a single edge razor blade forming 100 squares (1/16" on a side), Table 63. Scotch tape is applied over the crosshatched area and then removed. Since there is little market for non-adherent paints, 100% adhesion is desirable. Hardness was determined by cutting the films with lead pencils of graduated hardnesses. A pencil hardness of 2H or greater is desired (Table 63).

UV curable paints displayed good adhesion and flexibility. More intensive testing included microknife adhesion, Taber abrasion, conical mandrel, and impact resistance, shown in Table 64. In the direct and reverse impact tests using a die with a hemispherical head which fits into a hollow cylindrical anvil. After a panel is placed between the die and the anvil, the weight is lifted and then dropped to cause rapid deformation of the coating and the substrate. Direct and reverse impact test results are listed in Table 64. These tests evaluate the elongation and flexibility of the coating. The viscosity has been adjusted to give

TABLE 63

PROPERTIES OF UV CURABLE AND CONVENTIONAL
PIGMENTED COATINGS

Formulation	Pencil Hardness Test			Crosshatch Test			Microknife Test		
	ZnPhos	FePhos	Steel	ZnPhos	FePhos	Steel	ZnPhos	FePhos	Steel
PCP290 (Primer) P282 (Topcoat)	<6B	2B	4B	100%	100%	100%	8 Mils	6 Mils	8 Mils
PCP290 (Primer) CP246 (Topcoat)	2B	4B	<6B	100%	98% Heavy lines	98%	10 Mils	8 Mils	12 Mils
5-305 (Primer) P282 (Topcoat)	H	2H	F	100%	100%	100%	4 Mils	4 Mils	8 Mils
5-305 (Primer) CP246 (Topcoat)	HB	B	2B	100%	100%	100%	6 Mils	8 Mils	8 Mils
PCP290 (Primer) PCB T-12 (Topcoat)	4B	4B	6B	99%	99%	100%	8 Mils	6 Mils	8 Mils
PCP290 (Primer) SWG-T-14 (Topcoat)	4B	B	3B	100%	100%	100%	8 Mils	9 Mils	8 Mils
5-305 (Primer) PCBT-12 (Topcoat)	HB	HB	HB	100%	100%	100%	4 Mils	4 Mils	4 Mils
5-305 (Primer) SWG-T-14 (Topcoat)	F	2H	F	100%	100%	100%	12 Mils	10 Mils	12 Mils

TABLE 64

PROPERTIES OF UV CURABLE AND CONVENTIONAL
PIGMENTED COATINGS

Formulation	Direct Impact Test			Reverse Impact Test			Conical Mandrel		
	Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel	Zn Phos	Fe Phos	Steel
PCP290 (Primer) P282 (Topcoat)	60	80	60	20	5	30	Passed	Passed	Passed
PCP290 (Primer) CP246 (Topcoat)	40	30	30	5	1	1	Passed	Passed	Passed
5-305 (Primer) P282 (Topcoat)	60	80	60	10	10	10	Passed	Passed	Passed
5-305 (Primer) CP246 (Topcoat)	80	80	50	40	20	10	Passed	Passed	Passed
PCP290 (Primer) PCBT-12 (Topcoat)	30	30	40	10	10	10	Passed	Passed	Passed
PCP290 (Primer) SWG-T-14 (Topcoat)	30	110	30	20	5	1	Passed	Passed	Passed
5-305 (Primer) PCBT-12 (Topcoat)	50	80	40	10	20	10	Passed	Passed	Passed
5-305 (Primer) SWG-T-14 (Topcoat)	100	140	90	10	10	10	Passed	Passed	Passed

coatings capable of being applied with conventional spray equipment. The paints were sprayed onto panels and were subjected to further testings: salt spray (Table 65), humidity (Table 66), Weather-o-meter (Table 67), and Fade-o-meter exposure (Table 68). Shelf life and stability also were monitored by viscosity changes. UV curable paints were somewhat less resistant than conventional systems on the salt spray and humidity tests.

The goal of this program was to show the feasibility of using UV radiation as a replacement for natural gas in paint curing; it was not to develop paints for specific products. In order to determine the feasibility, UV curing primers and topcoats were evaluated in finish systems (primer plus topcoat) and compared with standard finish systems used by Westinghouse. Data shown in Tables 65 to 68 are the results of accelerated aging tests and must be interpreted in terms of actual coating performance. Westinghouse finish systems B5-305 primer topcoated with PCB T-12 enamel and B5-305 primer topcoated with SWG T-14 enamel, which are used on outdoor electrical apparatus, have service lives of 20 to 30 years.

Although further reformulation work must be done on the UV curable primers and topcoats developed in this program in order to make them meet performance requirements of specific manufactured products, they do show great promise.

Evaluations of primers without topcoats are generally not conducted because they are rarely used alone. Therefore, the UV curing cationic photoinitiated epoxy primer PCP 290 was evaluated in finish systems and compared with standard Westinghouse systems. A finish system consisting of PCP 290 topcoated with Westinghouse enamel PCB T-12 performed nearly as well on the tests as the standard finish system Westinghouse B5-305 primer topcoated with PCB T-12. The corrosion resistance (Table 65) of the PCP 290 primed system was not as good as the B5-305 primed system while the

TABLE 65-SALT SPRAY (Sheet 1 of 2)

	Zn Phos	Fe Phos	Steel
PCP 290 (Primer) P 282 (Top-Coat)			
7/7/81 Initial			
7/8/81 24 Hrs.	OK	OK	OK
7/9/81 48 Hrs.	OK	OK	OK
7/10/81 100 Hrs.	OK	OK	OK
7/14/81 200 Hrs.	OK	OK	OK
7/20/81 300 Hrs.	OK	OK *	OK Few Blisters
7/24/81 400 Hrs.	OK	1/4" Creepage *	OK Few Blisters
7/28/81 500 Hrs.	OK	1/4" Creepage *	OK *
8/3/81 600 Hrs.	OK	1/4" Creepage *	OK *
8/7/81 700 Hrs.	OK	1/4" Creepage *	OK *
8/11/81 800 Hrs.	OK	1/4" Creepage *	OK *
8/17/81 900 Hrs.	OK *	1/4" Creepage *	OK *
8/21/81 1000 Hrs.	OK *	1/4" Creepage *	OK *
PCP 290 (Primer) CP 246 (Top-Coat)			
7/7/81 Initial			
7/8/81 24 Hrs.	OK Wrinkles	OK Wrinkles	OK Wrinkles
7/9/81 48 Hrs.	OK Wrinkles	OK Wrinkles	OK Wrinkles
7/10/81 100 Hrs.	OK Wrinkles	OK Wrinkles	OK Wrinkles
7/14/81 200 Hrs.	OK Wrinkles	OK Wrinkles	OK Wrinkles
7/20/81 300 Hrs.	OK Wrinkles	OK Wrinkles	OK Wrinkles
7/24/81 400 Hrs.	OK Wrinkles	1/8" Creepage Wrinkles	OK Wrinkles
7/28/81 500 Hrs.	OK Wrinkles	1/8" Creepage Wrinkles	OK Wrinkles
8/3/81 600 Hrs.	OK Wrinkles	1/8" Creepage Wrinkles	OK Wrinkles
8/7/81 700 Hrs.	OK Wrinkles	1/8" Creepage Wrinkles	OK Wrinkles
8/11/81 800 Hrs.	OK Wrinkles	1/8" Creepage Wrinkles	OK Wrinkles
8/17/81 900 Hrs.	OK Wrinkles	1/8" Creepage Wrinkles	OK Wrinkles
8/21/81 1000 Hrs.	OK Wrinkles, Few Blisters	1/8" Creepage Wrinkles	OK Wrinkles
S-305 (Primer) P 282 (Top-Coat)			
7/7/81 Initial			
7/8/81 24 Hrs.	OK	OK	OK
7/9/81 48 Hrs.	OK	OK	OK
7/10/81 100 Hrs.	OK	OK	OK
7/14/81 200 Hrs.	OK	OK	OK
7/20/81 300 Hrs.	OK	OK	OK
7/24/81 400 Hrs.	OK	OK	1/32" Creepage
7/28/81 500 Hrs.	OK	OK	1/32" Creepage
8/3/81 600 Hrs.	OK	OK	1/32" Creepage
8/7/81 700 Hrs.	OK	OK	1/32" Creepage
8/11/81 800 Hrs.	OK	OK	1/32" Creepage
8/17/81 900 Hrs.	OK	1/32" Creepage	1/32" Creepage
8/21/81 1000 Hrs.	OK	1/32" Creepage	1/32" Creepage
S-305 (Primer) CP 246 (Top-Coat)			
7/7/81 Initial			
7/8/81 24 Hrs.	OK	OK	OK
7/9/81 48 Hrs.	OK	OK	OK
7/10/81 100 Hrs.	OK	OK	OK
7/14/81 200 Hrs.	OK	OK	OK
7/20/81 300 Hrs.	OK	OK	OK
7/24/81 400 Hrs.	OK	OK	OK
7/28/81 500 Hrs.	OK	OK	1/32" Creepage
8/3/81 600 Hrs.	OK	OK	1/32" Creepage
8/7/81 700 Hrs.	OK	OK	1/32" Creepage
8/11/81 800 Hrs.	OK	OK	1/32" Creepage
8/17/81 900 Hrs.	OK	1/32" Creepage	1/32" Creepage
8/21/81 1000 Hrs.	OK	1/32" Creepage	1/32" Creepage

* Blisters Throughout the Panel

TABLE 65—SALT SPRAY (Sheet 2 of 2)

