

ENGINEERING RESEARCH INSTITUTE
UNIVERSITY OF MICHIGAN
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Final Report

DEVELOPMENT OF A DRY POLYVINYL ACETATE LATEX
FOR USE IN WATER PAINTS

September 14, 1953, to June 30, 1955

L. L. Carrick

F. L. Keck

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TABLE OF CONTENTS

	Page
LIST OF TABLES	iii
LIST OF FIGURES	iv
ABSTRACT	v
OBJECTIVE	vi
DEVELOPMENT OF A DRY POLYVINYL ACETATE LATEX	1
INTRODUCTION	1
METHOD OF DRYING	1
DESCRIPTION OF FREEZE-DRY PROCESS AND EQUIPMENT	1
DISCUSSION OF EXPERIMENTAL RESULTS	2
USING A PROTECTIVE COLLOID	2
ELIMINATION OF PROTECTIVE COLLOID	3
1. By Washing and Use of Centrifuge	3
2. By Dialysis in Visking Dialysis Membrane	4
EFFECT OF DRY POLYVINYL ACETATE ON BONDEX AND CEMENT	6
CONCLUSIONS	7
SURFACE TREATMENT OF BONDEX	8
SUPPLEMENT ON THE DEVELOPMENT OF A LIQUID POLYVINYL ACETATE PAINT	12
SUPPLEMENT ON PAINT AND VARNISH REMOVERS	13
APPENDIX	17
GLOSSARY	45

LIST OF TABLES

	Page
1. EFFECT OF DRY POLYVINYL ACETATE ON BONDEX AND CEMENT	6
2. WASHABILITY OF SURFACE-TREATED BONDEX	9
3. EXTERNAL EXPOSURE RESULTS OF WATER BASE PAINTS	12
4. EXPERIMENTAL FREEZE-DRIED MODIFIED POLYVINYL ACETATE SYSTEMS	18

LIST OF FIGURES

	Page
1. Bondex surface sprayed with water.	10
2. Bondex surface sprayed with ammonium carbonate.	11
3. Bondex surface sprayed with ammonium sulfate.	11
4. Comparison of laboratory paint and varnish formulations.	15
5. Comparison of commercial and laboratory-compounded paint and varnish removers.	16

ABSTRACT

This report, together with the previous reports on the development of a polyvinyl acetate paint and the development of paint and varnish removers, constitutes a final report of the Reardon Company's contract, September, 1953 to June 30, 1955.

The work was directed toward the development of a dry polyvinyl acetate latex which could be used either as an additive to Bondex and similar materials or as a base for a dry polyvinyl acetate paint. The paint would have the characteristic of being nonre-emulsifiable when dispersed in water and applied. Work had been done previously along these lines before Reardon Company's Product Development Department requested the development of a liquid polyvinyl acetate masonry paint and, subsequently, a paint and varnish remover suitable for trade sales.

The reports covering the development of a liquid polyvinyl acetate paint and the development of a paint and varnish remover were issued in October, 1954. Further information on the external exposure results relative to the liquid polyvinyl acetate paint is summarized later on page 12, and on page 13 is a brief summary on paint and varnish removers, including pictures of the test panels which were not available at the time of our previous report. In addition to the three major phases of development work done for the Reardon Company, comparative tests were run on two texture paints for Mr. Ben Zmuda, the results of which were reported by letter and in the April, 1955 report. Photographs of the washing tests were sent to Mr. Zmuda later.

The work done on the development of a dry polyvinyl acetate latex and polyvinyl acetate paint is discussed on the following pages.

OBJECTIVE

The objectives of this project are as follows:

- a. The development and testing of liquid polyvinyl acetate latex paints.
- b. The formulation of paint and varnish removers.
- c. The testing of texture paints submitted by the Reardon Company.
- d. Development of a dry polyvinyl acetate latex which could be used either as an additive to Bondex and similar materials or as a base for a dry polyvinyl acetate paint.

DEVELOPMENT OF A DRY POLYVINYL ACETATE LATEX

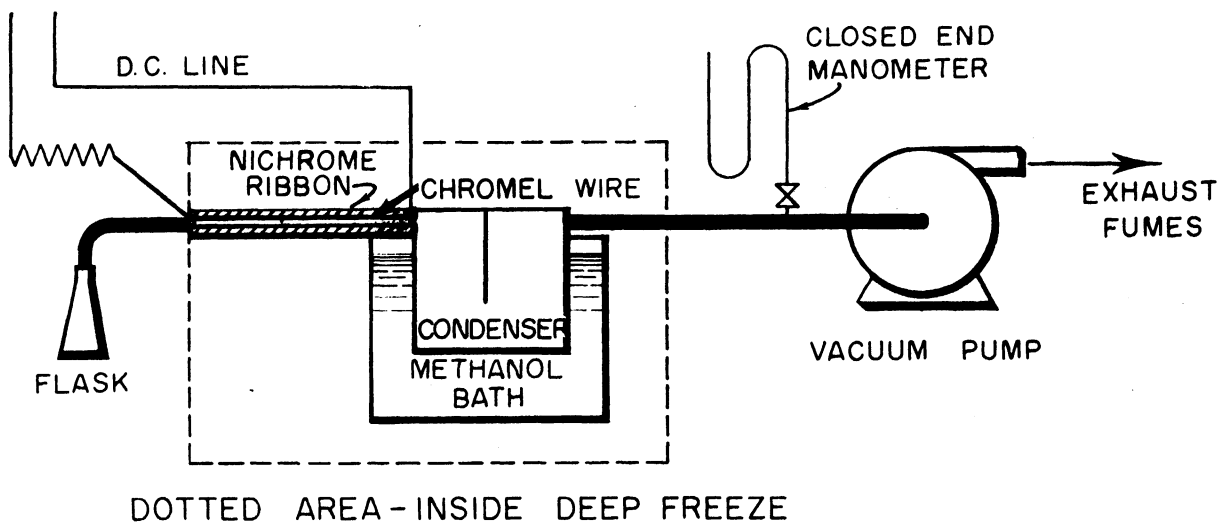
Introduction

METHOD OF DRYING

Polyvinyl acetate latex dries by evaporation to form a hard, tough, insoluble film. The problem was to make a dry polyvinyl acetate latex that, once redispersed, would form a film having similar properties. Two methods for drying the latex were considered: (1) spray drying and (2) freeze drying. Spray drying utilizes heat, which might lead to serious blocking problems of any dried polyvinyl acetate latex. Since laboratory equipment for freeze drying was available at the University of Michigan, this method was investigated first. The results were most gratifying; therefore, a freeze-dry unit was constructed in the laboratory.

DESCRIPTION OF FREEZE-DRY PROCESS AND EQUIPMENT

A brief sketch of the freeze-dry unit employed in our laboratory follows:



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Briefly, material is dried in our laboratory freeze-dry unit as follows. The material (polyvinyl acetate, etc.) is shell frozen in a flask by placing it inside the deep freeze, which has a temperature of -25°C . When frozen, the flask is connected to the freeze-dry unit and a vacuum applied. The ice is sublimed, leaving a network of polyvinyl acetate. The vacuum, which is about 100 microns, as noted from the closed-end mercury manometer, is applied for about four hours to ensure thorough drying of the sample. The sublimed water condenses in the condenser, which is at -25°C . When the sample is dried, the flask is removed from the unit and the material is removed from the flask with a spatula. The dried sample is powdered in a mortar. It is then suitable for redispersion and film casting.

Discussion of Experimental Results

All the experimental results on freeze drying various polyvinyl acetate latex samples are summarized in Table 4, pages 18 through 44. The components used in each sample run and the observations on the resultant films cast from the freeze-dried materials are listed.

USING A PROTECTIVE COLLOID

Initially, a protective colloid such as X-810, X-820 or Polymer C-3, all of which are alkali soluble, was added to the polyvinyl acetate sample before drying. Presumably, the protective colloid would be left surrounding the individual latex particles after the ice has been sublimed, thereby preventing the particles from fusing together as happens with straight polyvinyl acetate (see Sample 1). Also, the protective colloid would aid redispersion because of its solubility in water. All the sample runs from 1 through 59 were so treated.

For the most part, the films cast from the early sample runs had fair-to-good adhesion to glass. All were hard and brittle. Generally, the film surface was rough because of poor dispersion. Some of the samples had small quantities of 20% acetic acid added prior to drying. This method was soon discontinued since materials so dried deteriorated on storage and as a result their films were unsatisfactory. Other samples after drying had $(\text{NH}_4)_2\text{CO}_3$ added so that the redispersed system would be alkaline. Films from these systems (Samples 6, 7, 8, and 9) were very poor on all counts.

Because the films with good adhesion to glass were brittle, and the plasticizer (film conditioning agent) could not be present during the drying operation (Samples 11, 12, 33, and 34), dry blending a plasticizer into the

dried modified polyvinyl acetate was attempted. Neither Carbitol or dibutyl phthalate could be added directly to the dry powder (Samples 23 and 24) because they solvated the polyvinyl acetate so rapidly that an insoluble, gummy mass resulted. An attempt was made to dry blend the dibutyl phthalate into the extender pigments and to this add the dried polyvinyl acetate sample, dispersing agents, etc. A film from this system had no adhesion, cohesion, etc. However, the addition of a film conditioning agent to a previously dispersed polyvinyl acetate sample gives a film of excellent flexibility, adhesion, and water resistance.

