Non-Polluting Metal Surface Finishing Pretreatment and Pretreatment/Conversion Coating

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Abstract
Picklex ® , a proprietary formulation, is an alternative to conventional metal surface pretreatments and is claimed not to produce waste or lower production or lower performance. A laboratory program was designed to evaluate Picklex . in common, large scale, polluting surface finishing operations against conventional processes, using steel and aluminum panels, measuring product coating properties, process operability, and costs. Twenty-one surface finishing combinations were tested under both "contaminated" and "non-contaminated" conditions with respect to finish adhesion, bending, impact, hardness, and corrosion resistance. Results indicate that Picklex ® -pretreated
panels performed as well as panels that were conventionally pretreated, and
with a simpler process. Picklex ® is particularly acceptable for powder coated steel or aluminum, but may not be for certain metal plates. The results are interpreted in terms of the surface film produced by Picklex .. A use rate of 5,400 ft 2 /gal was estimated. Picklex . did not generate by-product waste solids, was effective at room temperature, used short processing times, and was easy to use. A field study in an actual power coating shop was conducted to validate the lab results. An engineering assessment indicated that Picklex . can have cost advantages as well.

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Note from CD-ROM Editor:
We acknowledge that this paper represents a deviation in style from AESF's policy regarding proprietary terms. Because the paper concerns an evaluation by the U.S. Environmental Protection Agency and Battelle of a specific proprietary product (Picklex®), style was waived. To use generic words or phrases in place of the proprietary term, in this evaluation, would have been very awkward for the author and the reader.

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Acronyms and Abbreviations

aq	aqueous solutions
ASTM	American Society for Testing and Materials
CC	conversion coating
CrCC	chromate conversion coating
CFR	Code of Federal Regulations
DI	de-ionized
EC	electro-cleaning
ENi	electroless nickel (plating)
HCr	Hard Chromium
ICP	International Chemical Products, Inc.
IWTP	Industrial Waste Treatment Plant
MSDS	material safety data sheets
NA	not applicable
NRMRL	National Risk Management Research Laboratory
PEC	purchased equipment cost
ppm	parts per million (weight/weight)
QA	quality assurance
QC	quality control
SEM	scanning electron microscope/microscopy
TCLP	Toxicity Characteristic Leaching Procedure
TOC	total organic carbon
U.S. EPA	United States Environmental Protection Agency
UV	ultraviolet
ZnP	Zinc phosphatizing (all variations)

Objectives

The overall objective of this study was to evaluate the ability of Picklex® as a metal pretreatment or pretreatment/conversion coat in finishing operations which can be used to eliminate or reduce the amount of hazardous and toxic chemicals while maintaining equal or better product performance properties, with economic benefit for some processes and no significant economic penalty for other processes. Reduction in waste produced would be accomplished through the elimination of processing steps, and hence, the waste stream volumes from these steps, especially those processes involving ventilation of warm or gassing solutions. These improvements are expected to decrease production costs. The cost of Picklex® raw material would offset these savings somewhat. The Phase II objective was to evaluate Picklex® applications for powder coating finishes on aluminum and steel through representative commercial field tests. The evaluation focused on technical performance and economics while validating the previous laboratory tests and environmental benefits. (Battelle, 2000)

Background

Metal surface finishing is a major manufacturing industry consisting of thousands of production shops that provide weather- and wear-resistant and/or aesthetically pleasing manufactured products. The volume of hazardous/toxic waste streams produced from metal surface finishing operations is significant (U.S. EPA, 1995).

It is common for product surfaces to undergo more than 10 finishing steps that include degreasing and cleaning (for oil removal and de-scaling), etching, de-smuting, pickling, plating, and rinsing. The elimination of any of the surface processing steps is desired by manufactures to reduce processing costs, waste production, and energy consumption. With this objective in mind, a no-waste surface-finishing agent designed to provide a nearly one-step metal surface preparation operation for metal finishing operations would be of great benefit. In this study, Picklex® provides metal surface cleaning, pickling, conversion coating, and priming using a process simply consisting of degreasing, one dip-step (can also be sprayed), one rinse, and then final process. For powder coating field tests, oven drying occurred after the one dip-step. Because many surface-finishing operations exist, the potential for sizable waste and cost reductions by using Picklex® are significant. Therefore, the National Risk Management Research Laboratory (NRMRL) of the United States Environmental Protection Agency (U.S. EPA) contracted Battelle to perform a joint assessment of the efficacy of Picklex® in major polluting surface-finishing operations. This paper summarizes these findings. The test conditions, test data and detailed process descriptions are available elsewhere (Ferguson, 2000).

Pertinent Surface Finishing Processes Tested

Pretreatment processes prepare the surface of the basis metal for conversion coating and final finish (e.g., painting or metal plating). Pretreatment is a critical part of the surface-finishing process because it determines whether the subsequent layers will adhere, and the density of defects.