		Zn Phos	Fe Phos	Steel
② 5-305 (Primer) PCB T-12 (Top-Coat)				
6/8/81 Initial				
6/9/81 24 Hrs.	OK	OK	OK	
6/10/81 48 Hrs.	OK	OK	OK	
6/12/81 100 Hrs.	OK	OK	OK	
6/16/81 200 Hrs.	OK	OK	OK	
6/22/81 300 Hrs.	OK	OK	OK	1/16" Creepage
6/26/81 400 Hrs.	OK	1/8" Creepage	1/8" Creepage	1/8" Creepage
6/30/81 500 Hrs.	1/8" Creepage	1/8" Creepage	1/8" Creepage	1/8" Creepage
7/7/81 600 Hrs.	1/8" Creepage	1/8" Creepage	1/8" Creepage	1/8" Creepage
7/13/81 700 Hrs.	1/8" Creepage	1/8" Creepage	1/8" Creepage	1/8" Creepage
7/17/81 800 Hrs.	1/8" Creepage	1/8" Creepage	1/8" Creepage	1/8" Creepage
7/21/81 900 Hrs.	1/8" Creepage	1/4" Creepage *	1/8" Creepage *	1/8" Creepage *
7/27/81 1000 Hrs.	1/8" Creepage	1/4" Creepage *	1/8" Creepage *	1/8" Creepage *
② 5-305 (Primer) SWG T-14 (Top-Coat)				
6/8/81 Initial				
6/9/81 24 Hrs.	OK	OK	OK	
6/10/81 48 Hrs.	OK	OK	OK	
6/12/81 100 Hrs.	OK	OK	OK	
6/16/81 200 Hrs.	OK	OK	OK	1/16" Creepage
6/22/81 300 Hrs.	OK	OK	OK	1/16" Creepage
6/26/81 400 Hrs.	OK	OK	OK	1/16" Creepage
6/30/81 500 Hrs.	OK	OK	OK	1/8" Creepage
7/7/81 600 Hrs.	OK	OK	OK	1/8" Creepage
7/13/81 700 Hrs.	OK	OK	OK	1/8" Creepage
7/17/81 800 Hrs.	OK	OK	OK	1/8" Creepage
7/21/81 900 Hrs.	OK	OK	OK	1/8" Creepage
7/27/81 1000 Hrs.	OK	OK	OK	1/8" Creepage
PCP 290 (Primer) PCB T-12 (Top-Coat)				
6/8/81 Initial				
6/9/81 24 Hrs.	OK	OK	OK	
6/10/81 48 Hrs.	OK	OK	OK	
6/12/81 100 Hrs.	OK	OK	OK	1/16" Creepage
6/16/81 200 Hrs.	OK	OK	OK	1/8" Creepage
6/22/81 300 Hrs.	1/8" Creepage	1/8" Creepage	1/4" Creepage	1/4" Creepage
6/26/81 400 Hrs.	1/8" Creepage	1/4" Creepage	1/4" Creepage	*
6/30/81 500 Hrs.	1/4" Creepage	1/4" Creepage	1/4" Creepage	*
7/7/81 600 Hrs.	1/4" Creepage	1/4" Creepage	1/4" Creepage	*
7/13/81 700 Hrs.	1/4" Creepage	Creepage Throughout	Creepage Throughout	*
7/17/81 800 Hrs.	1/4" Creepage *	Creepage Throughout	Creepage Throughout	*
7/21/81 900 Hrs.	1/4" Creepage *	Creepage Throughout	Creepage Throughout	*
7/27/81 1000 Hrs.	1/4" Creepage *	Creepage Throughout	Creepage Throughout	*
PCP 290 (Primer) SWG T-14 (Top-Coat)				
6/8/81 Initial				
6/9/81 24 Hrs.	OK	OK	OK	
6/10/81 48 Hrs.	OK	OK	OK	
6/12/81 100 Hrs.	OK	OK	OK	
6/16/81 200 Hrs.	OK	OK	OK	
6/22/81 300 Hrs.	OK	OK	OK	1/4" Creepage
6/26/81 400 Hrs.	OK *	1/8" Creepage	1/4" Creepage	1/4" Creepage
6/30/81 500 Hrs.	OK *	1/8" Creepage	1/4" Creepage	1/4" Creepage *
7/7/81 600 Hrs.	OK *	1/8" Creepage	1/4" Creepage	1/4" Creepage *
7/13/81 700 Hrs.	OK *	1/4" Creepage *	1/2" Creepage *	1/2" Creepage *
7/17/81 800 Hrs.	OK *	1/4" Creepage *	1/2" Creepage *	1/2" Creepage *
7/21/81 900 Hrs.	OK *	1/4" Creepage *	1/2" Creepage *	1/2" Creepage *
7/27/81 1000 Hrs.	OK *	1/4" Creepage *	1/2" Creepage *	1/2" Creepage *

* Blisters Throughout

TABLE 66—HUMIDITY (Sheet 1 of 2)

		Zn Phos	Fe Phos	Steel
PCP 290 (Primer) P 282 (Top-Coat)				
7/7/81 Initial		OK	OK	OK
7/8/81 24 Hrs.		OK	OK	OK
7/9/81 48 Hrs.		OK	OK	OK
7/10/81 100 Hrs.		OK	OK	#6 Medium Dense
7/14/81 200 Hrs.		OK	OK	#6 Medium Dense
7/20/81 300 Hrs.		OK	OK	#6 Dense
7/24/81 400 Hrs.		OK	#4 Medium Dense	#6 Dense
7/28/81 500 Hrs.		OK	#4 Medium Dense	#6 Dense
8/3/81 600 Hrs.		OK	#4 Medium Dense	#6 Dense
8/7/81 700 Hrs.		OK	#4 Medium Dense	#6 Dense
8/11/81 800 Hrs.		OK	#4 Medium Dense	#6 Dense
8/17/81 900 Hrs.		OK	#4 Medium Dense	#6 Dense
8/21/81 1000 Hrs.		OK	#4 Medium Dense	#6 Dense
PCP 290 CP 246				
7/7/81 Initial				
7/8/81 24 Hrs.		Wrinkles *	Wrinkles *	Wrinkles *
7/9/81 48 Hrs.		Wrinkles *	Wrinkles *	Wrinkles *
7/10/81 100 Hrs.		Wrinkles *	Wrinkles *	Wrinkles *
7/14/81 200 Hrs.		Wrinkles *	Wrinkles *	Wrinkles *
7/20/81 300 Hrs.		Wrinkles *	Wrinkles *	Wrinkles *
7/24/81 400 Hrs.		Wrinkles *	Wrinkles * #4 Few	Wrinkles *
7/28/81 500 Hrs.		Wrinkles *	Wrinkles * #4 Few	Wrinkles *
8/3/81 600 Hrs.		Wrinkles *	Wrinkles * #4 Few	Wrinkles *
8/7/81 700 Hrs.		Wrinkles *	Wrinkles * #4 Few	Wrinkles *
8/11/81 800 Hrs.		Wrinkles *	Wrinkles * #4 Few	Wrinkles *
8/17/81 900 Hrs.		Wrinkles *	Wrinkles * #4 Few	Wrinkles *
8/21/81 1000 Hrs.		Wrinkles *	Wrinkles * #4 Few	Wrinkles *
5-305 (Primer) P 282 (Top-Coat)				
7/7/81 Initial				
7/8/81 24 Hrs.		OK	OK	OK
7/9/81 48 Hrs.		OK	OK	OK
7/10/81 100 Hrs.		#8 Few	#8 Few	#8 Few
7/14/81 200 Hrs.		#8 Few	#8 Few	#8 Few
7/20/81 300 Hrs.		#8 Few	#8 Few	#8 Few
7/24/81 400 Hrs.		#8 Few	#8 Few	#8 Few
7/28/81 500 Hrs.		#8 Few	#8 Few	#8 Few
8/3/81 600 Hrs.		#8 Few	#8 Few	#8 Few
8/7/81 700 Hrs.		#8 Few	#8 Few	#8 Few
8/11/81 800 Hrs.		#8 Few	#8 Few	#8 Few
8/17/81 900 Hrs.		#8 Few	#8 Few	#8 Few
8/21/81 1000 Hrs.		#8 Few	#8 Few	#8 Few
5-305 (Primer) CP 246 (Top-Coat)				
7/7/81 Initial				
7/8/81 24 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
7/9/81 48 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
7/10/81 100 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
7/14/81 200 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
7/20/81 300 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
7/24/81 400 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
7/28/81 500 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
8/3/81 600 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
8/7/81 700 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
8/11/81 800 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
8/17/81 900 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense
8/21/81 1000 Hrs.		#4 Dense	#6 Medium Dense	#6 Medium Dense

* Throughout the Panel

TABLE 66—HUMIDITY (Sheet 2 of 2)

		Zn Phos	Fe Phos	Steel
5-305 (Primer)				
PCB T-12 (Top-Coat)				
6/15/81	Initial			
6/16/81	24 Hrs.	#8 Dense	#8 Dense	#6 Few
6/17/81	48 Hrs.	#8 Dense	#8 Dense	#6 Few
6/19/81	100 Hrs.	#8 Dense	#8 Dense	#6 Few
6/23/81	200 Hrs.	#8 Dense	#8 Dense	#6 Few
6/29/81	300 Hrs.	#8 Dense	#8 Dense	#6 Few
7/2/81	400 Hrs.	#8 Dense	#8 Dense	#6 Few
7/7/81	500 Hrs.	#8 Dense	#8 Dense	#6 Few
7/13/81	600 Hrs.	#8 Dense	#8 Dense	#6 Few
7/17/81	700 Hrs.	#8 Dense	#8 Dense	#6 Few
7/21/81	800 Hrs.	#8 Dense	#8 Dense	#6 Few
7/27/81	900 Hrs.	#8 Dense	#8 Dense	#6 Few
7/31/81	1000 Hrs.	#8 Dense	#8 Dense	#6 Few
5-305 (Primer)				
SWG T-14 (Top-Coat)				
6/15/81	Initial			
6/16/81	24 Hrs.	OK	#8 Medium Dense	OK
6/17/81	48 Hrs.	OK	#8 Medium Dense	OK
6/19/81	100 Hrs.	OK	#8 Medium Dense	OK
6/23/81	200 Hrs.	OK	#8 Medium Dense	OK
6/29/81	300 Hrs.	OK	#8 Medium Dense	OK
7/2/81	400 Hrs.	OK	#8 Medium Dense	OK
7/7/81	500 Hrs.	OK	#8 Medium Dense	OK
7/13/81	600 Hrs.	OK	#8 Medium Dense	OK
7/17/81	700 Hrs.	OK	#8 Medium Dense	OK
7/21/81	800 Hrs.	OK	#8 Medium Dense	OK
7/27/81	900 Hrs.	#8 Dense	#8 Dense	#8 Few
7/31/81	1000 Hrs.	#8 Dense	#8 Dense	#8 Few
PCP 290 (Primer)				
PCB T-12 (Top-Coat)				
6/15/81	Initial			
6/16/81	24 Hrs.	OK	OK	OK
6/17/81	48 Hrs.	OK	OK	OK
6/19/81	100 Hrs.	OK	OK	OK
6/23/81	200 Hrs.	OK	OK	OK
6/29/81	300 Hrs.	OK	OK	OK
7/2/81	400 Hrs.	OK	OK	OK
7/7/81	500 Hrs.	#2 Few	#8 Dense	#8 Dense
7/13/81	600 Hrs.	#2 Few	#8 Dense	#8 Dense
7/17/81	700 Hrs.	#2 Few	#8 Dense	#8 Dense
7/21/81	800 Hrs.	#2 Few	#8 Dense	#8 Dense
7/27/81	900 Hrs.	#2 Few	#8 Dense	#8 Dense
7/31/81	1000 Hrs.	#2 Few	#8 Dense	#8 Dense
PCP 290 (Primer)				
SWG T-14 (Top-Coat)				
6/15/81	Initial			
6/16/81	24 Hrs.	OK	OK	OK
6/17/81	48 Hrs.	OK	OK	OK
6/19/81	100 Hrs.	OK	OK	OK
6/23/81	200 Hrs.	OK	OK	#2 Medium Dense
6/29/81	300 Hrs.	OK	OK	#2 Medium Dense
7/2/81	400 Hrs.	#8 Medium Dense	#4 Few	#2 Medium Dense
7/7/81	500 Hrs.	#8 Medium Dense	#4 Few	#2 Dense
7/13/81	600 Hrs.	#8 Medium Dense	#4 Few	#2 Dense
7/17/81	700 Hrs.	#8 Medium Dense	#4 Dense	#2 Dense
7/21/81	800 Hrs.	#8 Medium Dense	#4 Dense	#2 Dense
7/27/81	900 Hrs.	#8 Medium Dense	#4 Dense	#2 Dense
7/31/81	1000 Hrs.	#8 Medium Dense	#4 Dense	#2 Dense

TABLE 67-WEATHEROMETER (Sheet 1 of 2)