Early attempts to add pigments and extenders to the modified latex gave films of unexpected results. Films from Samples 14 and 15 (both containing extender pigments) were excellent, while films from Samples 13, 16, and 18 (titanium dioxide pigment) were very poor.

Samples of WC-130 having higher and lower molecular weights than the standard material were investigated to see if molecular weight is a factor in obtaining a satisfactory dry polyvinyl acetate (see Samples 39, 40, 48, and 49). Both the low- and high-molecular-weight material gave similar films; however, the WC-130 having higher molecular weight seemed to be better than the material having low molecular weight.

The question of aging the material to insure distribution of all ingredients before drying was investigated (see Samples 41, 42, 53, and 54). In general, the results were the same as when the materials were not aged before drying.

Drying under basic conditions (Samples 45 and 46) was investigated to see if better water resistance and other film properties could be achieved. It was found that the films had good adhesion but were very water-sensitive. Dispersion was more difficult because the basic materials were harder to wet.

Latices other than WC-130 were investigated. Everflex G (Samples 33 and 34) and Rhoplex AC-33 (Samples 51 and 52) dried to a gummy, insoluble mass unsuitable for redispersion. Polymer Y (Samples 35, 36, and 38) dried and granulated easily; however, dispersion was unsatisfactory. Elvacet 81-900, which contains polyvinyl alcohol as a protective colloid, was dried (Sample 43). Results were satisfactory except that water resistance was poor. As a result, most of our work was done with WC-130, which gave us satisfactory results.

ELIMINATION OF PROTECTIVE COLLOID

1. By Washing and Use of Centrifuge.—Since the main function of a film conditioning agent is to solvate the resin particles, it was decided to

investigate the effect of the addition and removal of a film conditioning agent from the polyvinyl acetate latex prior to drying, the idea being to swell the resin particles and remove the film conditioning agent and other water soluble components, leaving the resin particles in a swollen condition. Carbitol was chosen as the conditioning agent instead of dibutyl phthalate because of its water solubility.

Initial attempts to dialyze this system were unsuccessful; therefore, the system was subjected to a centrifuge. The polyvinyl acetate system was washed and centrifuged repeatedly. Samples 62 and 64 (WC-130 plus 10% Carbitol based on the latex solids) and Sample 63 (WC-130) were treated this way prior to freeze drying. In each case the dried material powdered with very little effort and wet readily, even though the resultant dispersions contained agglomerated resin particles. The films had excellent water resistance and excellent adhesion to glass. The addition of a protective colloid (Sample 63) made the dried material more difficult to powder and lessened the degree of wetting. Increasing the protective colloid content (Samples 73 and 74) lowered film adhesion to glass and destroyed water resistance. These results suggest that a protective colloid is not required.

2. By Dialysis in Visking Dialysis Membrane.—Use of a Visking dialysis membrane resulted in successful dialysis of polyvinyl acetate systems. Sample 78 (WC-130 plus 10% Carbitol) was dialyzed for two days before freeze drying. The dried material was not gummy, as might be expected (see Sample 60), but could be powdered easily. It formed an excellent dispersion from which a very good film was made. The film was hard, clear, somewhat brittle, and had good adhesion to glass. Its surface was smooth because the resin particles, for all practical purposes, completely redispersed. Water resistance was good. The major difference between a polyvinyl acetate system which is dialyzed and one which has been washed and centrifuged is that the dialyzed material redisperses almost completely. As a result, a better or at least a smoother film can be made because agglomerated resin particles are not present. Water resistance of the dialyzed systems was lower, but this may have been caused by thinner films.

High- and low-molecular-weight samples of WC-130 (Samples 81 and 82) were dialyzed and also washed and centrifuged (Samples 88 and 89). In the case of dialysis, the high-molecular-weight material gave a slightly rougher surface, better adhesion, and better water resistance than the low-molecular-weight material. Washed and centrifuged high- and low-molecular-weight WC-130 gave similar films, with the low-molecular-weight material producing a somewhat smoother surface.

Dialyzed Samples 84 and 85 were freeze dried to see what effect, if any, Carbitol had on a pigmented system. As reported in Table 4, both samples formed very good films on glass; however, the water resistance of Sample 85

was poor instead of good. It appears that Carbitol detracts from the water resistance when a pigmented system is dialyzed.

The addition of glyoxal to the polyvinyl acetate system before and after dialysis was investigated to see if it would increase the water resistance by tying up any free hydroxyl groups on the Cellosize. Glyoxal increased the water resistance of Sample 98, although it also increased the difficulty in removing the dried material from the flask. Glyoxal added after dialysis (Sample 101) was detrimental to the resultant film.

Three paint formulations mentioned in our report "The Development of a Liquid Polyvinyl Acetate Paint," October, 1954, were dialyzed and freeze dried (Samples 94, 95A, and 96B). These paints had been aged from six to ten months. They dried and powdered satisfactorily and formed very good dispersions from which films were cast on glass. In each case the films were tough and had good-to-excellent adhesion to glass. Water resistance is excellent, although the films soften in time when in contact with water. These same aged paints, washed and centrifuged six times, are not satisfactory for redispersion (formulas 95B and 96B). When dry, they are very hard, making powdering for redispersion impossible. Sample 104 is the same as Sample 96A except that the paint was fresh. No observable differences in properties were noted, although dialysis of the fresh paint caused it to thicken appreciably.

It was expected that the dibutyl phthalate present in the aged polyvinyl acetate paints (Samples 94, 95A, and 96A) would cause the dialyzed material to dry into a gummy, insoluble mass as it does WC-130 (Samples 11 and 12). Since it did not, Sample 97 (WC-130 plus 10% dibutyl phthalate) was dialyzed three days to see if the dibutyl phthalate was removed. After drying, Sample 97 was tough and rubbery, indicating that at least part of the dibutyl phthalate did not dialyze. As a result, the effect of pigment on a polyvinyl acetate system containing dibutyl phthalate was investigated. In the case of Sample 110, where MetroNite BXXXX is incorporated into the system, the dibutyl phthalate imparts flexibility to the film and at the same time increases water resistance to a rating of excellent. Both samples powder satisfactorily and form dispersion from which films can be cast. Sample 111 is similar to 110 except that it is dialyzed as a unit. The properties are very similar. Both have flexibility and excellent water resistance and therefore are promising as starting points for compounding a "dry" polyvinyl acetate paint.

Samples 116, 117, 118, and 119 all point to the fact that dibutyl phthalate can be present during dialysis, providing a pigment is present. Dibutyl phthalate cannot be added to dialyzed polyvinyl acetate (Sample 108) and obtain a film having flexibility and water resistance. Possibly the pigments absorb the excess dibutyl phthalate not used in solvating the latex.

Various dispersing agents were added to polyvinyl acetate systems to

aid dispersion of the dry powder. In general, they had little effect on the dispersion or the resultant film. However, Tergitol NP-35 did impart flexibility to a film, as does dibutyl phthalate or any film conditioning agent (see Samples 102, 105, and 106). However, the Tergitol NP-35 detracted from the water resistance. This can be seen by comparing pigmented Samples 116 and 117 with 118 and 119, respectively. Either one of these would serve as excellent starting points from which to build a dry polyvinyl acetate paint.

EFFECT OF DRY POLYVINYL ACETATE ON BONDEX AND CEMENT

A check of the relative merits of dialyzed WC-130 (Sample 83) and washed and centrifuged WC-130 (Sample 77) was made with Bondex and cement applied to prewet cement blocks and asbestos shingles. Varying quantities of the dried material were added to the Bondex and cement. From the results listed in Table 1, it appears that the quantity of lime present in the Bondex controls the amount of dry polyvinyl acetate needed to eliminate dusting of the coating. Straight cement required 10% of Sample 83 to prevent dusting, while 15% of the same material added to Bondex did not prevent dusting.

Both the compounded polyvinyl acetate paint and the polyvinyl acetate formula No. 8 (both liquid and freeze dried) were included in the investigation and are shown in Table 1.

TABLE 1

EFFECT OF DRY POLYVINYL ACETATE ON BONDEX AND CEMENT

<u>System</u>	<u>Observations</u>
1. Cement plus 15% Sample 83	No dusting. Good adhesion and film cohesion.
2. Cement plus 25% Sample 83	No dusting. Good adhesion and film cohesion.
3. Cement plus 25% Sample 77	No dusting. Good adhesion and film cohesion.
4. Bondex plus 25% Sample 77	No dusting. Good adhesion and film cohesion.
5. Bondex plus 15% Sample 83	Slight dusting when rubbed.
6. Bondex plus 25% Sample 83	No dusting. Good adhesion and film cohesion.
7. Bondex	Dusted when rubbed.
8. Cement	Dusted when rubbed.
9. Cement plus 10% Sample 83	No dusting. Good adhesion and film cohesion.
10. Cement plus 5% Sample 83	Dusted when rubbed.
11. Dry Paint No. 1	No dusting. Smooth, adherent film.
12. Formula No. 8	No dusting. Smooth, adherent film.
13. Sample No. 96 (Formula 8 Dry)	No dusting. Adherent film.