Conversion coatings are performed immediately after the pretreatment operation to preserve the clean surface and to provide the transition “primer” layer between the basis metal and any top coats.
Topcoats represent the final finish, the following that were selected as representing unique surface finishing features: hard chrome, electroless nickel, zinc plate, and powder coating.

Approach – Phase I, Laboratory Testing

Commercial operations require that Picklex® provide: (1) finished material properties similar to those produced by conventional processing, (2) be cost-effective, (3) meet or exceed environmental concerns, and (4) be simple to use and not require higher skilled labor than what is already used in surface finishing shops. Therefore, the approach used to evaluate Picklex® for metal finishing operations was to perform full multi-step, bench-scale, batch operation tests using side-by-side processing lines of seven conventional processes and of Picklex®. The metals that were evaluated were the high commercial volume basis metals (Q-Panel, 1999); low-carbon C1010 steel, and Al 3105, with some Al 2024 aluminum, at low and high (corroded) contamination. The metal test panels were degreased, stripped of corrosion, conversion coated or primed, and then powder top coated or metal-plated. These panels were subjected to a series of materials performance tests and the results compared. In addition, an attempt was made to exhaust a Picklex® bath to produce consumption rate for the preliminary engineering assessment (PEA).

Selection of Surface-Finishing Test Systems

Twenty-one (21) surface-finishing test combinations were selected for the systematic side-by-side comparisons with Picklex®. These systems are composed of the fundamental surface-finishing operations consisting of individual process steps (Table 1). To obtain the Picklex® (P) test system combination, the conventional process step(s) was simply removed and replaced with a Picklex® dip and rinse. Full details of the specific test conditions and process flow schemes used in the bench scale testing are available (Ferguson, 2000).

Methods and Materials

The experimental methods and materials were selected to represent 7 and 2.5 liter scale conventional surface finishing process operations using readily available materials (Ferguson, 2000). A Picklex® bath with one rinse tank was set up alongside the conventional line. This Picklex® bath was used in place of the pretreatment steps (except for degreasing) and/or the conversion coating step as required by the individual procedure for a particular test system (Table 2). To test a higher demand on the process, both “as supplied” (rust free) and “contaminated” (corroded) panels were processed through each surface-finishing line in both the conventional and Picklex® arrangements. The top coats or final finishes that were used included a polyurethane powder coat (PC), hard chromium plate (HCr), electroless nickel plate (ENi), and electrolytic zinc plate (Zn). Non-Picklex® surface-finishing baths were monitored and analyzed according to established procedures (Ferguson, 2000). The use rate of Picklex® was determined by processing sufficient surface area such that an estimate of bath usage can be made through actual exhaustion of the baths.

Table 1. Test Matrix for Both Contaminated and Noncontaminated Panels

Index	Test System ID[a]	Basis Metal	Pretreatment System	Conversion Coating	Final Finish
1	Fe-C-ZnP-PC	Low-carbon steel (C1010)	Conventional	Zinc Phosphate	Powder Coat
2	Fe-C-N-HCr			None	Hard Chromium
3	Fe-C-N-ENi			None	Electroless Nickel
4	Fe-C-N-Zn			None	Electrolytic Zinc
5	Fe-C- NiS-N			Nickel Strike	None
6	Fe-C-P-PC			Picklex®	Powder Coat
7	Fe-P-ZnP-PC	Low-carbon steel (C1010)	Picklex®	Zinc Phosphate	Powder Coat
8	Fe-P-N-HCr			None	Hard Chromium
9	Fe-P-N-ENi			None	Electroless Nickel
10	Fe-P-N-Zn			None	Electrolytic Zinc
11	Fe-P- NiS-N			Nickel Strike	None
12	Fe-P-PC[b]			Picklex®	Powder Coat
13	Al-C-N-N	Aluminum Al 2024 or Al 3105	Conventional	None	None
14	Al-C-N-PC			None	Powder Coat
15	Al-C-Cr-N			Chromate	None
16	Al-C-Cr-PC			Chromate	Powder Coat
17	Al-C-P-PC			Picklex®	Powder Coat
18	Al-P-N-N	Aluminum Al 2024 or Al 3105	Picklex®	None	None
19	Al-P-Cr-N			Chromate	None
20	Al-P-Cr-PC			Chromate	Powder Coat
21	Al-P-PC[b]			Picklex®	Powder Coat

(a) Test system identification (excluding redundant Indexes 12 and 21) consists of four abbreviations separated by hyphens. First abbreviation is basis metal: Fe = carbon steel, Al = aluminum; second abbreviation is pretreatment system: C = conventional, P = Picklex® pretreatment; third abbreviation is conversion coating: Cr = chromate, ZnP = zinc phosphate, N = none, P = Picklex®; fourth abbreviation is final finish coating: N = none, PC = powder coat, HCr = hard chromium, ENi = electroless nickel, Zn = electrolytic zinc, and NiS = nickel strike.

(b) In these tests, Picklex® served as the pretreatment and the conversion coating applied in one step.