	Zn Phos	Fe Phos	Steel	
			Initial	1000 Hrs.
5-305 (Primer) CP 246 (Top-Coat)				
7/8/81 Initial	Standard #10	76	Standard #10	57
7/13/81 100 Hrs.	Standard #10	65	Standard #10	40
7/17/81 200 Hrs.	Standard #10	25	Standard #10	21
7/21/81 300 Hrs.	Standard #10	21	Standard #10	17
7/27/81 400 Hrs.	Standard #10	16	Standard #10	12
7/31/81 500 Hrs.	Standard #10	9	Standard #10	8
8/4/81 600 Hrs.	Standard #10	8	Standard #10	6.5
8/10/81 700 Hrs.	Standard #10	5	Standard #10	4.5
8/14/81 800 Hrs.	Standard #10	4.5	Standard #10	4
8/18/81 900 Hrs.	Standard #10	3	Standard #10	2
8/24/81 1000 Hrs.	Standard #10	2	Standard #10	1.5
5-305 (Primer) P282 (Top-Coat)				
7/8/81 Initial	Standard #10	11	Standard #10	9.5
7/13/81 100 Hrs.	Standard #10	11	Standard #10	8
7/17/81 200 Hrs.	Standard #10	2.5	Standard #10	2
7/21/81 300 Hrs.	Standard #10	2.5	Standard #10	2.5
7/27/81 400 Hrs.	Standard #10	1.5	Standard #10	2
7/31/81 500 Hrs.	Standard #10	1.5	Standard #10	2
8/4/81 600 Hrs.	Standard #10	2	Standard #10	1.5
8/10/81 700 Hrs.	Standard #10	2.5	Standard #10	2
8/14/81 800 Hrs.	Standard #10	1.5	Standard #10	1.5
8/18/81 900 Hrs.	Standard #10	1.5	Standard #10	1
8/24/81 1000 Hrs.	Standard #10	1	Standard #10	1.5
PCP 290 (Primer) CP 246 (Top-Coat)				
7/8/81 Initial	Standard #10	7	Standard #10	22.5
7/13/81 100 Hrs.	Standard #10	10	Standard #10	24
7/17/81 200 Hrs.	Standard #10	7	Standard #10	10.5
7/21/81 300 Hrs.	Standard #10	5	Standard #10	9
7/27/81 400 Hrs.	Standard #10	5.5	Standard #10	6
7/31/81 500 Hrs.	Standard #10	4	Standard #10	5
8/4/81 600 Hrs.	Standard #10	3.5	Standard #10	4
8/10/81 700 Hrs.	Standard #10	3	Standard #10	3
8/14/81 800 Hrs.	Standard #10	2.5	Standard #10	2.5
8/18/81 900 Hrs.	Standard #10	2	Standard #10	2
8/24/81 1000 Hrs.	Standard #10	1.5	Standard #10	1.5
PCP 290 P 282				
7/8/81 Initial	Standard #10	10.5	Standard #10	17
7/13/81 100 Hrs.	Standard #10	34	Standard #10	27
7/17/81 200 Hrs.	Standard #10	20	Standard #10	14
7/21/81 300 Hrs.	Standard #10	15	Standard #10	9
7/27/81 400 Hrs.	Standard #10	10	Standard #10	8.5
7/31/81 500 Hrs.	Standard #10	6.5	Standard #10	4
8/4/81 600 Hrs.	Standard #10	6	Standard #10	3.5
8/10/81 700 Hrs.	Standard #10	5.5	Standard #10	3
8/14/81 800 Hrs.	Standard #10	3.5	Standard #10	2.5
8/18/81 900 Hrs.	Standard #10	1.5	Standard #10	1.5
8/24/81 1000 Hrs.	Standard #10	1	Standard #10	0

TABLE 67-WEATHEROMETER (Sheet 2 of 2)

	Zn Phos	Fe Phos	Steel
5-305 (Primer) T-12 (Top-Coat)			
6/9/81 Initial Gloss	Standard #40 41.5	Standard #40 39.5	Standard #40 34.5
6/15/81 100 Hrs.	Standard #10 16	Standard #10 14.5	Standard #10 15
6/19/81 200 Hrs.	Standard #10 15	Standard #10 14	Standard #10 13.5
6/24/81 300 Hrs.	Standard #10 15	Standard #10 13	Standard #10 12
6/29/81 400 Hrs.	Standard #10 15	Standard #10 15	Standard #10 14
7/2/81 500 Hrs.	Standard #10 12	Standard #10 13	Standard #10 13
7/7/81 600 Hrs.	Standard #10 13	Standard #10 13.5	Standard #10 11
7/13/81 700 Hrs.	Standard #10 10	Standard #10 10	Standard #10 13
7/17/81 800 Hrs.	Standard #10 8	Standard #10 8.5	Standard #10 12
7/21/81 900 Hrs.	Standard #10 5.5	Standard #10 4.5	Standard #10 7
7/27/81 1000 Hrs.	Standard #10 2	Standard #10 1.5	Standard #10 3.5
5-305 (Primer) T-14 (Top-Coat)			
6/9/81 Initial Gloss	Standard #10 13.5	Standard #10 10.5	Standard #10 10
6/15/81 100 Hrs.	Standard #10 10	Standard #10 8	Standard #10 8
6/19/81 200 Hrs.	Standard #10 9	Standard #10 7	Standard #10 6.5
6/24/81 300 Hrs.	Standard #10 9	Standard #10 7	Standard #10 6.5
6/29/81 400 Hrs.	Standard #10 8.5	Standard #10 6.5	Standard #10 7
7/2/81 500 Hrs.	Standard #10 6.5	Standard #10 6.5	Standard #10 6.5
7/7/81 600 Hrs.	Standard #10 6.5	Standard #10 6.5	Standard #10 6
7/13/81 700 Hrs.	Standard #10 7	Standard #10 5.5	Standard #10 6
7/17/81 800 Hrs.	Standard #10 6.5	Standard #10 3	Standard #10 3
7/21/81 900 Hrs.	Standard #10 4	Standard #10 2.5	Standard #10 2
7/27/81 1000 Hrs.	Standard #10 2.5	Standard #10 2	Standard #10 1.5
PCP 290 (Primer) PCB T-12 (Top-Coat)			
6/9/81 Initial Gloss	Standard #60 70	Standard #60 65	Standard #60 72
6/15/81 100 Hrs.	Standard #60 64.5	Standard #50 51	Standard #60 70
6/19/81 200 Hrs.	Standard #60 56	Standard #50 46	Standard #60 58.5
6/24/81 300 Hrs.	Standard #40 37	Standard #40 41	Standard #40 44
6/29/81 400 Hrs.	Standard #40 28.5	Standard #40 32	Standard #40 34
7/2/81 500 Hrs.	Standard #10 20	Standard #10 15	Standard #10 22
7/7/81 600 Hrs.	Standard #10 21.5	Standard #10 21	Standard #10 25
7/13/81 700 Hrs.	Standard #10 11	Standard #10 9	Standard #10 11.5
7/17/81 800 Hrs.	Standard #10 9	Standard #10 6	Standard #10 9.5
7/21/81 900 Hrs.	Standard #10 6.5	Standard #10 4.5	Standard #10 6.0
7/27/81 1000 Hrs.	Standard #10 5.0	Standard #10 2.0	Standard #10 4.0
PCP 290 (Primer) SWG T-14 (Top-Coat)			
6/9/81 Initial Gloss	Standard #10 15	Standard #10 11	Standard #10 13.5
6/15/81 100 Hrs.	Standard #10 22	Standard #40 28	Standard #10 22
6/19/81 200 Hrs.	Standard #40 20	Standard #40 32	Standard #10 24
6/24/81 300 Hrs.	Standard #10 19	Standard #10 22.5	Standard #10 19
6/29/81 400 Hrs.	Standard #10 15	Standard #10 16.5	Standard #10 15
7/2/81 500 Hrs.	Standard #10 12	Standard #10 12	Standard #10 10
7/7/81 600 Hrs.	Standard #10 12	Standard #10 12	Standard #10 10
7/13/81 700 Hrs.	Standard #10 9	Standard #10 9.5	Standard #10 7.5
7/17/81 800 Hrs.	Standard #10 6.5	Standard #10 5.5	Standard #10 5.5
7/21/81 900 Hrs.	Standard #10 6.0	Standard #10 4.5	Standard #10 5.0
7/27/81 1000 Hrs.	Standard #10 4.0	Standard #10 2.0	Standard #10 4.0

TABLE 68-FADE-O-METER

18-6147-11

	Zn Phos	Fe Phos	Steel
5-305 (Primer) CP 246 (Top-Coat)			
8/13/81 Initial	x y z 75.66 72.63 82.18 YI = 7.40	x y z 70.36 67.30 78.66 YI = 3.57	x y z 74.77 71.68 81.72 YI = 6.56
8/14/81 24 Hrs.	x y z 74.33 71.70 83.20 YI = 4.54	x y z 71.12 68.54 80.85 YI = 2.54	x y z 75.16 72.50 83.25 YI = 5.78
8/18/81 48 Hrs.	x y z 76.53 73.77 84.72 YI = 5.76	x y z 69.98 67.35 79.86 YI = 1.93	x y z 74.45 71.63 82.98 YI = 4.71
8/19/81 72 Hrs.	x y z 74.33 71.80 84.80 YI = 4.65	x y z 69.12 66.55 79.60 YI = 0.68	x y z 74.82 72.22 83.79 YI = 4.55
8/20/81 100 Hrs.	x y z 75.36 72.60 84.59 YI = 4.05	x y z 69.58 67.06 80.36 YI = 0.65	x y z 75.30 72.56 84.26 YI = 4.41
5-305 (Primer) P 282 (Top-Coat)			
8/13/81 Initial	x y z 70.12 67.12 82.71 YI = -2.78	x y z 70.64 68.10 84.38 YI = -3.73	x y z 70.70 68.28 84.90 YI = -3.52
8/14/81 24 Hrs.	x y z 69.70 67.38 84.34 YI = -4.61	x y z 73.42 71.04 88.80 YI = -4.64	x y z 71.23 68.88 86.14 YI = -4.69
8/18/81 48 Hrs.	x y z 68.40 65.90 82.79 YI = -5.26	x y z 73.04 70.56 88.31 YI = -4.79	x y z 71.50 69.06 86.25 YI = -4.52
8/19/81 72 Hrs.	x y z 69.76 67.31 84.70 YI = -5.48	x y z 72.70 70.28 88.00 YI = -4.64	x y z 71.68 69.30 86.42 YI = -4.53
8/20/81 100 Hrs.	x y z 69.72 67.31 84.76 YI = -5.56	x y z 73.22 70.80 88.79 YI = -5.06	x y z 71.52 69.14 86.52 YI = -4.78
PCP 290 (Primer) CP 246 (Top-Coat)			
8/13/81 Initial	x y z 71.60 69.30 80.53 YI = 4.32	x y z 72.78 70.32 80.37 YI = 6.30	x y z 58.52 57.42 69.50 YI = -0.60
8/14/81 24 Hrs.	x y z 70.42 68.57 81.27 YI = 2.06	x y z 72.85 70.81 83.26 YI = 2.99	x y z 58.79 57.78 70.22 YI = 1.12
8/18/81 48 Hrs.	x y z 70.07 68.20 80.80 YI = 2.04	x y z 73.35 71.11 82.30 YI = 4.66	x y z 59.89 58.58 70.69 YI = -0.22
8/19/81 72 Hrs.	x y z 70.37 68.40 81.51 YI = 1.28	x y z 73.56 71.30 82.80 YI = 4.46	x y z 62.48 61.07 73.79 YI = -0.56
8/20/81 100 Hrs.	x y z 70.84 68.83 82.09 YI = 1.24	x y z 73.64 71.36 82.84 YI = 4.50	x y z 60.14 58.90 71.35 YI = -0.69
PCP 290 P 282			
8/13/81 Initial	x y z 65.68 63.68 75.83 YI = 1.40	x y z 68.51 66.15 78.00 YI = 2.60	x y z 77.84 75.58 84.42 YI = 6.08
8/14/81 24 Hrs.	x y z 67.52 66.02 81.09 YI = -2.44	x y z 69.92 68.32 83.80 YI = 2.28	x y z 79.33 77.20 90.88 YI = 2.67
8/18/81 48 Hrs.	x y z 67.82 66.14 81.09 YI = -2.20	x y z 70.10 68.23 83.88 YI = -2.56	x y z 79.14 76.56 90.24 YI = 2.66
8/19/81 72 Hrs.	x y z 66.56 65.04 79.82 YI = -2.54	x y z 69.36 67.70 83.49 YI = -2.94	x y z 78.56 76.37 89.72 YI = 3.08
8/20/81 100 Hrs.	x y z 66.28 64.80 79.64 YI = -2.50	x y z 71.22 69.37 85.17 YI = -2.58	x y z 78.35 76.00 89.90 YI = 2.24