TABLE 1 (continued)

The formulation of the dry paint used in system 11 follows:

<u>System</u>	<u>Grams</u>
TiPure R-510	2.5
ASP 400	0.94
BXXXX (MetroNite)	1.0
WP-300 (Cellosize)	0.025
Daxad 11	0.025
Daxad 23	0.0125
Dry PVA (Sample 83)	2.0
Water	5.0

When applied to glass the paint (dry) showed good adhesion although it was somewhat brittle. The surface was rough because of occluded air. The water resistance of the film was fair.

CONCLUSIONS

Our experimental work has shown that a polyvinyl acetate properly treated can be transformed into a useful powder by freeze drying. However, dry polyvinyl acetate has an affinity for water; therefore, the material must be protected to prevent possible blocking.

Freeze-dried polyvinyl acetate systems like Samples 116 or 117 offer excellent starting points from which to build a dry polyvinyl acetate paint. These systems which incorporate dialysis of a preplasticized polyvinyl acetate with an extender pigment give a film having excellent adhesion to glass, toughness, flexibility, surface smoothness, and excellent water resistance.

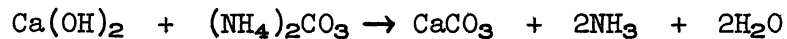
In addition, dry polyvinyl acetate latex can be used as an additive to materials like Bondex, cement, etc. The coating is improved, although fair percentages of the dry polyvinyl acetate are necessary. Since straight cement required less dried polyvinyl acetate to prevent dusting than Bondex (see Table 1), it appears that the large quantity of lime in the Bondex is responsible for the dusting of the coating. As a result, means of counteracting this lime were investigated and are reported under the following section, "Surface Treatment of Bondex."

Based upon the promising results and the potential market for a dry polyvinyl acetate latex and paint, further work, particularly formulation and testing of a dry polyvinyl acetate paint, appears to be justified.

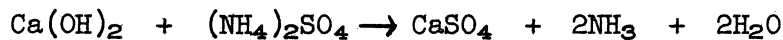
SURFACE TREATMENT OF BONDEX

While working with Bondex we found that the surface dusted regardless of whether a one- or two-coat system was applied. However, spraying water on the Bondex surface eliminated this dusting phenomenon. Because water was beneficial, it seemed advisable to observe the effect various salt solutions might have on Bondex.

Two salts were used: ammonium carbonate and ammonium sulfate. The general idea was that the ammonium carbonate would react with the lime in the Bondex to form crystals of calcium carbonate, which would harden the surface. The equation for this reaction follows:



Limestone, CaCO_3 , is inert and therefore does not undergo any further reaction with the Bondex. However, when ammonium sulfate is used, it reacts with the lime to form gypsum. Some of the gypsum, either in the calcined or uncalcined form, acts by combining with the tricalcium aluminate in the Portland cement to form a complex sulfoaluminate. This sulfoaluminate is very insoluble and precipitates, thus taking the $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ out of solution and rendering it unavailable for crystal formation. The equations for these reactions follow:



Six weathered asbestos shingles (three sandblasted and three washed) were coated with two coats of Bondex one hour apart. Before applying the Bondex each shingle was thoroughly wet. Before the second coat of Bondex was applied, each shingle was again moistened to facilitate ease of application. Each shingle was allowed to dry four hours and then sprayed with water, ammonium carbonate, or ammonium sulfate. Sufficient spray was given each shingle so that the Bondex surface was thoroughly wet. The shingles were allowed to age ten days before they were scrubbed on the Gardner Model 105 Straight Line Washability and Abrasion Machine. Water was used for the scrubbing test to

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provide moderate lubrication for the bristle brush. Results of the washability tests are listed below.

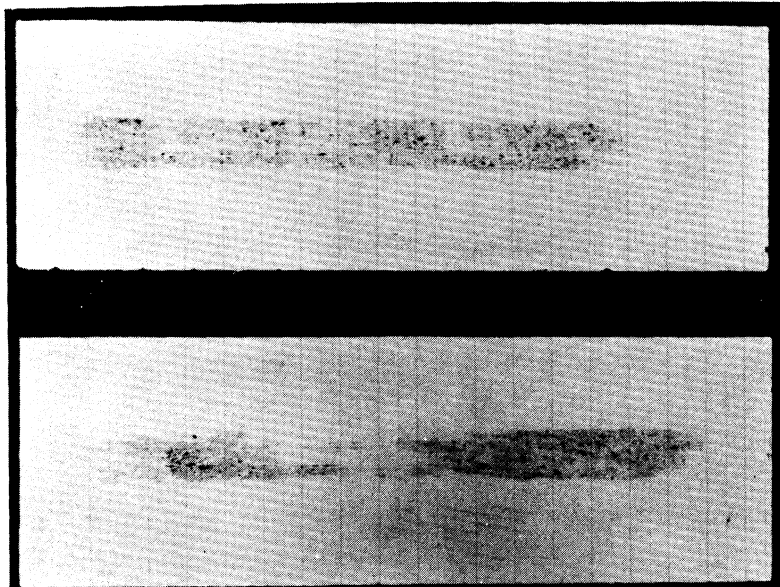
TABLE 2

WASHABILITY OF SURFACE-TREATED BONDEX

<u>Sample No.</u>	<u>Description</u>	<u>Results</u>
1	Two coats of Bondex applied to washed asbestos shingle, followed by water spray.	Scrubbed 25 cycles. Coating completely removed. Most of the damage was done during the first 10 cycles.
2	Two coats of Bondex applied to sandblasted asbestos shingle, followed by a water spray.	Scrubbed 45 cycles. Coating began to break down after 20 cycles.
3	Two coats of Bondex applied to washed asbestos shingle, followed by a spray of concentrated ammonium carbonate.	Coating 50% destroyed after scrubbing 100 cycles. Break-down began after 50 cycles.
4	Two coats of Bondex applied to sandblasted asbestos shingle, followed by a spray of concentrated ammonium carbonate.	Scrubbed 500 cycles. Surface of coating barely broke down except for one end. This end wore away before 200 cycles, but further scrubbing was not detrimental to the coating.
5	Two coats of Bondex applied to washed asbestos shingle, followed by a spray of concentrated ammonium sulfate.	Scrubbed 1000 cycles. Coating did not break down.
6	Two coats of Bondex applied to sandblasted asbestos shingle, followed by a spray of concentrated ammonium sulfate.	Scrubbed 2500 cycles. Coating did not break down.

Photographs of the above shingles after being scrubbed are found on this and the following page.

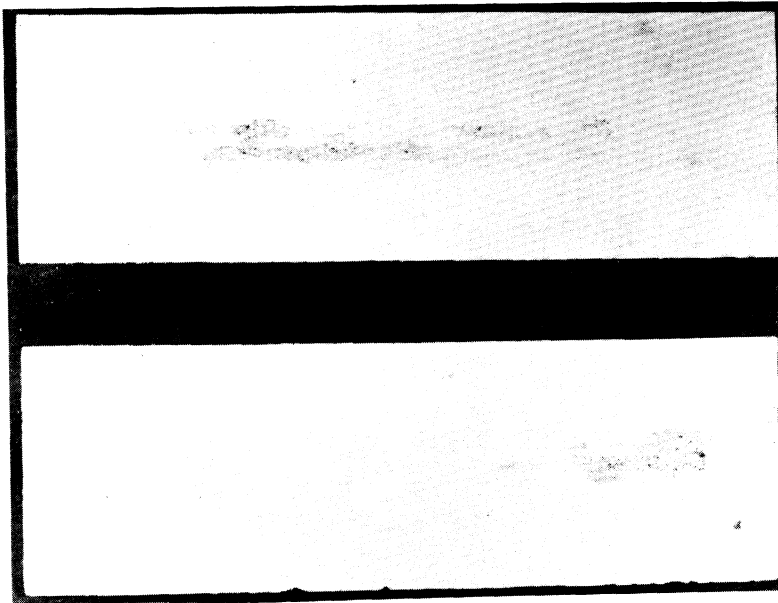
From these data it is evident that the water resistance and abrasion resistance of a Bondex coating can be increased manyfold by spraying the coating with a compound such as ammonium sulfate, which will react with the lime. Some of the gypsum formed renders insoluble complexes while the rest forms crystalline gypsum which hardens the Bondex and also acts as a binding agent. Therefore, by utilizing a common practice of the cement industry, it has been possible to produce a more dense Bondex film which has better cohesive and adhesive properties. A comparison of formulas 1 and 2 with 5 and 6 in the experimental work bears this out.