PCP 290 system was superior in humidity resistance (Table 66). The other properties, adhesion, hardness, flexibility, and impact resistance were similar for both systems. Although it was as good as the PCP 290/PCB T-12 finish system, when PCP 290 was used as a primer in another finish system topcoated with Westinghouse enamel SWG T-14 the overall performance was not as good as the standard Westinghouse finish system SWG T-14/B5-305. The corrosion and humidity resistance of the PCP 290 primed finish system was inferior to the B5-305 system, while the adhesion, hardness, and flexibility and impact resistances were equivalent. Reformulation of the primer PCP 290 by the addition of special corrosion inhibiting pigments would enhance the corrosion and humidity resistance.

UV curable free radical photoinitiated acrylated polyurethane and cationic photoinitiated epoxy enamels were evaluated by using them as topcoats in finish systems over conventional and UV curable primers. The finish system using acrylated polyurethane P 282 enamel over PCP 290 primer had corrosion and humidity resistance as well as adhesion, hardness, flexibility, and impact resistance properties which were equivalent to the standard Westinghouse finish system PCB T-12/B5-305. UV curable cationic photoinitiated epoxy CP 246 was tested in finish systems as a topcoat over PCP 290 and B5-305 primers. CP 246 was wrinkled with PCP 290. However, when applied over B5-305, the resulting finish system performed extremely well in corrosion resistance but performed poorly in humidity resistance. On the other hand, the CP 246/B5-305 finish system was equivalent in humidity resistance to PCB T-12/B5-305 standard finish system.

Paint chemists can modify base resins, pigments, monomers and additives to tailor these UV curable primers and enamels for use as finishes on specific products.

5.3 Application and Curing of Paints

The temperature of the substrate during cure was monitored and the effect of temperature on the coatings evaluated. At 200 ft./min. and 200 W/in., the temperature rise from irradiation with a medium pressure

mercury lamp is only a few degrees (Table 69). Studies were made comparing the temperature rise of the substrate when the lower blower of the UV processor was not on (Figure 9) to the temperature rise during cure when the lower blower was on (Fig. 10). When the lower blower is not on, one H bulb alone will not raise the temperature above 70 degrees C in 15 passes. An H bulb a D bulb together will raise the temperature beyond 70 degrees C only after 3 passes. When the lower blower is on, neither one H bulb alone, one D bulb, nor both together will raise the temperature to 70 degrees C in 15 passes (about 50 seconds for one lamp). Most of the UV curable paints were cured within 3 to 30 seconds.

The effects of temperature upon the properties of pigmented coatings are described in Table 70. A V bulb alone proved ineffective in curing formulation P72D. However, H and D bulbs, alone and in combination, produced cured, adherent coatings.

The coated substrate was irradiated under a liquid nitrogen-cooled quartz filter to eliminate IR radiation. The paint cured with 7 seconds using an H bulb. The UV curing of the paints appears to be largely independent of temperature.

Previously, three-dimensional objects coated with clear and lightly pigmented materials had been cured by UV radiation (Fusion Systems⁹, Coors³², American Production Machinery³³, Tioxide International²⁻⁶, UCB¹⁶⁻¹⁹, Metal Box Co.¹⁰, and Ciba-Geigy^{14,15}). UV curable paints, however, presented a somewhat more complicated problem.

After demonstrating the ability to cure paints on flat panels using UV radiation, the problem of curing them on three-dimensional objects was addressed. Since most of the commercial UV coating lines are used for finishing flat or cylindrical objects, the radiation sources can be focused and placed rather close to the ware, since the light intensity falls off rapidly with increasing lateral distance (Figure 11). On the other hand,

TABLE 69
TEMPERATURE INCREASES RESULTING FROM EXPOSURE TO UV SOURCE

<u>Number of Passes Under UV Source</u>	<u>Initial Temperature Millivolts</u>	<u>°C</u>	<u>Exit Temperature Millivolts</u>	<u>°C</u>
1	0.98	24.5	1.04	26
2	1.00	25.0	1.04	26
3	1.04	26.0	1.08	27
4	1.08	27.0	1.12	28

Clear sample irradiated at 200 ft/min.
 200 W/in.
 medium pressure mercury bulbs

A. Grosset
d.m.s. - jd 7-20-81

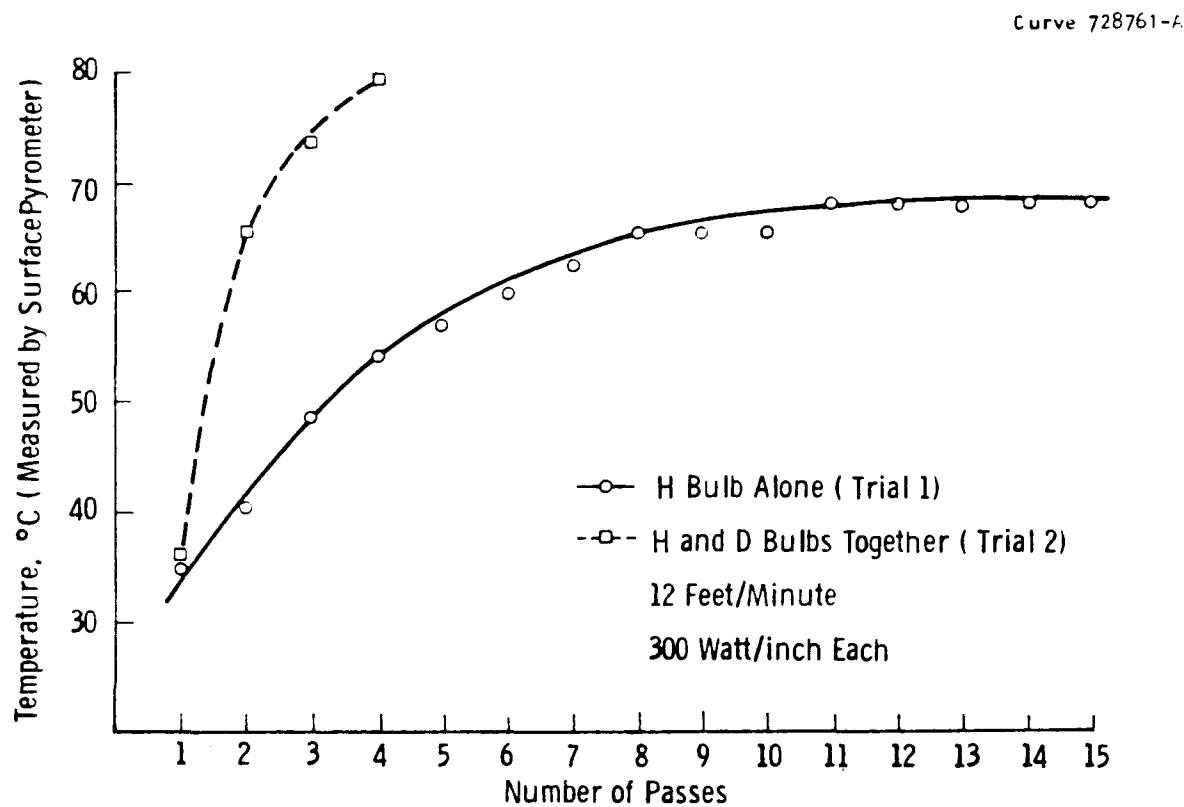


Fig. 9 - Heating cycles of zinc phosphated panels coated with P72D. Lower blower of UV lamps is not on

Curve 728761-A

A. Grosset
dms - jd 7-20-81

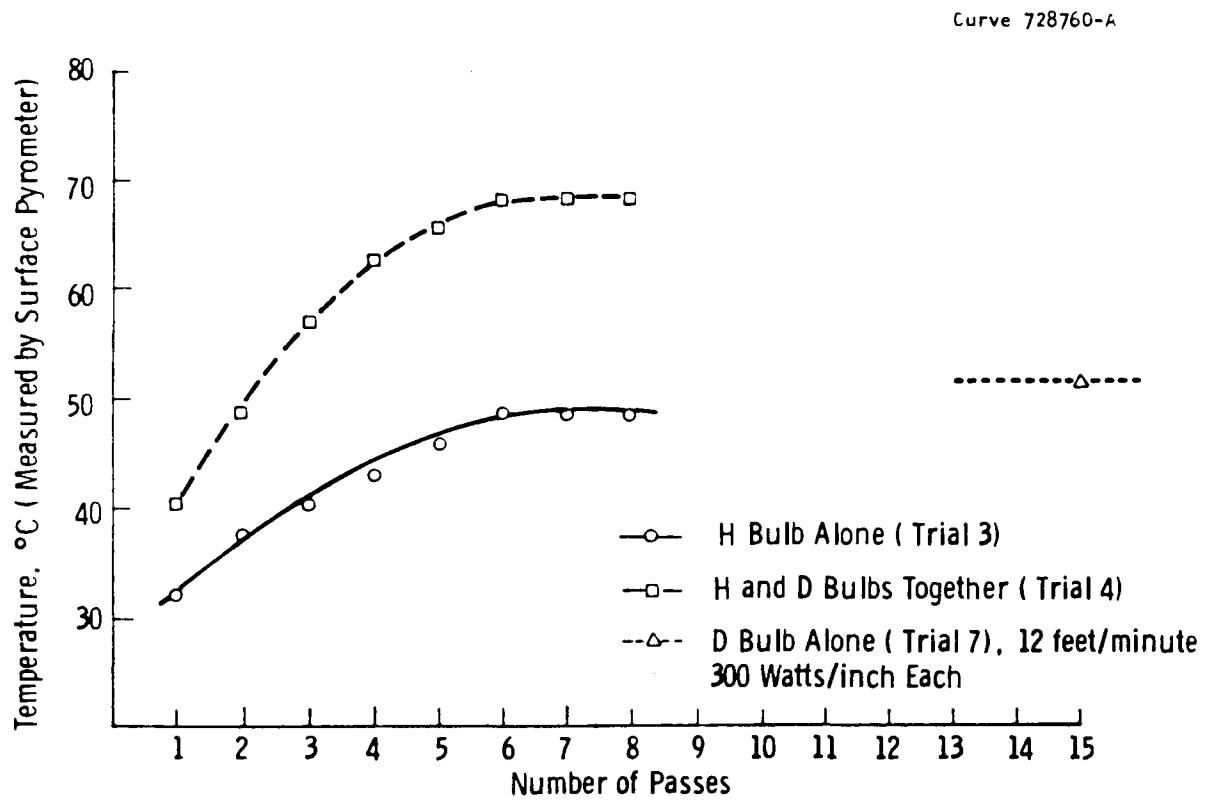


Fig. 10 - Heating cycles of panels coated with P72D. Lower blower of UV lamps is on

Curve 728760-A

TABLE 70-EFFECTS OF TEMPERATURE UPON CURING OF PIGMENTED COATINGS (Sheet 1 of 3)

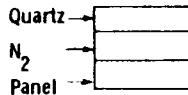
Formulation & Trial Conditions	Trial No. & Date	Cure Time	Crosshatch Test			Pencil Hardness Test			Crosshatch & Hardness Test Dates & Comment
			Zn Phos	Fe Phos	Tin Plated Steel (5 mil)	Zn Phos	Fe Phos	Tin Plated Steel (5 mil)	
P72D TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.20 Irg. 0.28 MDEA 0.28 Rutile TiO ₂ 18.25 Lower Blower Not On	(1) 6/19/80	15 Passes 12 fpm 300 W/in H Bulb	100 %	-	-	5B	-	-	6/23/80 After 15 Passes Temperature Was 68°C
P72D Lower Blower Not On	(2) 6/19/80	8 Passes 12 fpm 300 W/in ea H and D Bulbs Simultaneously	100 %	-	-	HB	-	-	6/23/80 After 4 Passes Temperature Was 79°C
P72D Lower Blower On	(3) 6/19/80	15 Passes 12 fpm 300 W/in H Bulb	-	-	100 %	-	-	B	6/23/80 After 8 Passes Temperature Was 49°C
P72D Lower Blower On	(4) 6/19/80	8 Passes 12 fpm 300 W/in ea H & D Bulbs Simultaneously	-	100 %	-	-	4B	-	6/23/80 After 8 Passes Temperature Was 68°C

TABLE 70-EFFECTS OF TEMPERATURE UPON CURING OF PIGMENTED COATINGS (Sheet 2 of 3)

Formulation & Trial Conditions	Trial No. & Date	Cure Time	Crosshatch Test			Pencil Hardness Test			Crosshatch & Hardness Test Dates & Comment
			Zn Phos	Fe Phos	Tin Plated Steel (5 mil)	Zn Phos	Fe Phos	Tin Plated Steel (5 mil)	
P72D TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.20 Irg. 0.28 MDEA 0.28 Rutile TiO_2 18.25 Lower Blower On	(5) 6/19/80	7 Passes 12 fpm 300 W/in H and D Bulbs Simultaneously	-	-	64 %	-	-	5B	6/23/80
P72D Lower Blower On	(6) 6/19/80	15 Passes 12 fpm 300 W/in D Bulb	-	100 %	-	-	< 6B	-	6/23/80
P72D Lower Blower On	(7) 6/19/80	15 Passes 12 fpm 300 W/in D Bulb	-	-	100 %	-	-	< 6B	6/23/80 After 15 Passes, Temperature Was 52°C
P72D Lower Blower On	(8) 6/19/80	15 Passes 12 fpm 300 W/in V Bulb	-	-	0	-	-	0	6/23/80 Tacky

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TABLE 70-EFFECTS OF TEMPERATURE UPON CURING OF PIGMENTED COATINGS (Sheet 3 of 3)

Formulation & Trial Conditions	Trial No. & Date	Cure Time	Crosshatch Test			Pencil Hardness Test			Crosshatch & Hardness Test Dates & Comment
			Zn Phos	Fe Phos	Tin Plated Steel (5 mil)	Zn Phos	Fe Phos	Tin Plated Steel (5 mil)	
P72D TS3577-11 20.00 g HDDA 5.00 EEA 2.50 DTBP 0.20 Irg. 0.28 MDEA 0.28 Rutile TiO ₂ 18.25 Lower Blower On	(9) 6/19/80	7 Passes Under D Bulb 8 Passes Under H Bulb (Sequentially) 12 fpm 200 W/in ea	-	-	100 %	-	-	< 6B	6/23/80
P72D Lower Blower On Under N ₂ Cooled Quartz Filter 	(10) 6/19/80	2 Passes 12 fpm 300 W/in H Bulb	-	-	42 %	-	-	< 6B	6/23/80
P72D Lower Blower On Under N ₂ Cooled Quartz Filter	(11) 6/19/80	2 Passes 12 fpm 300 W/in V Bulb	-	-	56 %	-	-	< 6B	6/23/80 Tacky

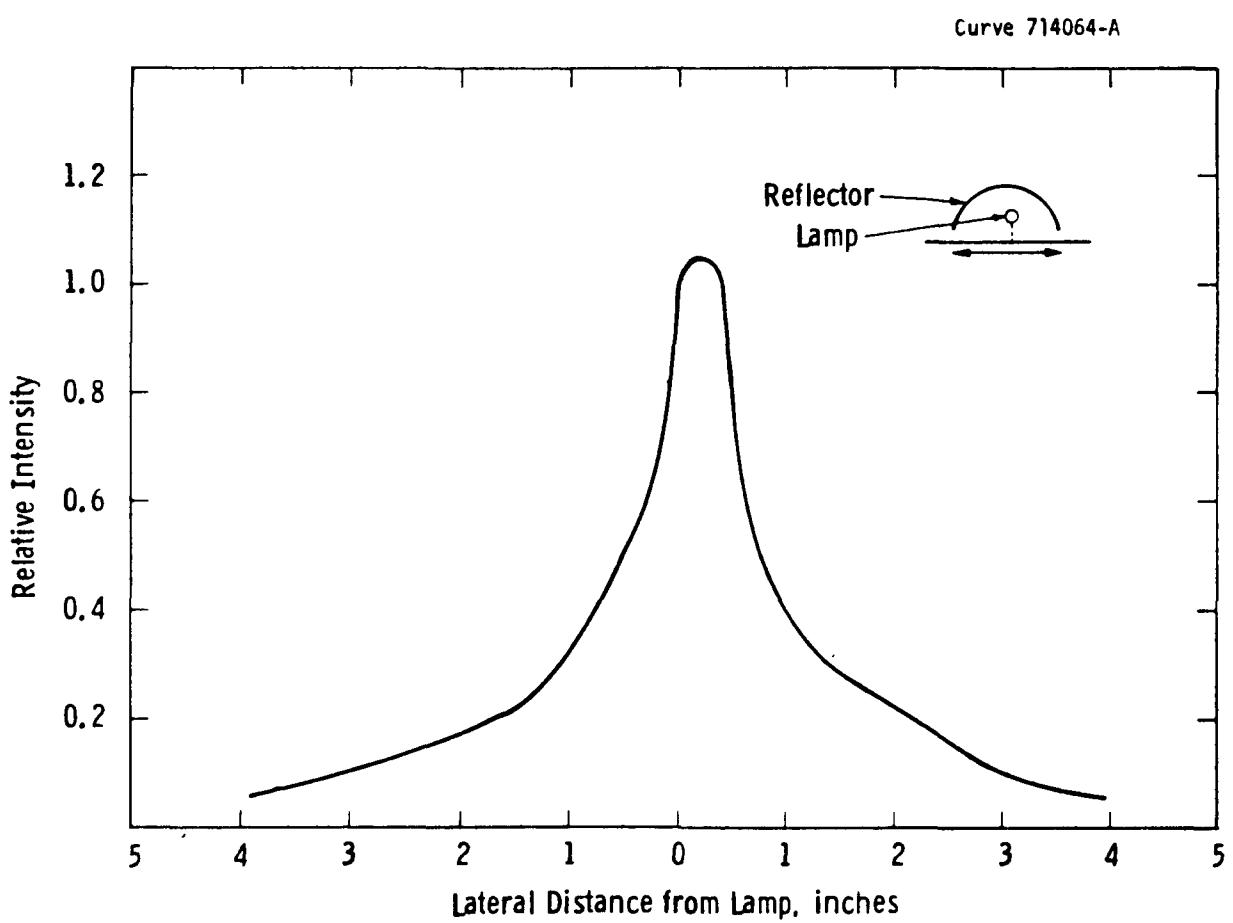


Fig.11 – Variation in radiation intensity with lateral distance from medium pressure mercury lamp (Courtesy PPG Industries)

to cure these coatings on irregularly shaped three-dimensional objects in a light tunnel, the radiation must be non-focused or diffused. This process was simulated in the laboratory by passing the radiation from the lamps through a tin-plated, hollow steel cylinder. Properties of panels painted with P72D which were exposed to diffuse radiation at distances of 7-1/2 and 15 inches from the source are illustrated in Table 71. Pencil hardness and adhesion of these films were tested immediately after cure and also after two days.

5.4 Laboratory Scale Finishing Line Design and Construction

Based on results of these tests, a UV light curing tunnel was designed and constructed at the Westinghouse R&D Center. To simulate a typical industrial paint line, an overhead conveyor moving at 8 ft. per min. takes painted parts through a 5 ft. long tunnel which has six UV lamps (Tables 72 and 73) arranged radially around the travel path. The UV curing tunnel is illustrated in Figure 12. The three-dimensional objects coated for this pilot plant study were 3 in. by 5 in. file card steel boxes. Westinghouse has successfully demonstrated the ability to cure films at a nominal thickness of 1 mil (0.001 in.) on these objects by UV light radiation. Three in. diameter and 3 in. long cylindrical metal paint cans were sprayed with P324 and were run through the UV curing tunnel for about 45 sec. A lidless can was suspended by its sides. The temperature of the can was measured by a thermocouple before it entered the tunnel, and immediately at the exit. The temperature of a can filled only with air rose from 27°C at the tunnel entrance to 116°C at the exit; however, when another can filled with cold water was run through the curing tunnel, its temperature rose from 13°C to only 19°C, well below the thermal initiation temperatures of the paint components. The uncooled can cured completely, and the cooled can cured everywhere but a section about 3/4" thick on the leading and trailing sections of the can, which were not illuminated by the lamps in this configuration. Pencil hardnesses were HB for the uncooled can, and 3B for the cooled can. The paint was slightly yellow on the cooled can. Although the

TABLE 71
PROPERTIES OF PAINTS CURED BY DIFFUSE RADIATION

<u>Panel Type</u>	<u>Distance from Source, In.</u>	<u>Cure Time (seconds)</u>	<u>Panel Hardness</u>	<u>Crosshatch Adhesion, %</u>	<u>Date</u>
ZnPhos	7-1/2	32	HB	100	6/25/80
	"		HB	100	6/27/80
FePhos	7-1/2	30	B	100	6/25/80
	"		2B	100	6/27/80
Steel	7-1/2	30	F	98	6/25/80
	"		F	98	6/27/80
ZnPhos	15	64	2B	100	6/26/80
	"		HB	100	6/30/80
FePhos	15	62	2B	100	6/26/80
	"		HB	100	6/30/80
Steel	15	62	2B	100	6/26/80
	"		HB	100	6/30/80

TABLE 72



FUSION SYSTEMS CORPORATION

12140 Parklawn Drive, Rockville, Maryland 20852 U.S.A. • (301) UV1-5400

MODEL F440
STANDARD 10 INCH LAMP SYSTEM SPECIFICATIONS

All specifications are for a single lamp system, whose length is 10.5". A larger system is made by adding more irradiators and power supplies. Modules placed end-to-end form continuous lamps in lengths which are multiples of 10.5". Items with an asterisk (*) increase proportionately with the size of the system.