Panel 1
Scrubbed 25 cycles

Panel 2
Scrubbed 45 cycles

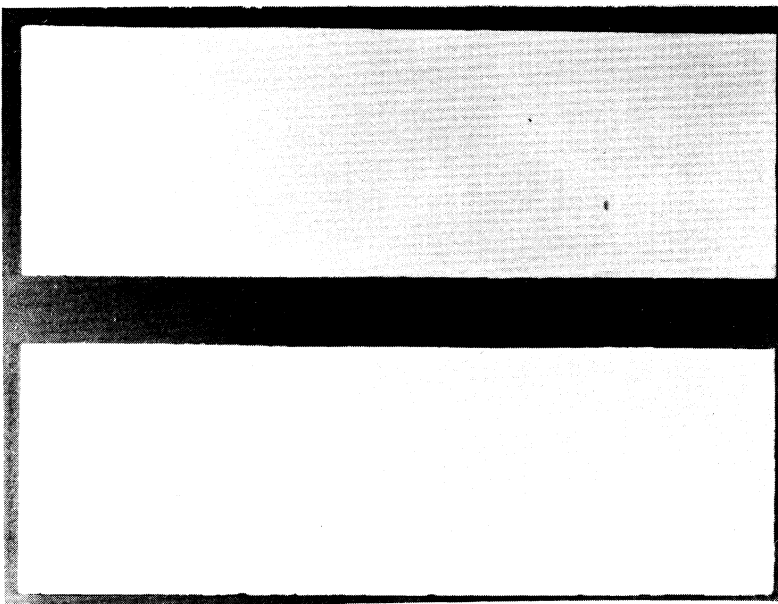
Figure 1. Bondex surface sprayed with water.



Panel 3
Scrubbed 100 cycles

Panel 4
Scrubbed 500 cycles

Figure 2. Bondex surface sprayed with ammonium carbonate.



Panel 5
Scrubbed 1000 cycles

Panel 6
Scrubbed 2500 cycles

Figure 3. Bondex surface sprayed with ammonium sulfate.

SUPPLEMENT ON THE DEVELOPMENT OF A LIQUID POLYVINYL ACETATE PAINT

Supplementing the report on the Development of a Liquid Polyvinyl Acetate Paint, dated October 1954, we have listed below the external exposure results as of June 30, 1955 of the polyvinyl acetate paints exposed on the roof of the East Engineering Building, University of Michigan. These paints were applied in one- and two-coat systems on asbestos shingles which had been weathered for five years. The asbestos shingles were prewet before the polyvinyl acetate paints were applied in order to prevent pinholing. The panels were held in a test rack at an angle of 45° facing toward the south. The formulation of formulas 5, 6A, and 8 are listed in the glossary; the other formulations may be found in the report on the Development of a Liquid Polyvinyl Acetate Paint.

TABLE 3

EXTERNAL EXPOSURE RESULTS OF WATER BASE PAINTS

Formulation	Blistering				Chalking
	9 Months Exposure		12 Months Exposure		
	1 coat	2 coats	1 coat	2 coats	
1	badly	slightly	severely	badly	yes
2	badly	slightly	badly	badly	yes
3	some	none	badly	none	yes
4	none	none	none	none	
5	severely	some	severely	badly	yes
6a	none	none	slightly	none	yes
6b	none	none	slightly	none	yes
6c	none	none	slightly	none	yes
	7 Months Exposure		10 Months Exposure		
6d	some	none	slightly	slightly	yes
7	none	none	none	none	yes
8	none	slightly	none	badly	yes

The film integrity of formulas 4 and 7 is good. Formula 4 is very white in color and appears to be the best paint applied to the asbestos shingles. No explanation is offered for the blistering of the two coats of formula 8. The single coat of formula 8 does not show this type of failure. It is interesting to note that the paint rack itself, coated with different systems of polyvinyl acetate and white lead, has stood up very well. Those sections painted with white lead, both primer and top coat, are considerably darker than the others. All the wood regardless of the system used showed checking. The whitest system was the polyvinyl acetate primer acrylic top coat.

SUPPLEMENT ON PAINT AND VARNISH REMOVERS

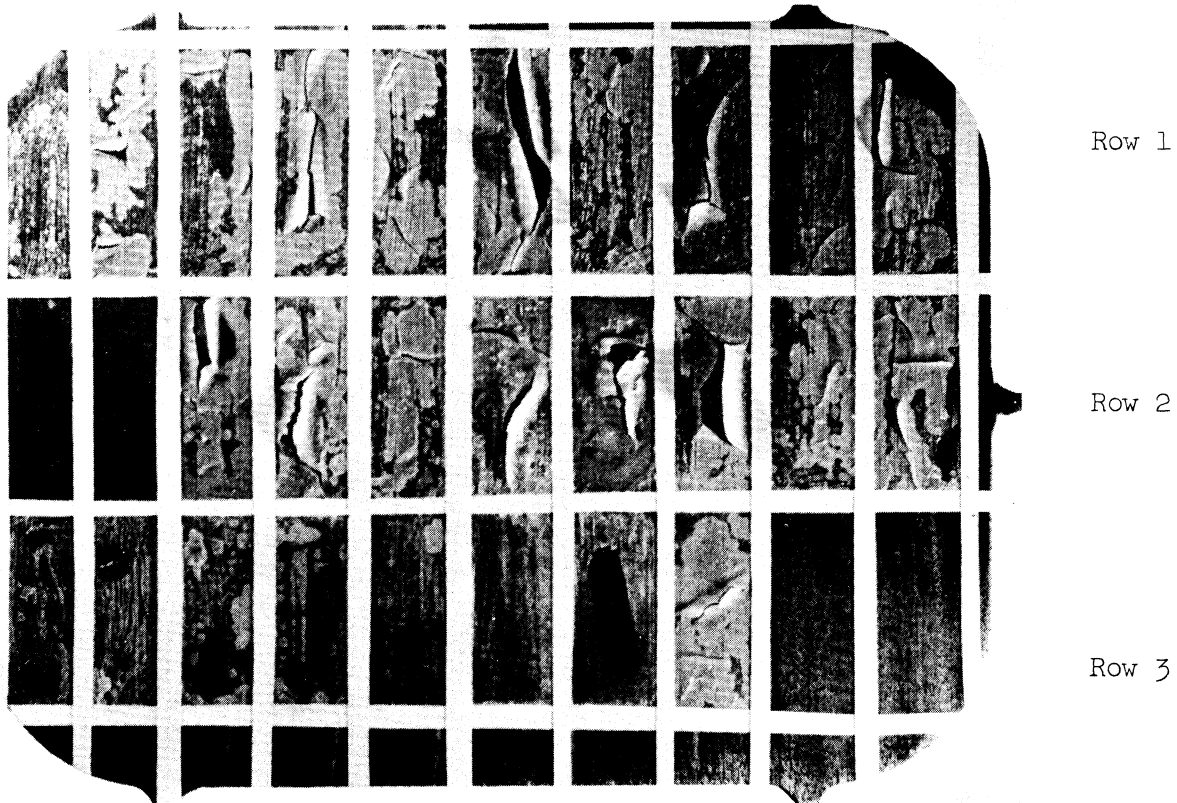
The report on the Development of Paint and Varnish Removers issued October, 1954 compared the performance characteristics of 25 commercial paint and varnish removers and 39 laboratory formulations. The differences in the performance of these paint and varnish removers were observed after they were applied to panels finished with clear alkyd urea furniture varnishes. At the time of the report, pictures of the test panels, particularly panels A and D, were not available. Pictures of these two panels are included in this report on pages 15 and 16.

Almost all the laboratory paint and varnish removers were applied to panel A for comparison purposes. Panel D gives a comparison of the eight best commercial paint and varnish removers with the better laboratory-compounded paint and varnish removers. Of the better commercial removers applied to panel D, only formulas C, T, and V, represented by J. B. Day Company's Kut-Kote Remover, Universal Technical Products' Universal Remover, and Dux Paint and Chemical Company's Atomic Remover, respectively, gave satisfactory results. On the other hand, many of the laboratory formulations such as formulas 23, 27, 28, 29, 35, 36, 37, and 39 were equivalent to or better than the best of the commercial paint and varnish removers tested.

It should be noted that a clear alkyd urea furniture varnish such as used on the test panels provides an excellent finish for comparing paint and varnish removers. Oleoresinous-type finishes were unsatisfactory because all the paint and varnish removers, commercial and laboratory, gave satisfactory results. Most of the commercial paint and varnish removers tested had little or no effect on the alkyd urea furniture finish (see picture of panel D, page 16), while almost all the laboratory formulations gave satisfactory results. A general laboratory formulation found to be very satisfactory follows:

	<u>Parts by Weight</u>
Methylene chloride	177 to 172
Paraffin	2
Methocel 4000 HG	3 to 4
Methanol	12
Active solvent	6
Water	4 to 6

The solvent may be Carbitol, Cellosolve acetate, Tetralin, cyclohexanol, furfural, or combination of same. More detailed results may be found in the report on the Development of Paint and Varnish Removers.



Panel A

Figure 4. Comparison of laboratory paint and varnish formulations (left to right).

<u>Row 1</u>	<u>Row 2</u>	<u>Row 3</u>
*Formula 10	*Formula 20	*Formula 30
9	19	29
8	18	28
7	17	27
6	16	26
5	15	25
4	14	24
3	13	23
2	12	22
1	11	21

*Formulations may be found on pages 9 and 10 in the report on Development of Paint and Varnish Removers, issued October 1954.



Panel D

Figure 5. Comparison of commercial and laboratory-compounded paint and varnish removers (left to right).