IRRADIATOR/BLOWER: Model I223B with K523 Blower **

Lamp Power	300 W/in (3000 Watts total) *
* Dimensions (max)	10.59" long x 8.28" wide at base; 12.31" wide at blower top, x 27.38" high
* Weight	60 pounds
* Cooling	220 cfm of filtered air at 4.2" of static water pressure, as measured in irradiator
Reflector Geometry	Elliptical, bulb at focus
Substrate Location	2.1" from irradiator
Mounting Position	Any angle with respect to vertical
Mounting Enclosure	Can be supplied as custom item

** As an option, the integral blower can be replaced by a remote blower connected with flexible hoses to the irradiator, (Model I223). The irradiator's weight is then reduced to 30 pounds and height to 14.1".

POWER SUPPLY: Model P140A

* Dimensions (max)	17.5" wide x 18" deep x 7.4" high
* Weight	90 pounds
Cooling	Self contained rotary fans
Control Functions	Power Off, Standby, Lamps On
Interlocks	Lamp Out, Lamp Cooling, Power Supply Cooling, RF Leakage, External (user) Signal
Connections	25' cable assembly from power supply to irradiator supplied by Fusion; line power connections supplied by user

UTILITIES:

* Line Power	5550 Watts
Line Voltage	208/240V or 460VAC, 60 Hz; other voltages and 50 Hz available as options
Phase	3 phase
* Control Power	3 Amps at 117 Volts per power supply, 50 or 60 Hz
* Exhaust	External exhaust optional for systems of one or two irradiators; for larger systems 300 cfm per irradiator must be exhausted by user.
Compressed Air	None
Water	None

TABLE 73



FUSION SYSTEMS CORPORATION

12140 Parklawn Drive, Rockville, Maryland 20852 U.S.A. • (301) UV1-5400
TWX 710-828-0085
Answer Back/ "Fusion Rose"

OUTPUT SPECTRA OF VARIOUS BULBS (in W/inch)

Wavelength Band	Mercury		Non-Mercury Bulbs			
	H	D	X	M	V	A
200-210 nm	1.3	0.1	1.0	0.8	0	1.0
210-220	6.0	0.1	2.3	2.2	0	4.3
220-230	9.3	2.0	5.7	3.4	0	5.4
230-240	8.6	2.3	5.4	4.1	0	5.5
240-250	7.2	4.0	3.2	4.9	0.3	8.4
250-260	9.5	5.6	4.3	8.4	0	8.6
260-270	11.3	4.3	3.4	10.7	0	5.0
270-280	1.7	5.2	11.9	1.3	0	4.3
280-290	5.9	4.9	7.8	15.2	0	14.6
290-300	9.7	9.6	6.2	3.8	0	13.6
300-310	5.1	6.4	2.8	1.4	0	7.1
310-320	8.9	4.1	5.3	3.6	0	5.9
320-330	0	1.4	2.9	0.4	0	2.8
330-340	2.0	1.5	2.6	0.5	0	1.9
340-350	0	3.6	6.8	0	0	.5
350-360	0	8.5	2.0	2.5	0	8.5
360-370	23.0	19.7	13.9	27.8	0.9	12.2
370-380	0	15.7	0	7.6	0	7.2
380-390	0	17.4	0	0	0	0
390-400	0	5.1	4.1	0	6.0	0
400-410	9.1	12.3	16.8	9.7	23.3	5.0
410-420	0	6.8	4.5	0.3	37.0	1.0
420-430	0	10.5	11.8	0	18.5	0
430-440	28.6	26.5	10.0	24.3	11.0	27.0
440-450	0	3.9	0	0	10.4	0
450-460	0	1.0	0	0	5.6	0
460-470	0	0	9.7	0	2.0	0
470-480	0	0	0	0	0	0
480-490	0	1.5	11.3	0	0	5.0
490-500	0	3.5	4.0	0	0	0
500-510	0	2.7	0	0	0	0
510-520	0	1.8	8.4	0	0	0
520-530	0	0	3.8	0.7	0	0
530-540	0	0	11.9	0.8	0	0
540-550	23.9	20.0	0.7	0	2.5	22.6
550-560	0	3.6	0	0	0	0
560-570	0	5.2	0	9.2	0	0
570-580	22.0	11.1	20.0	2.2	0	20.0
580-590	0	0	5.0	0	2.5	4.0
590-600	0	0	0	2.5	0	
UV: 200-400 nm	109.5	121.5	91.6	98.6	7.2	116.8
Visible: 400-600 nm	83.6	110.4	117.9	49.7	112.8	84.6
TOTAL:	193.1	231.9	209.5	148.3	120.0	201.4

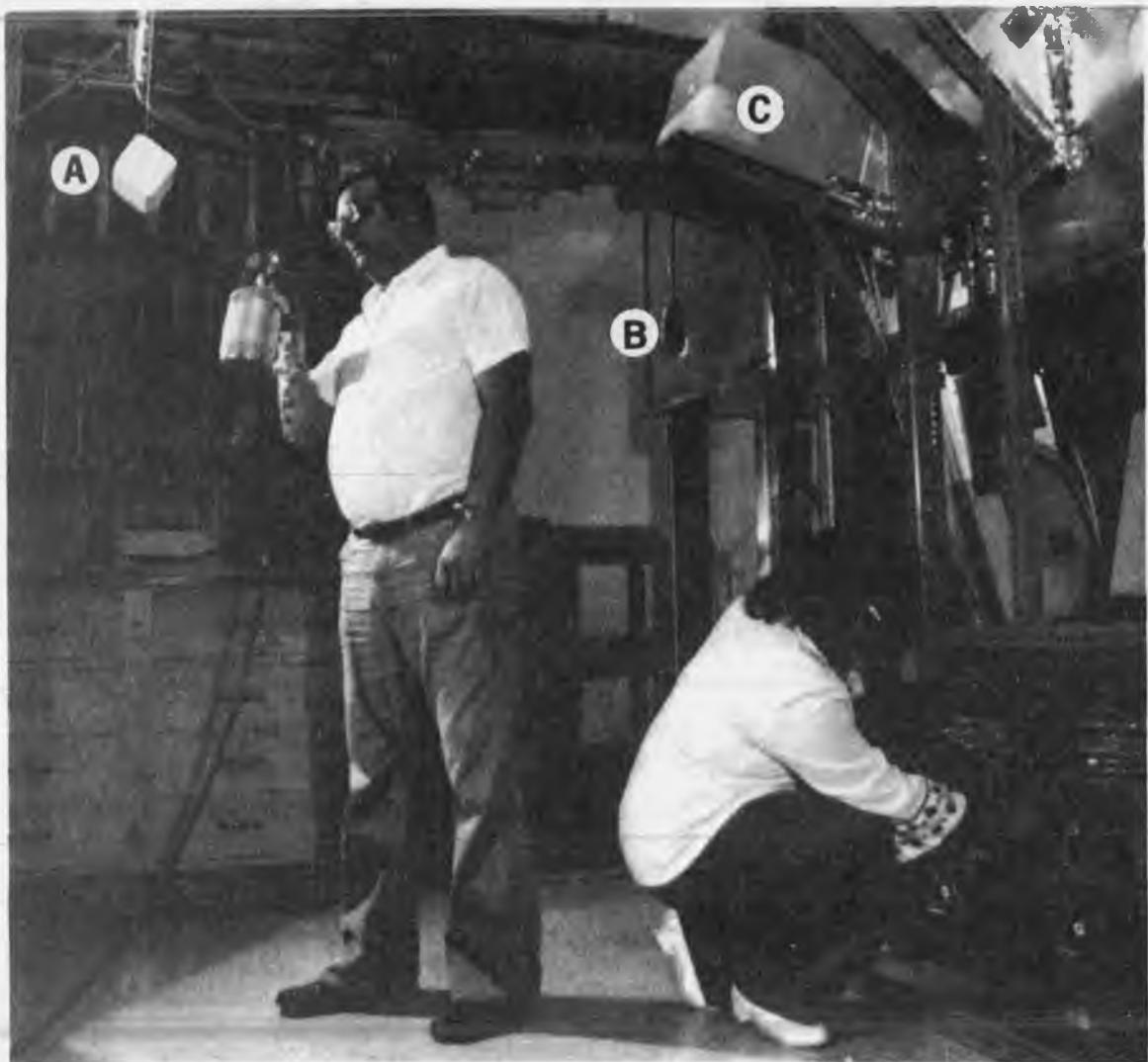


Figure 12 - Laboratory setup for energy-efficient UV curable paints. Object (A) is sprayed with our new paint, then moved on conveyor to curing tunnel (B), where UV irradiators (C) speedily cure the paint.

objective of this program was not to develop paints for specific end uses, with modifications these coatings should find many applications in finishing industrial products at great benefit to the end users. Members of industry have witnessed the operation of the laboratory scale unit (see Appendix III) and a news release was sent to various trade journals (see Appendix IV).

When the tunnel was monitored for safety and environmental protection, no volatile organic emissions or generation of ozone was detected. The Ames test indicated that P324 and PCP290 are nonmutagenic.

5.5 Industrial Application of UV Curing of Paints

An economic comparison of operating costs was made for a conventional finishing line and an equivalent UV curing finishing line (Table 74). Although the average paint line is designed to finish 10,000,000 sq. ft./year, this analysis is based on a small unit finishing 1,000,000 sq. ft./year. This unit is expected to be operated 4000 hr./year, the usual operating time of a typical paint line. The energy efficiency of the UV curing tunnel measured as the output energy delivered compared to the energy consumed is: (1215 watts/5550 watts) x 100 = 22%. Data concerning Fusion Systems Corp. irradiators are listed in Tables 72 and 73. The actual production unit will use 18 Fusion Systems Corp. UV irradiators. Since an irradiator uses 18,950 Btu/hr., the unit will require 341,100 Btu/hr.

The toxicity of the acrylic monomers used in UV curable coatings is a potential restriction on applicability of UV curing processes. Shielding personnel from UV radiation and ozone production could be also minor restrictions. A possible additional difficulty is that some UV curable coatings may have a limited shelf life.

The advantages of UV curing over conventional technologies (Ref. 1 and 2) are:

1. Requires 1/3 to 1/4 less energy.
2. Higher throughput because of shorter dwell time in the curing chamber. A part coated with conventional paint would need baking times of about 20 min. at 300°C, while a part coated with UV curable paint would cure in about 45 sec., based on laboratory trials.
3. Less floor space required for UV curing tunnel.
4. Elimination of air pollution emissions and reduction in safety hazards.
5. No volatile organic compounds emitted.
6. Temperature-sensitive substrates could be coated.

This UV finishing process could be used by the following: furniture, metal fabrication, machinery, electrical equipment, transportation equipment, instruments and other similar manufacturing industries. Although at this writing film properties of UV cured paints are not equal to those of conventional paints, it is felt that there will be no significant changes to the painted end product.

The primary components of this technology which are proven and readily available are UV lamps, power supplies, conveyors, paint application equipment, spray paint booths and exhaust systems. Not yet optimized components are UV lamp curing tunnels and the UV cured paints. The relative costs of conventional ovens and UV curing lines are listed in Table 74. The higher capital costs of UV curing equipment could be offset by the lower cost of energy consumption for UV. Operating experience with UV light curing processes has demonstrated an overall operating cost which is approximately one-fourth that of natural gas. The combination of this cost reduction and the elimination of dependence on the limited supply of natural gas should be welcomed by industry.