<u>Row 1</u>	<u>Row 2</u>	<u>Row 3</u>
*Formula C	*Formula 36	*Formula 5
E	37	6
Q	3	15
S	13	28
T	23	29
U	17	33
V	35	14
W	27	38 (multiple coats)
39	9	--

*For formulations see Tables I and III in report on Development of Paint and Varnish Removers, issued October 1954.

APPENDIX

TABLE 4
EXPERIMENTAL FREEZE-DRIED MODIFIED POLYVINYL ACETATE SYSTEMS

Sample Run No.	Components	pH	Observations
1	WC-130		Material dried into an insoluble, unpowderable mass.
2	WC-130	35.0 gm	A hard and brittle film with good adhesion to glass was formed.
	X-810 10% NH ₄ OH	3.2 gm	
3	WC-130	43.5 gm	Material powdered easily. Films made with and without the addition of (NH ₄) ₂ CO ₃ had an orange-peel effect. Cause was poor dispersion.
	X-810 10% NH ₄ OH	4.2 gm	
	20% acetic acid	10.0 cc	
4	WC-130	42.9 gm	Film was very rough because of agglomerated resin particles. It had good adhesion to glass.
	X-810 10% NaOH	4.2 gm	
5	WC-130	42.0 gm	Material powdered easily. Film, which was hard and brittle, had good adhesion to glass.
	X-810 10% NaOH	4.6 gm	
	20% acetic acid	8.0 cc	
6	WC-130	32.1 gm	Very poor film formed. Poor adhesion to glass. Making system alkaline with ammonium carbonate resulted in the same kind of poor film.
	X-820 10% NH ₄ OH	3.3 gm	

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
7	WC-130	3.4	A hard but brittle film with fair adhesion to glass formed. Making system alkaline with ammonium carbonate resulted in a film with poor adhesion, etc.
	X-820 10% NH ₄ OH		
	20% acetic acid		
8	WC-130	5.5	A hard but brittle film with fair adhesion to glass formed. Film is white rather than translucent. Surface of film is severely checked. Making system alkaline with ammonium carbonate resulted in a film with poor adhesion, no cohesion, and no checking.
	X-820 10% NH ₄ OH NPX		
9	WC-130	3.3	A hard but brittle film with fair adhesion to glass formed. Film is not smooth but is translucent. Making system alkaline with ammonium carbonate resulted in a film with poor adhesion, etc.
	X-820 10% NH ₄ OH NPX		
	20% acetic acid		
10	WC-130	5.5	A hard but brittle film with good adhesion to glass formed. Film slightly more translucent than Sample No. 9.
	X-820 10% NH ₄ OH NPX		
	Water		
11	WC-5510	3.9	On drying, the system fused into an insoluble, unpowderable mass.
	X-820 10% NH ₄ OH NPX		
12	WC-5510	3.4	On drying, the system fused into an insoluble, unpowderable mass.
	X-820 10% NH ₄ OH NPX		
	20% acetic acid		

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
13	TiPure R-510	3.0	Very poor film formed. No adhesion to glass. Considerable evidence of checking.
	WC-130		
	X-810 10% NH ₄ OH 20% acetic acid		
14	MetroWhite BXXXX	6	A hard but brittle film with good adhesion to glass formed. Resultant film was smooth and somewhat porous, like that of commercial poly-vinyl acetate paints.
	WC-130		
	X-810 10% NH ₄ OH		
15	ASP 400	6	A hard but brittle film with good adhesion to glass formed. Film is porous, as was No. 14.
	WC-130		
	X-810 10% NH ₄ OH		
16	TiPure R-510	5	Very poor film formed. No adhesion or cohesion. Film mud-cracked, as did Sample No. 13.
	WC-130		
	X-810 10% NH ₄ OH		
	Water		
17	WC-130	3.3	A hard but somewhat brittle film with good adhesion to glass was formed. Film was translucent and quite porous.
	X-810 10% NH ₄ OH		
	20% acetic acid		
18	TiPure R-510	5	Very poor film formed. No adhesion to glass. Severe mud-cracking resulted.
	WC-130		
	X-820 10% NH ₄ OH NPX		
	Water		

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
19	MetroNite BXXXX		Film that resulted had much poorer adhesion to glass than anticipated. Mud-cracking still prevalent. Addition of ammonium carbonate to the system resulted in a poor film. Mud-cracking did not occur, however.
	TiPure R-510	5.2	
	WC-130		
	X-810 10% NH ₄ OH		
20	Nyral 300		Film formed on glass was rough and contained air bubbles. Had fair-to good-adhesion to glass.
	WC-130	5.5	
	X-810 10% NH ₄ OH		
	Water		
21	WC-130		For ultimate dry blending.
	X-810 10% NH ₄ OH	3.3	
22	WC-130		Used in experiments with Bondex and compounded cement paints.
	X-810 10% NH ₄ OH	3.3	
23	WC-130		Used for dry blending with Carbitol.
	X-810 10% NH ₄ OH	3.3	
24	WC-130		Used for dry blending with dibutyl phthalate.
	X-810 10% NH ₄ OH	3.3	
25	WC-130		Film is rough because of excessive foaming. Has fair-to-good adhesion to glass, although brittle.
	X-810 10% NH ₄ OH	5	
26	WC-130		Film had good adhesion to glass in addition to having fair cohesion. Film was not hard and brittle as with the other samples.
	10% C-3	4.2	

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
27	WC-130	5	A hard but brittle film with good adhesion to glass formed.
	X-810 10% NH ₄ OH NPX		
28	WC-130	3.4	Film shows good to excellent adhesion to glass.
	X-810 10% NH ₄ OH NPX		
	20% acetic acid		
29	WC-130	3.4	Same as Sample No. 28. For ultimate dry blending.
	X-810 10% NH ₄ OH NPX		
	20% acetic acid		
30	WC-130	3.5	Same as Sample No. 28. For ultimate dry blending.
	X-810 10% NH ₄ OH NPX		
	20% acetic acid		
31	WC-130	4.3	A hard but brittle film with fair adhesion to glass was formed. Film was white instead of translucent.
	X-810 10% NH ₄ OH		
32	WC-130	4.1-4.2	Same material as Sample No. 26.
	10% C-3		
33	Everflex G	4	Material dried to a gummy, insoluble, unpowderable mass.
34	Everflex G	5	Material dried to a gummy, insoluble, unpowderable mass.
	X-820 10% NH ₄ OH NPX		

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
35	Darex Polymer Y 10% C-3	6 35.0 gm 3.5 gm	Material could not be dispersed satisfactorily; hence, a very poor film having a granular appearance and no adhesion to glass was formed. The addition of ammonium carbonate made a slurry possible; however, the film results were the same.
36	Darex Polymer Y 10% C-3 K-707	35.0 gm 3.5 gm	A film with a rough surface and fair adhesion to glass was formed. Film was granular in appearance. Addition of ammonium carbonate aided dispersion of the material. Resultant film was the same except that its adhesion to glass was slightly better.
37	WC-130 X-810 10% NH ₄ OH NPX K-707	14.7 gm 0.7 gm 0.7 gm	5.5 A hard film, white in color, with fair-to-good adhesion to glass was formed. The K-707 caused the dispersion to foam. Film is water soluble.
38	Polymer Y	5.9	Material granulates readily and is difficult to wet. Satisfactory slurry not obtained. Film formed on glass is granular and has very poor adhesion to glass. Addition of ammonium carbonate makes wetting of the material easier; however, foaming occurs. Film results are identical to original results.
39	WC-130 H.M.W. X-810 10% NH ₄ OH 20% acetic acid	13.1 gm 1.3 gm 2.2 cc	3.6 Film very white in color; fair adhesion to glass. It developed very noticeable mud cracks.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
40	WC-130 L.M.W.	3.5	Surface of film having a white appearance was severely mud-cracked. Film had fair adhesion to glass.
	X-810 10% NH ₄ OH		
	20% acetic acid		
41	WC-130	5.5	Sample aged 3 days before drying. Dried material is very difficult to powder; hence, dispersion of material is poor. A fairly hard and brittle film with good adhesion to glass was formed. Film, translucent in color, has a rough surface caused by poor dispersion. Water is detrimental to the film even though it has some cohesive properties.
	X-810 10% NH ₄ OH		
42	WC-130	5.4	Sample aged 3 days before drying. Dried material powdered with minimum difficulty. Film formed on glass had a smooth surface, translucent appearance, good adhesion, but no cohesive properties. Film is very water sensitive.
	X-810 10% NH ₄ OH		
	20% acetic acid		
43	Elvacet 81-900	5.4	Dried material is extremely difficult to powder; hence, dispersion is poor. Film formed on glass is rough, translucent, and shows good adhesion. It is also tough, i.e., not as brittle as WC-130. Film is affected by water, but not to the extent anticipated.
	X-820 10% NH ₄ OH NPX		
44	Elvacet 81-900	4.2	Unable to redisperse the material and cast a film because the dried material could not be powdered.
	10% polyvinyl alcohol 72-60		