TABLE 74

CAPITAL AND OPERATING COSTS FOR CONVENTIONAL THERMAL CURING AND FOR UV CURING

	<u>Conventional</u>	<u>UV Curing</u>	<u>Reference</u>
1. Capital cost to user (\$/unit)	\$52,000 ^(a)	\$99,000 ^(b)	(a) Spray Con Corp. (b) Fusion Systems
2. Installation cost to user (\$/unit)	24,000 ^(a)	5,000 ^(b)	(a) Spray Con Corp. (b) Estimated
3. Installation/construction time (years)	1/12 year ^(a)	1/12 year ^(b)	(a) Spray Con Corp. (b) estimated
4. Annual Energy Consumption (Btus/yr/unit)			
a. Gas	1.00×10^{10} Btu/ yr/unit ^(a)	1.36×10^9 Btu/ yr/unit ^(b)	(a) Westinghouse Research Memo 74-1B5-FINSY-M3 (b) Geo. Hack & Sons
b. Oil	--	--	
c. Coal	--	--	
d. Electricity (@ 10,500 Btus/kWh)	--	4.68×10^9 kWh/yr total (90% market penetration)	
e. Other (explain below)	--	--	
5. Non energy savings or costs (\$/unit/yr) (Use positive numbers for savings, negative numbers for costs)	--	--	Geo. Hack & Sons
6. System's Useful Life (Yrs)	7 to 10 yrs ^(a)	7 to 10 yrs ^(b)	(a) Spray Can Corp. (b) estimated
7. Scrap Value (% of Capital Costs)	negligible ^(a) less than 10%	negligible ^(b) less than 10%	(a) Spray Can Corp. (b) estimated

Comments:

4.b $18,950 \text{ Btu/hr/lamp} \times 18 \text{ lamps/unit} \times 4,000 \text{ hrs/year} = 1,364,400,000 \text{ Btu/year/unit.}$

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APPENDIX I

THE ENERGY EFFICIENCY OF GAS FIRED OVENS

High temperature baking cycles are generally accomplished in gas fired ovens. The efficiency of such an oven is approximately 20%, only a small fraction of which is really used to cure the paint. The ratio of used heat to wasted heat will be a product of the thickness, density, and specific heat capacity of the paint. In the case of a 1 mil thick coating on a 40 mil sheet of steel, the utilization factor is as follows:

$$1/40 \times 1/8 \times 3/1 =$$

(thickness) x(density) x(sp. heat)

$$0.94 \text{ of } 20\% = 0.18\%.$$

APPENDIX II

TECHNICAL BACKGROUND

1. Ultraviolet Radiation Curing Chemistry

Two different mechanisms were investigated for the ultraviolet curing of paints: free radical and cationic photopolymerization. The oligomers which serve as base resins are usually low to medium weight mono or multifunctional monomers. Typical resins are acrylates, polyesters, polyethers, urethanes, and epoxies.⁴⁰ The photoinitiator attacks the compounds at the unsaturation or the epoxy ring to initiate polymerization.

In free radical initiation a photosensitive molecule absorbs UV radiation and is raised to an excited state (Figure 1). If it is a photoinitiator, it may participate in intersystem crossing to the excited triplet state and then cleave to form radicals. If the molecule is a photosensitizer it could interact with another molecule through energy transfer, electron transfer, or H-abstraction. These reactions could occur by means of excited complex formation (Figure 2).

The free radicals formed then interact with a monomer to initiate the chain polymerization (Figure 3). The molecule thus formed contains an active center which continues to react with other monomer molecules and propagates the polymerization. The initiators also react with oxygen, which may retard the reaction.³⁴⁻³⁸

Another photoinitiation mechanism is the cationic process. In the cationic process "onium" salts, such as aryl diazonium, diaryliodonium, and triarylsulfonium, containing complex metal halide anions, such as hexafluorophosphate, hexafluoroarsenate, and hexafluoroantimonate (PF_6^- , AsF_6^- , SbF_6^-) are the photoinitiators. The molecule absorbs radiation and is raised to the excited state (Figure 4). The photoinitiator cleaves homolytically to form a free radical and a diarylsulfonium salt. The salt abstracts a proton from the monomer to yield another radical and a protonated diarylsulfide.

FIGURE 1

FREE RADICAL PHOTINITIATION MECHANISMS

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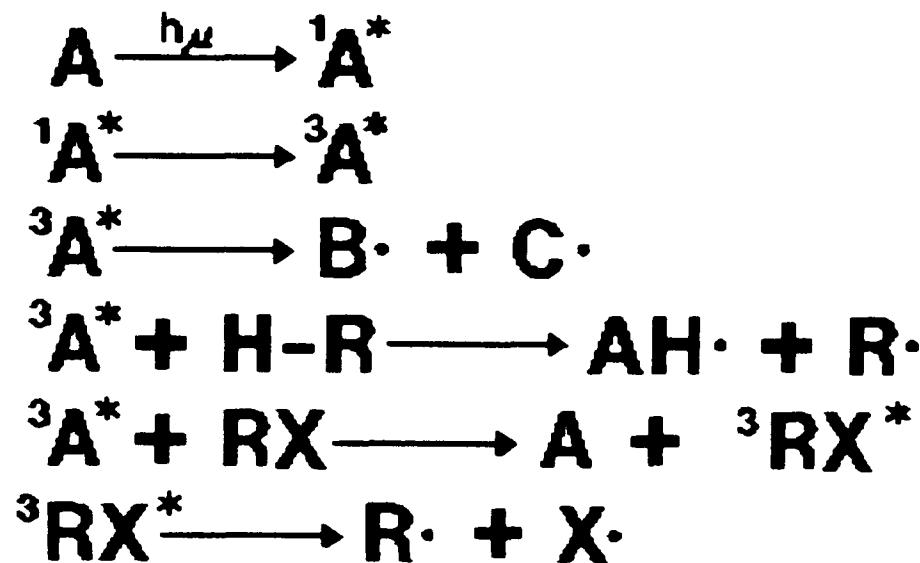


FIGURE 2

FREE RADICAL PHOTOINITIATION WITH EXCITED COMPLEX FORMATION

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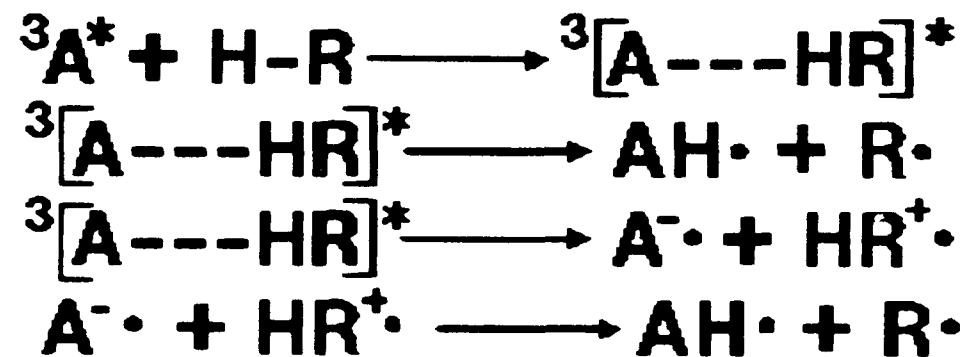


FIGURE 3

FREE RADICAL PHOTONITIATION AND PROPAGATION

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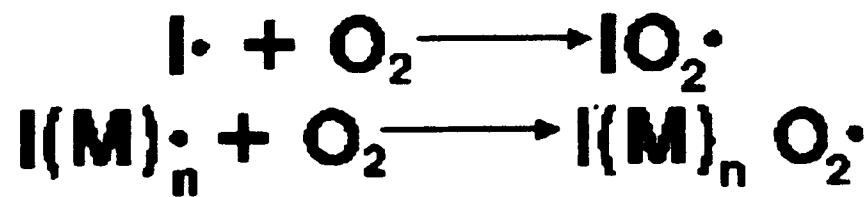
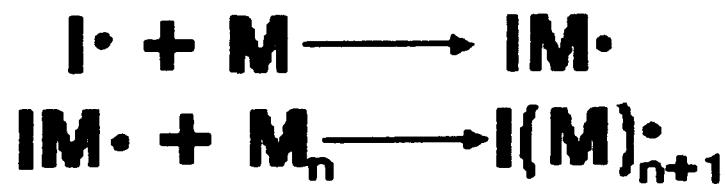
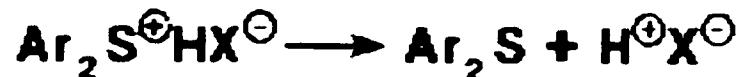
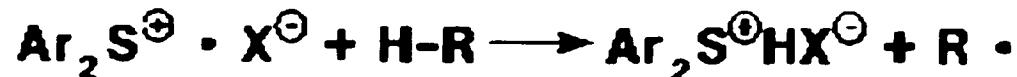


FIGURE 4

CATIONIC PHOTINITIATION BY
TRIARYLSULFONIUM SALTS OF
COMPLEX METAL HALIDE ANIONS

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The resulting Bronstead acid HX initiates the cationic polymerization of the monomers (Figure 5). Cationic "living" polymerization continues even after removal of light, and is not inhibited by oxygen.²⁴⁻³⁰

2. The Hiding Power of Pigments

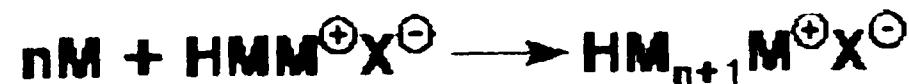
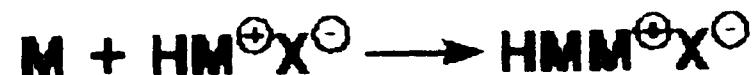
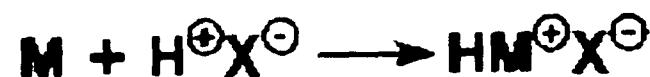
The hiding power of a pigment is determined by its ability to scatter and absorb visible radiation. For white pigments, hiding is accomplished primarily by light scattering which depends on the difference in refractive index between pigment and binder. The greater this difference, the more effective is the light scattering.

The hiding power of a pigment can be expressed in terms of SX value. X is film thickness and can be determined by area, density and weight measurements. S is the Kubelka-Munk scattering coefficient and is obtained at 550 nm from the relationship $SX = ((1-T)/T) - 0.5$, where T is the fraction of incident light transmitted through the film and the factor 0.5 compensates for back-scatter reflectance.

FIGURE 5

CATIONIC PHOTINITIATION AND PROPAGATION

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APPENDIX III

THE LIST OF VISITORS FOR THE LABORATORY DEMONSTRATION OF THE UV-CURING PROCESS

<u>Company Name</u>	<u>Visiting Date</u>
Insulating Materials Division (IMD) of Westinghouse	5/11/81
Nordson Corporation	6/24/81
PPG Industries	6/28/81
Hughson Chemical Company	6/30/81
Advanced Power Systems Division (APSD) of Westinghouse	7/8/81
Advanced Energy Systems Division (AESD) of Westinghouse	
Advanced Reactors Division (ARD) of Westinghouse	
Technical Writers of R&D Center of Westinghouse	
Electric Power Research Institute (EPRI)	7/22/81
Sartomer Company	8/19/81
Navy	8/25/81
Productivity Center of Westinghouse	8/25/81
Siemens Allis Company	8/25/81
Applied Physics of R&D Center of Westinghouse	8/26/81
International Service Division of Westinghouse	9/3/81
Philip Holzmann Company	9/3/81
Ciba-Geigy Corporation	9/23/81
Union Carbide Corporation	9/24/81
Mine Safety Appliances Company	9/29/81
Gateway Paint Co.	10/6/81
Reliance Electric Co.	10/16/81

APPENDIX IV

THE LIST OF TRADE JOURNALS FOR THE ANNOUNCEMENTS OF THE UV-CURING PROCESS DEMONSTRATION

Products Finishing Magazine	33
Journal of Coatings Technology	Wire Industry
American Paint & Coatings Journal	Wire Journal
Pittsburgh Post Gazette	Wire Technology
Pittsburgh Press	Modern Plastics
Industrial Finishing Magazine	NAPF
Metal Finishing	Plastics Design & Processing
Modern Paint & Coatings Magazine	Plastics Machinery & Equipment
Air/Water Pollution Report	Plastics Technology
Chemical & Engineering News	Plastics World
Clean Water Report	Compressed Air Magazine Division
Environmental Science & Technology	Electrical Energy Management
Ground Water Age	Energy Management
Industrial Wastes	Energy User News
Industrial Water Engineering	Energy Users Report
Journal of Environmental Health	Industrial Engineering
Journal of Environmental Sciences	Industrial Maintenance & Plant Operation
Journal Water Pollution Control Federation	Industrial World
Pollution Engineering	Manufacturing Engineering
Pollution Equipment News	Material Handling Engrg.
Water & Sewage Works	Modern Industrial Energy
Water & Wastes Digest	Modern Materials Handling
Water & Wastes Engineering	Plant Energy Management
Water Well Journal	Plant Engineering
American Machinist	Power Transmission Design
American Metal Market/Metalworking News	Production
Automotive Engineering	Production Engineering
Automotive Industries	Quality
Heating/Combustion Equip. News	Quality Progress
Heat Treating	Saving Energy
Industrial Heating	Tooling & Production
Industry Week	Actuator Systems
Iron Age	Control Engineering
Iron Age Metalworking International	Design Engineering
Iron & Steel Engineer	Design News
Machine and Tool Blue Book	Electromechanical Design
Manufacturing Engineering	Industrial Design
Materials Engineering	Machine Design
Metal Progress	New Tech
Metals Week	Sound & Vibration
Metalworking News	Alternative Energy Developments
Metflax Magazine	American Laboratory
Midwest 88 Manufacturing	Bek Industries, Inc.
NC Shop Owner	Industrial Research/Development (2)
	Inside R&D

APPENDIX IV (continued)

Instrument & Apparatus News
Laboratory Equipment
Measurements & Control News
Review of Scientific Instruments
Science
Science Digest

Science International
Science News
Science World
Scientific American
Technology News
Technology Tomorrow

NOMENCLATURE

1. White Pigments for UV Curable Paint

<u>Material</u>	<u>Supplier</u>
Zinc oxide XX-602	New Jersey Zinc Co.
Antimony trioxide White Star M	NL Industries Inc.
Zinc sulfide	Aceto Chemical and Pfaltz & Bauer
Anatase titanium dioxide - Titanox 1000	NL Industries Inc.
Rutile titanium dioxide - Titanox 2010	NL Industries Inc.
Rutile titanium dioxide - Titanox 2020	NL Industries Inc.
Tioxide RHD6X	Tioxide America, Inc.
Unitane 0-310	American Cyanamid Co.

2. Reactive Oligomers for UV Curable Paint

Acrylated urethane TS3577-11	Hughson
Acrylated urethane TS3577-12	Hughson
Acrylated polyester FS10,037	Westinghouse
Vinyl terminated butadiene 2000X164	Goodrich
Vinyl terminated butadiene 2216XZ	Goodrich
Acrylated butadiene G1000-AC	Westinghouse
Acrylated butadiene 1300X17-AC	Westinghouse
Acrylated butadiene LX10-AC	Westinghouse
Polyester Alpolit V81	Hoechst
Acrylated fatty acid Actomer X80	Union Carbide
Acrylated urethane RD2970-1	Hughson
Acrylated urethane RD2970-5	Hughson
Acrylated urethane RD3073-35	Hughson
Acrylated epoxy Celrad 3700	Celanese
Acrylated epoxy Celrad 3701	Celanese
Acrylated epoxy RR0383	Celanese
Acrylated epoxy RR27482	Celanese
Acrylated epoxy XD9002	Dow
Acrylated epoxy 301	Shell
Acrylated epoxy 303	Shell
Aronix	Mitsui
Cycloaliphatic diepoxide resin ERL4221	Union Carbide
Cycloaliphatic diepoxide resin ERL4299	Union Carbide

3. Reactive Monomers for UV Curable Paint

<u>Material</u>	<u>Supplier</u>
Pentaerythritol tetra-acrylate (PETTA)	Sartomer
Pentaerythritol triacrylate (PETA)	Union Carbide
Trimethylol propane triacrylate (TMPTA)	Sartomer
Neopentyl glycol diacrylate (NPGDA)	Sartomer
1,6-Hexanediol diacrylate (HDDA)	Celanese
Tetraethylene glycol diacrylate (TTEGDA)	Celanese
Diethylene glycol diacrylate (DEGDA)	Sartomer
Trimethylol propane trimethacrylate (TMPTMA)	Sartomer
Neopentyl glycol dimethacrylate (NPGDMA)	Sartomer
Triethylene glycol dimethacrylate (TEGDMA)	Sartomer
1,3-Butylene dimethacrylate (BDMA)	Rohm/Haas
Ethoxylated bis-phenol A dimethacrylate (EBPADMA)	Sartomer
2-Hydroxy ethyl acrylate (HEA)	Dow
2-Hydroxy ethyl methacrylate (HEMA)	Eastman
Hydroxypropyl methacrylate (HPMA)	Rohm/Haas
Ethyl acrylate (EA)	Rohm/Haas
Butyl acrylate (BA)	Eastman
n-Hexyl acrylate (HA)	Haven
2-Ethyl hexylacrylate (EHA)	Celanese
Crotyl acrylate (CA)	Haven
Furfuryl acrylate (FA)	Haven
Dicyclopentadiene acrylate (DCPA)	Dow
Isobornyl acrylate (IBA)	Rohm/Haas
Methoxyethyl acrylate (MEA)	Haven
2-Ethoxyethyl acrylate (EEA)	Proctor
Butoxyethyl acrylate (BEA)	Proctor
Phenoxyethyl acrylate (PEA)	Daubert
Methyl methacrylate (MMA)	Eastman
Ethyl methacrylate (EMA)	Eastman
n-Hexyl methacrylate (HMA)	Haven
Glycidyl methacrylate (GMA)	Haven
Vinyl acetate (VA)	Celanese
N-Vinyl pyrrolidone (NVP)	Eastman

3. Continued

<u>Material</u>	<u>Supplier</u>
Vinyl toluene (VT)	Dow
Diallyl phthalate (DAP)	Borden
Styrene	Aldrich
Divinyl benzene (DVB)	Chem Procurement Lab.
1,5-cyclo-octadiene (CO)	Aldrich
Methacrylic acid (MAA)	Haven
Limonene dioxide	Viking
Butyl glycidyl ether (RD-1)	Ciba-Geigy
1,4-Butanediol diglycidyl ether (RD-2)	Ciba-Geigy
Epoxide 8	Proctor & Gamble
Diepoxy octane	Aldrich

4. Photoinitiators for UV Curable Paint

Benzoin isopropyl ether (V-10)	Stauffer
Benzoin isobutyl ether (V-30)	Stauffer
Diethoxyacetophenone (DEAP)	Union Carbide
Darocur 1116 (D-1116)	Merck
Darocur 1173 (D-1173)	Merck
Irgacure 651 (I-651)	Ciba-Geigy
Benzophenone (Bz)	Fisher
Mischler's ketone (MK)	Eastman
Chlorothioxanthone (CTX)	Aldrich
2-Ethylanthraquinone (EAQ)	Aldrich
FC-508	3M
FC-509	3M
2-Methylanthraquinone (MeAQ)	Aldrich
2-Isopropylthioxanthone	Polysciences, Inc.

5. Photosensitizers or Coinitiators for UV Curable Paint

Ethyl para-dimethylaminobenzoate (EPDMAB)	Aldrich
8-Quinolinesulfonyl chloride (QSC)	Aldrich
Uvecryl P101	Virginia
Uvecryl P102	Virginia
Uvecryl P104	Virginia
i-Propylamine (IPA)	Eastman
Phenethylamine (PEA)	Fisher

5. Continued

<u>Material</u>	<u>Supplier</u>
Aniline (A)	Fisher
Diisopropyl amine (DiPA)	Fisher
Diphenyl amine (DPA)	Fisher
Triphenyl amine (TPA)	Eastman
N,N-dimethyl-m-toluidine (DMMT)	Eastman
N,N-dimethyl-p-toluidine (DMPT)	Fisher
Triethylamine (TEA)	Fisher
Monoethanolamine (MEOA)	Fisher
Diethanolamine (DEOA)	Fisher
Triethanolamine (TEOA)	Fisher
N-Methyl diethanolamine (MDEA)	Aldrich
N,N-Dimethyl acetamide (DMAA)	Fisher
Triethylene diamine (TEDA)	Eastman
Diethylene triamine (DETA)	Fisher
N,N-Diemethylaminoethyl methacrylate (DMAEMA)	Haven
N,N-Dimethylethanolamine (DMEA)	Aldrich
FC-510	3M
Benzil	Aldrich
Morpholine	Aldrich
Thioxanthone	Aldrich
Benzanthrone	Aldrich
1,3-Diphenyl-2-propane	Aldrich
Triphenyl acetophenone	Aldrich
Fluorenone	Aldrich
Biphenyl	Aldrich
Flavone	Aldrich
4-Acetyl Biphenyl	Aldrich
Diacetyl	Aldrich
Fluorene	Aldrich
9,10-Phenanthrenequinone	Aldrich

6. Additives for UV Curable Paint

18W286 White color Pennco radiation curable epoxy pigment dispersion	Penn Color
A-186 Adhesion promoter	Union Carbide
Epi-Rez 502 Epoxy flexibilizer	Celanese
LHT-240 Polyol flexibilizer	Union Carbide
FC-171 Surfactant	3M
FC-170 Surfactant	3M

6. Continued

<u>Material</u>	<u>Supplier</u>
Chemlink Z Zinc diacrylate	Ware
Red iron oxide corrosion inhibiting pigment	Pfizer
Halox BW-111 corrosion inhibitor	Halox Pigments
Fibrene C-400 pigment extender	Cypress Industrial Mineral
Bentone 34 Rheological additive	N.L. Industries Inc.
Ganex Surfactant	GAF
Zinc chromate corrosion inhibitor	Dominion

7. Thermal Initiators for UV Curable Paint

t-Butyl hydroperoxide (TBH)	Lucidol
Hydroxy peroxide (30%) (HP)	Fisher
2,5-Dimethyl-2,5-bis (t-butyl peroxy) hexane (TBP)	Lucidol
Lauroyl peroxide (LP)	Lucidol
Dicumyl peroxide (DP)	Hercules
2,5-Dimethyl-2,5-bis (hydroperoxy) hexane	Lucidol
Azobis (isobutyronitrile) (AIBN)	DuPont
Bis(t-butyl peroxy i-propyl) benzene (DMBPH)	Chenetron
Cumene hydroperoxide (CH)	Haven
Benzoyl peroxide (BPO)	Fisher
Methyl ethyl ketone (60%) (MEK)	Lucidol
Di-t-butyl perbenzoate (DTBP)	Lucidol