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
45	WC-130 X-810 10% NH ₄ OH Conc. NH ₄ OH	17.7 gm 1.8 gm 0.8 cc 10.3	Dried material powders satisfactorily but does not wet readily, making dispersion very difficult. Film formed on glass has good adhesion and a rough surface. Film brittleness is less than an acidic film. Film is more water sensitive than an acidic film.
46	WC-130 X-810 10% NH ₄ OH 20% conc. NH ₄ OH	23.4 gm 2.3 gm 1.0 cc 8.1	Dried material powders satisfactorily but lumps remain and are present in the film. Film, with good adhesion to glass, is hard and quite brittle. It is very water sensitive.
47	WC-130 10% polyvinyl alcohol 72-60	28.8 gm 3.0 gm 4.0	Dried material wets easily but disperses poorly. Film formed on glass had good-to-excellent adhesion although it was not continuous. Film was slightly white, hard, and rather brittle. Water softens the film rather than dissolves it.
48	WC-130 H.M.W. X-810 10% NH ₄ OH	29.1 gm 2.9 gm 5.2	Powdered material wets but does not disperse well. Film is very tough and has good adhesion to glass. Its surface is translucent and quite rough. Water resistance is better than average although water softens the film.
49	WC-130 L.M.W. X-810 10% NH ₄ OH	24.6 gm 2.5 gm 5.2	Powdered material wets but does not disperse well. Film is fairly tough and has good adhesion to glass. Surface is translucent and quite rough. Water resistance is fair. Film softens in contact with water.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
50	WC-130 X-820 10% NH ₄ OH NPX 10% polyvinyl alcohol 72-60	4.3	A hard and brittle film with good adhesion to glass is formed. Surface is translucent and rough. Water resistance is above average, although film softens when in contact with water.
51	Rhoplex AC-33	7.8	Material, in drying, formed a partial film, making redispersion impossible.
52	Rhoplex AC-33 X-810 10% NH ₄ OH	7.4	Material dried to a gummy, insoluble mass unsuitable for redispersion.
53	WC-130 X-810 10% NH ₄ OH	5.3	This sample aged 7 days before drying. Dried material difficult to powder. It wets fairly well, but lumps of resin remain in the poor dispersion. Film formed is hard and brittle with good-to-excellent adhesion to glass. Surface is rough. Film is water sensitive.
54	WC-130 X-810 10% NH ₄ OH 20% acetic acid		Material after aging 7 days is partially coagulated. Material did not dry satisfactorily. No film cast from this material.
55	Elvacet 81-900 X-810 10% NH ₄ OH Daxad 11	6.2	Dried material powders quite easily but wets poorly. Resultant dispersion contained resin particles and was foamy. Film formed on glass was tough (not brittle) and very rough. It has fair-to-good adhesion to glass. Water dissolves the film.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
56	Elvacet 81-900 X-820 10% NH ₄ OH NPX Conc. ammonium hydroxide	27.0 gm 2.7 gm 1.0 cc 10.2	Dried material is hard, powders with difficulty, but wets well. A hard and brittle film with good adhesion to glass was formed. Film was very rough (caused by resin particles) and translucent. Water dissolves the film.
57	WC-130 X-810 10% NH ₄ OH	29.2 gm 2.9 gm 5.5	Material to be used for dry blending.
58	MetroNite BXXXX WC-130 Water X-810 10% NH ₄ OH	20.7 gm 21.0 gm 2.0 cc 2.1 gm	Dried material powders satisfactorily. To be used for ultimate dry blending.
59A	WC-130 5% Cellosize WP-300	47.8 gm 11.0 gm 4.1	Material aged two days before drying. Dried material powders satisfactorily, wets readily, but dispersion contains resin lumps. A brittle film with fair adhesion to glass was formed. Film was slightly rough and translucent. Water softens the film.
59B	Sample 59A X-820 10% NH ₄ OH NPX	20.9 gm 2.1 gm 5.1	Material aged five days before drying. Dried material powders easily, wets readily, disperses well, but foams. A brittle film, white in color, was formed on glass. It had no adhesion to glass. Surface of the film was white.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
60	WC-130 Carbitol	78.2 gm 4.7 gm	3.8 Method used to dialyze sample was unsatisfactory. Material dried to a gummy, insoluble mass.
61	Sample 60 X-810 10% NH ₄ OH	28.1 gm 2.8 gm	5.5 Material dried to a gummy, insoluble mass.
62	WC-130 plus Carbitol (10% based on latex solids). (System washed and centrifuged 6 times.) To this X-820 10% NH ₄ OH NPX was added.		Basic screening test. See Sample 68 for results on a controlled sample.
63	WC-130 (System washed and centrifuged 6 times).		Basic screening test. See Sample 65 for results on a controlled sample.
64	WC-130 plus Carbitol (10% based on latex solids). (System washed and centrifuged 6 times).		Basic screening test. See Sample 67 for results on a controlled sample.
65	WC-130 (system washed and centrifuged 6 times). Water added to the washed system to reconstitute the material to a solids content approximating the original.		Dried material powders satisfactorily into fine granules. These wet readily. Film is clear in color. Surface of film is rough because of agglomerated resin particles. Film is tough and has excellent adhesion to glass. Water resistance is excellent.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
66	Sample 65 X-820 10% NH ₄ OH NPX	9.5 gm 0.95 gm	Material powders easily and wets satisfactorily. It requires agitation to obtain any degree of dispersion. Film is partially white in color. Surface is rough because of the resin agglomerates. Film has excellent adhesion to glass, is hard and slightly brittle. Water resistance is excellent.
67	WC-130 plus Carbitol (10% on the latex solids). (System washed and centrifuged 6 times.) Water added to the washed system to reconstitute the material to a solids content approximating the original.		Material powders easily and has the appearance of powdered sugar. It wets readily. Film is clear in color. Surface is rough. Film is check-free, hard, slightly brittle, and has excellent adhesion to glass. Water resistance is excellent.
68	Sample 67 X-820 10% NH ₄ OH NPX	10.9 gm 1.1 gm	Material powders satisfactorily but is not like powdered sugar (see Sample 67). It wets satisfactorily. Film is partially white in color. Surface is somewhat rough. Film, which is hard but not brittle, has excellent adhesion to glass. Water resistance is excellent.
69	Elvacet 81-900 (system washed and centrifuged 6 times). Water added to the washed system to reconstitute the materials to a solids content approximating the original.		Material powders easily into fine granules (not like powdered sugar). It wets satisfactorily and disperses very well. Film is clear in color. Surface is rough, caused by air bubbles instead of agglomerated resin particles. Film, which is hard and quite brittle, has excellent adhesion to

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
70	Sample 69 X-820 10% NH ₄ OH NPX	10.0 gm 1.0 gm	<p>glass. Water resistance is fair-to-good, although water tends to dissolve the film.</p> <p>Material powders easily but does not produce fine granules (Sample 69). It wets slowly. Film is white in color. Surface is very rough because of agglomerated resin particles. Film is partly checked. It has good adhesion even though it is brittle and hard. Water resistance is poor.</p>
71	Elvacet 81-900 plus Carbitol (10% on latex solids). (System washed and centrifuged 6 times). Water added to reconstitute material to 60% solids.		<p>Material powders easily into fine granules. It wets satisfactorily, forming an excellent dispersion containing entrapped air. Film is clear in color. Surface is very smooth except for occluded air bubbles. Film, which is quite hard and brittle, has excellent adhesion to glass. It has poor water resistance, dissolving when in contact with water.</p>
72	Sample 71 X-820 10% NH ₄ OH NPX	10.0 gm 1.0 gm	<p>Material powders easily into fine granules resembling powdered sugar. It wets readily although resin agglomerates are present in the dispersion. Film is almost clear in color. Surface is rough because of the agglomerated resin particles. Film, which is hard and quite brittle, has excellent adhesion to glass. Water softens the film.</p>

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
73	WC-130 plus Carbitol (10% on latex solids).	19.0 gm	Material powders easily into fine granules. It wets readily; however, dispersion contains resin agglomerates. Film is white in color. Surface is rough because of the agglomerated resin particles. Film, which is hard and brittle, has good adhesion to glass. Water resistance is poor.
	X-820 10% NH ₄ OH NPX (System washed and centrifuged 6 times). Water added to reconstitute material to 47.5% solids.	1.5 gm	
74	Sample 73	12.0 gm	Material powders easily into fine granules. It wets much slower than Sample 73. Dispersion is very poor. Film is white. Film has no adhesion to glass and is severely checked throughout.
	X-820 10% NH ₄ OH NPX	3.0 gm	
75	WC-130 plus 10% Carbitol Unsuccessful dialysis attempt (not using a dialysis membrane).		Dried material adhered together and developed a yellow color on standing.
76	MetroNite BXXXX	25.8 gm	Material easily removed from flask after drying. Powders with some difficulty but wets readily. <u>Type of Film Formed on Glass</u> Color - white; surface - very rough. Film has excellent adhesion to glass and is very hard and tough. Water resistance is excellent.
	WC-130 plus 10% Carbitol based on resin solids. (Washed and centrifuged 8 times.)	39.0 gm	
77	WC-130 (Washed and centrifuged 8 times.)		Material same as Sample 65 used as an additive to Bondex.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
78	WC-130 plus 10% Carbitol based on resin solids. (Material dialyzed two days in Visking membrane.)		Material powders easily and wets satisfactorily. Forms an excellent dispersion with very few resin particles present. <u>Type of Film Formed on Glass</u> Color - clear; surface - very smooth. Film, which is hard and brittle, has good adhesion to glass. Water resistance is fair to good.
79	WC-130 plus 10% Carbitol based on resin solids. (Material dialyzed four days in Visking membrane.)		Material powders easily into very fine granules similar to powdered sugar. It wets satisfactorily but foams somewhat. <u>Type of Film Formed on Glass</u> Color - clear; surface - very smooth. Film is tough and has excellent adhesion to glass. Water resistance is fair to good.
80	WC-130 (Dialyzed for two days in Visking membrane.)		Similar to Sample 78 used as an additive to Bondex.
81	WC-130, high molecular weight. (Dialyzed for two days in Visking membrane.)		Dried material more difficult to remove from flask than Sample 80. Material powders into fine granules which wet satisfactorily. Dispersion has some foam but is almost free of resin particles. <u>Type of Film Formed on Glass</u> Color - clear; surface - slightly rough. Film, which is hard and brittle, has excellent adhesion to glass. Water resistance is fair to good.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
82	WC-130, low molecular weight. (Dialyzed two days in Visking membrane.)		Material powders easily into fine granules which in turn wet satisfactorily. <u>Type of Film Formed on Glass</u> Color - clear; surface - smooth. Film, which is hard and brittle, has good adhesion to glass. Water resistance is poor.
83	WC-130 (Dialyzed five days in Visking membrane.)		Similar to Sample 80. Will be used as an additive to Bondex.
84	MetroNite BXXXX WC-130 Water (Dialyzed two days in Visking membrane.)	36.8 gm 29.2 gm 5.0 cc	Material powders satisfactorily. It wets fairly well and forms a foamy dispersion. <u>Type of Film Formed on Glass</u> Color - white; surface - fairly smooth. Adhesion to glass is excellent. Water resistance is good.
85	MetroNite BXXXX WC-130 plus 10% Carbitol Based on resin solids Water (Dialyzed two days in Visking membrane.)	36.8 gm 29.2 gm 5.0 cc	Material powders satisfactorily, somewhat easier than Sample 84. It wets well and forms a foamy dispersion. <u>Type of Film Formed on Glass</u> Color - white; surface - smooth. Adhesion is excellent. Water resistance is poor.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
86	Elvacet 81-900 (Dialyzed two days in Visking membrane.)		Material does not powder well and does not wet. In order to make a dispersion satisfactory for casting a film the material was ground in a mortar. Film that resulted was clear, had a rough surface, good adhesion to glass, and very poor water resistance.
87	Elvacet 81-900 plus 10% Carbitol based on resin solids (Dialyzed two days in Visking membrane.)		Material was more difficult to powder than Sample 86 and did not wet. In order to make a satisfactory dispersion, material was ground in a mortar. Film that resulted was clear, had a fairly rough surface and good adhesion to glass but very poor water resistance.
88	WC-130, high molecular weight. (Washed and centrifuged 6 times.)		Material powders easily and wets readily. However, dispersion contains agglomerated resin particles. <u>Type of Film Formed on Glass</u> Color - clear; surface - very rough. Film, which is hard and tough, has excellent adhesion to glass. Water resistance is excellent.
89	WC-130, low molecular weight. (Washed and centrifuged 6 times.)		Material powders easily and wets readily. However, dispersion contains agglomerated resin particles. <u>Type of Film Formed on Glass</u> Color - clear; surface - rough Film, which is hard and tough, has excellent adhesion to glass. Water resistance is excellent.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
90	WC-130 (Dialyzed two days in Visking membrane.)		Same as Sample 80; to use for blending with other materials.
91	WC-130 (Washed and centrifuged 7 times.)		Material used as an additive for Bondex.
92	Vinac WR-20 (Dialyzed two days in Visking membrane.)		Material powders with some difficulty. It wets satisfactorily. <u>Type of Film Formed on Glass</u> Color - clear; surface - smooth. Adhesion is fair. Water resistance is very poor.
93	TIS-22 (Dialyzed two days in Visking membrane.)		Material powders easily but does not wet, although dispersion is possible. <u>Type of Film Formed on Glass</u> Color - clear; surface - rough. Adhesion is excellent. Water resistance is fairly good.
94	Formula No. 5 (formulation appears on page 19 of the final report on Development of a Liquid Polyvinyl Acetate Paint and also appears in glossary of this report.) (Material dialyzed 3 days in Visking membrane.)		Material thickened appreciably on dialysis. Dried material powdered and wet satisfactorily. Dispersion contained a few agglomerated resin particles. <u>Type of Film Formed on Glass</u> Color - white; surface - relatively smooth. Film is tough and has good adhesion. Water resistance is excellent.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
95A	Formula No. 6A (formulation appears on page 2 of the final report on Development of a Liquid Polyvinyl Acetate paint and also in glossary of this report). (Material dialyzed 3 days in Visking membrane.)		Material did not thicken on dialysis. Dried material powdered and wet satisfactorily. Dispersion contained a few agglomerated resin particles. <u>Type of Film Formed on Glass</u> Color - white; surface - some roughness. Film is tough and has fair-to-good adhesion to glass. Water resistance is excellent.
95B	Same as 95A (Washed and centrifuged 6 times.)		The solids when centrifuged packed so tightly together in the bottom of the centrifuge tube that they could not be separated. As a result, no redispersed polyvinyl acetate film was cast from this product.
96A	Formula No. 8 (formulation appears on page 8 of the final report on Development of a Liquid Polyvinyl Acetate Paint and also in the glossary of this report). (Material dialyzed 3 days in Visking membrane.)		Material powdered satisfactorily, but wet with considerable difficulty. Agglomerated resin particles are present in the dispersion. <u>Type of Film Formed on Glass</u> Color - white; surface - fairly rough. Film is very tough and has excellent adhesion to glass. Water resistance is excellent.
96B	Same as 96A (Washed and centrifuged 6 times.)		Material dried satisfactorily but could not be powdered, making the casting of a film impossible.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
97	WC-130 plus dibutyl phthalate to the extent of 10% based on latex solids. (Material dialyzed 3 days in Visking membrane.)		Material was difficult to remove from the flask because it is slightly rubbery. Material, which is hard and tough, could not be powdered.
98	WC-130 plus a small quantity of glyoxal (Material dialyzed 3 days in Visking membrane.)		Material dried satisfactorily but was difficult to remove from flask. It powders easily into fine granules which wet satisfactorily. Dispersion is foamy and contains a few scattered resin particles. <u>Type of Film Formed on Glass</u> Color - clear; surface - smooth. Adhesion is excellent. Water resistance is good.
99	WC-130 (Material dialyzed 3 days in Visking membrane.)		Material dried for eventual use in dry paints.
100	WC-130 (washed and centrifuged 6 times) plus a small quantity of Daxad 11.		Material powders easily and wets well. Dispersion contains some flocculated resin particles. <u>Type of Film Formed on Glass</u> Color - clear; surface - slightly rough. Film is slightly brittle but has excellent adhesion to glass. Water resistance is good. The dispersing agent detracts from the water resistance.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
101	WC-130 (dialyzed) plus a small quantity of glyoxal.		<p>Material dried satisfactorily. It powders easily and wets well. The addition of glyoxal results in a dispersion that tends to settle out.</p> <p style="padding-left: 40px;"><u>Type of Film Formed on Glass</u></p> <p>Color - white; surface - rough because film is discontinuous. Film is brittle and has good adhesion to glass. Water resistance is fair because the film formed is discontinuous.</p>
102	WC-130 (dialyzed) plus Tergitol NPX.		<p>Dried material is somewhat rubbery. Material wets well but makes a dispersion which is discontinuous.</p> <p style="padding-left: 40px;"><u>Type of Film Formed on Glass</u></p> <p>Color - almost clear; surface - very rough. Film, which is flexible, has fair adhesion to glass. Water softens the film.</p>
103	WC-130 (dialyzed) plus X-820 10% NH ₄ OH NPX to the extend of 10% based on latex solids.		<p>Material powders and wets satisfactorily. Disperses very well.</p> <p style="padding-left: 40px;"><u>Type of Film Formed on Glass</u></p> <p>Color - opaque; surface - fairly smooth. Film, which is hard and brittle, has good adhesion to glass. Water softens but does not whiten the film.</p>

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
104	Formula No. 8 (see Sample 96A). (Dialyzed for 2 days after paint had been freshly made.)		Dialysis thickens paint appreciably. It is easily removed from the flask. It powders easily and wets satisfactorily. Disperses well, although a few agglomerated resin particles are present. <u>Type of Film Formed on Glass</u> Color - white; surface - fairly smooth. Film is not brittle and has excellent adhesion to glass. Water resistance is excellent.
105	WC-130 (dialyzed) Tergitol NPX	7.5 gm 4 drops	Dried material satisfactorily removed from flask. It powders with difficulty but wets readily. Flocculated resin particles cause dispersion to be lumpy. <u>Type of Film Formed on Glass</u> Color - clear; surface - rough. Film does not have the flexibility of Sample 103. Adhesion is good-to-excellent. Water softens the film.
106	WC-130 (dialyzed) Tergitol NPX	7.5 gm 10 drops	Dried material very difficult to remove from flask. It is very hard and rubbery, making powdering impractical. No film was cast from this material because of the dispersion difficulties.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
107	WC-130 (dialyzed) Tergitol NPX Daxad 11	7.5 gm 2 drops trace	Dried material powders satisfactorily. It wets readily but the dispersion contains some flocculated resin particles. <u>Type of Film Formed on Glass</u> Color - clear; surface - rough. Film, which is hard and brittle, has good adhesion to glass. Water resistance is poor to fair.
108	WC-130 (dialyzed) Dibutyl phthalate	7.5 gm 3 drops	Material dried satisfactorily. It powders very well and wets readily. Dispersion contains agglomerated resin particles. <u>Type of Film Formed on Glass</u> Color - clear; surface - rough. Film, which is hard and brittle, has good adhesion to glass. Water resistance is poor to fair.
109	WC-130 (dialyzed) Tergitol NP-35	7 gm 2 drops	Dried material powders into fine granules which tend to block on standing. Material wets very fast. Dispersion is relatively free of agglomerated resin particles, but the film appears to be discontinuous. <u>Type of Film Formed on Glass</u> Color - clear; surface - fairly rough. Film, which is very flexible, has good adhesion to glass. Water resistance is excellent. Aging of film results in some loss of flexibility.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
110	MetroNite BXXXX WC-130 (dialyzed) Dibutyl phthalate	10.0 gm 7.5 gm 5 drops	Material is difficult to remove from flask. It powders very easily into fine granules. It wets satisfactorily. <u>Type of Film Formed on Glass</u> Color - white; surface - smooth. Film is flexible and has excellent adhesion. Water resistance is excellent.
111	MetroNite BXXXX WC-5510 Water (Dialyzed for 5 days.)	20 gm 15 gm 5 gm	Dried material difficult to powder. It wets readily. Dispersion foams. <u>Type of Film Formed on Glass</u> Color - white; surface - smooth. Adhesion is excellent. Film is tough and flexible. Water resistance is excellent.
112	WC-130 (Basic with NH ₄ OH) (Dialyzed)		Dried material was very easily removed from flask. Material, which powders easily, resembles powdered sugar. Wets slowly, but dispersion that results is excellent except for the foam. <u>Type of Film Formed on Glass</u> Color - clear; surface - smooth. Film has good adhesion to glass. Water resistance is very poor. Film dissolves rapidly.

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
113	WC-130 (dialyzed) (Basic with NH_4OH)	7.5 gm	Dried material is very hard and consequently powders with difficulty. It wets readily. Dispersion contains agglomerated resin particles and appears to need an emulsifying agent. <u>Type of Film Formed on Glass</u> Color - white; surface - discontinuous. Film lacks adhesion and flexibility. Water resistance is very poor.
	Tergitol NP-35	2 drops	
114	MetroNite BXXXX	10 gm	Material powders with difficulty into granules which wet readily. Dispersion contains a few agglomerated resin particles and appears to need an emulsifying agent. <u>Type of Film Formed on Glass</u> Color - white; surface - discontinuous and rough. Film has no strength nor adhesion to glass. Water resistance is very poor.
	WC-130 (dialyzed; basic with NH_4OH)	7.5 gm	
	Tergitol NP-35	2 drops	
	Dibutyl phthalate	2 drops	
	Daxad 23	trace	
115	MetroNite BXXXX	20 gm	Material is difficult to powder because of its tendency to be rubbery. <u>Type of Film Formed on Glass</u> Color - white; surface - rough, caused by a few scattered resin particles. Film is flexible and has good adhesion to glass. Water resistance is excellent.
	WC-5510	15 gm	
	Water	5 gm	
	Tergitol NP-35 (Material dialyzed 4 days)	5 drops	

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
116	MetroNite BXXXX	20 gm	Material powders easily and wets satisfactorily. Dispersion is good although it does foam. <u>Type of Film Formed on Glass</u> Color - white; surface - smooth except for occasional air bubbles. Film is flexible and has excellent adhesion to glass. Water resistance is excellent.
	WC-5505	15 gm	
	Water (Material dialyzed 5 days)	5 gm	
117	MetroNite BXXXX	20 gm	Material powders easily and wets satisfactorily. Dispersion is better than Sample 116, although it does foam. <u>Type of Film Formed on Glass</u> Color - white; surface - some roughness caused by occasional resin agglomerates and air bubbles. Adhesion to glass is excellent. Film is flexible but not as much as Sample 116. Water resistance is excellent.
	WC-55025	15 gm	
	Water (Material dialyzed 5 days)	5 gm	
118	MetroNite BXXXX	20 gm	Material, easily removed from flask, powders easily and wets readily. Dispersion contains air bubbles and agglomerated resin particles both of which tend to disappear on stirring. <u>Type of Film Formed on Glass</u> Color - white; surface - slight roughness caused by very fine resin agglomerates. Film is tough and flexible and has excellent adhesion to glass. Water resistance is good.
	WC-5505	15 gm	
	Water	5 gm	
	Tergitol NP-35 (Material dialyzed 6 days)	2 drops	

TABLE 4 (continued)

Sample Run No.	Components	pH	Observations
119	Metronite BXXXX WC-55025 Water Tergitol NP-35 (Material dialyzed 6 days).	20 gm 15 gm 5 gm 2 drops	Material, easily removed from flask, powders easily and wets satisfactorily. Dispersion is very good, although it does contain air bubbles. <u>Type of Film Formed on Glass</u> Color - white; surface - smooth. Film is flexible, tough, and has good adhesion to glass. Flexibility is less than Sample 118. Water resistance is good.

GLOSSARY

Trade Name	Description	Supplier
WC-130	Polyvinyl acetate latex	Bakelite Company
WC-130 L.M.W.	Polyvinyl acetate latex, low molecular weight	Bakelite Company
WC-130 H.M.W.	Polyvinyl acetate latex, high molecular weight	Bakelite Company
TS-22	Polyvinyl acetate latex	Shawinigan Products Corp.
Vinac WR-20	Polyvinyl acetate latex	Colton Chemical Co.
Elvacet 81-900	Polyvinyl acetate latex	Dupont Corp.
Darex Polymer Y	Polyvinyl acetate latex copolymer	Dewey and Almy Co.
Everflex G	Modified polyvinyl acetate latex	Dewey and Almy Co.
Rhoplex AC-33	Acrylic latex	Rohm and Haas Co.
WC-5510	WC-130 plasticized with 10% dibutyl phthalate based on latex solids	
WC-5505	WC-130 plasticized with 5% dibutyl phthalate based on latex solids	
WC-55025	WC-130 plasticized with 2-1/2% dibutyl phthalate based on latex solids	

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Trade Name	Description	Supplier
72-60	Polyvinyl alcohol	Dupont Corp.
K-707	Ammonium polyacrylate	Goodrich Chemical Co.
Cellosize WP-300	Hydroxyethyl cellulose	Carbide and Carbon Chem.
Tergitol NPX and NP-35	Nonionic wetting agents	Carbide and Carbon Chem.
Daxad 11 and 23	Sodium salts of polymerized alkyl naphthalene sulfonic acids	Dewey and Almy Co.
X-810	Alkali soluble copolymer	Monsanto Chemical Co.
X-820	Alkali soluble copolymer	Monsanto Chemical Co.
Polymer C-3	Modified vinyl acetate, alkali soluble	Monsanto Chemical Co.
X-810 10% NH ₄ OH	10% solution of X-810 in water made alkaline with NH ₄ OH	
X-820 10% NH ₄ OH	10% solution of X-820 in water made alkaline with NH ₄ OH	
ASP 400	Clay - aluminum silicate	Edgar Bros., Inc.
TiPure R-510	Titanium dioxide - rutile	Dupont Corp.
Nyral 300	Talc	R. T. Vanderbilt Co.
MetroNite BXXXX	Magnesium and calcium silicates and carbonates	MetroNite Company

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Formula No. 5

	<u>Percent</u>
TiPure R-510	25.2
Mica 325 mesh w.g.	2.8
WC-10*	23.3
Cellosize WPHS (5%)	9.3
Ethylene glycol	2.3
Glyoxal (30%)	0.5
Daxad 11	0.2
Tergitol NPX	0.1
Water	<u>36.3</u>
	100.0

Formula No. 6A

	<u>Percent</u>
Water	20.1
Daxad 11	0.2
TiPure R-510	20.0
ASP 400	7.5
MetroNite BXXXX	8.0
Ethylene glycol	2.9
WC-5510	27.6
Cellosize WPHS (5%)	13.0
Glyoxal 30%	0.6
Tergitol NPX	<u>0.1</u>
	100.0

*Composition of WC-10:

WC-130 latex solids	13.0 lb
Dibutyl phthalate	1.3
Water	<u>9.0</u>
	23.3 lb

Formula No. 8

	<u>Percent</u>
TiPure R-510	20.0
ASP 400	7.5
MetroNite BXXXX	8.0
Water	20.1
Daxad 11	0.2
Ethylene glycol	2.5
WC-5510	28.3
K-707 (15% T.S.)	2.6
Carbitol	1.7
Cellosize WP-300 (5%)	4.0
Tergitol NPX	0.1
Water	<u>5.0</u>
	100.0

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