Processing >2,000 corroded steel panels was chosen as the exhaustion test to provide a significant challenge to 2.5 L (0.66 gal) of bath.

Surface coatings on treated test panels were evaluated by common techniques including tape adhesion, salt fog corrosion resistance, hardness, burnishing, bending, impact adhesion, and microscopic examination. Most of these evaluation procedures were standard tests performed in accordance with ASTM practices (Ferguson, 2000).

Low carbon steel (C1010) and aluminum (Al 3105 H24 and Al 2024 T3) test panels [Q-Panel Laboratory Products (Cleveland, OH)] were used (Ferguson, 2000). These alloys represent the most common commercially used alloys that are surface treated and coated for consumer and commercial products (Q-Panel).

Picklex® Solution

Picklex® (International Chemical Products,Inc.of Huntsville, AL) was clear green, stored at room temperature, and appeared not to change during the 4 months of testing or during use. The MSDS indicated that the material was green in color and contained no hazardous substances as defined by 29 Code of Federal Regulations (CFR) 1910. The Picklex® process used in this study are: 1) degrease in toluene, 2) rinse once in DI water (to simulate water carryover from an aqueous degreaser should one be used), 3) immerse for 5.0 minutes in Picklex® (range is 3-5 min.), 4a) rinse once in DI water for 45 seconds if Picklex® served as conversion coat, or 4b) rinse for two minutes if Picklex® served as a pretreatment only (with use, this rinse solution developed the light green color exhibited by the Picklex® bath.), 5) perform water-break test, 6a) apply metal surface finish, if applicable, or 6b) dry panels for powder coating at 350-400°F for 10-15 minutes, then powder coat. All of the remaining chemicals were of laboratory reagent grade (99% or purer, Aldrich Chemical Co.) and used as supplied.

Phase I - Results and Discussion

A general description of these test results is provided in the following sections. Specifics are available elsewhere (Ferguson, 2000). Coating performance test results include adhesion, bending, burnishing, hardness, impact, and salt spray exposure. Table 2 summarizes the comparative test results between Picklex® and the conventional processes. All of the tape adhesion tests for coated aluminum and steel passed at the highest level (5B), namely no paint removal from cross-hatched surfaces. It is concluded that Picklex® offers an advantage over the conventional process with respect to top coat adhesion because it provided equivalent mechanical strength with fewer and simpler steps as well as reducing waste production.

In the bend tests, no peeling or flaking was observed for all test systems. These results indicate that Picklex® is at least "as good as" the conventional process in bending performance of the powder top coat. The powder-coated C1010 steel panels on which either a ZnP conversion coating or a Picklex® conversion coating passed the bend adhesion test. Therefore, it is concluded that Picklex® passed the pretreatment screening test for powder top coats for C1010 steel with respect to the bend test.

Bend test results for hard chromium, electrolytic zinc, and electroless nickel metallic coatings on non-contaminated and contaminated steel substrates exhibited some degree of cracking, but only those coatings that could be lifted off using the standard tape were considered to have failed the test. Hard chromium passed the bend test for all pretreatment conditions. The only pretreatment condition that resulted in a bend test failure for electrolytic zinc was conventionally pretreated and contaminated steel substrates. Hence, it is concluded that the contamination level was beyond what could be handled by the conventional pretreatment process. All the Picklex®-pretreated steel panels coated with electrolytic zinc passed this test, both non-contaminated and contaminated. Therefore, it is a significant advantage that Picklex® was able to handle this contamination without having to alter the process. Interestingly, corroded steel substrates using Picklex® as a pretreatment passed the bend adhesion test for ENi coatings, but the non-corroded panels did not. This result may reflect that Picklex® can use surface corrosion conversion products in forming its pretreatment/conversion coat (Carpenter, 1999).

Metallography

SEM photomicrographs, including cross-sectioned panels, for the metal plated test systems are available (Ferguson, 2000). All plates representing the Picklex® process for chromium, zinc ENi, and Ni showed similar thicknesses and morphologies relative to the conventional plates. This result indicates that Picklex® does not appreciably alter the nucleating features or plating rates for these metals. However, some plates exhibited low adhesion (Table 2).

Table 2. Comparison of Coating Performance Results of Conventionally Produced Panels versus Picklex®-Produced Panels

Coating	Tape Adhesion	Bend	Burnishing	Impact Adhesion	Hardness	Corrosion Resistance
Powder Top coat on Aluminum	Equivalent[(a)], good on contaminated and noncontaminated surfaces	Equivalent[(a)], passed, , no peeling/flaking	NA[(b)]	Equivalent[(a)]	NA[(b)]	Equivalent[(a)]
Powder Top Coat on Steel	Equivalent[((a)], good on contaminated and noncontaminated surfaces	Equivalent[(a)], passed, , no peeling/flaking[(c)]	NA[(b)]	Conventional slightly better than Picklex®	NA[(b)]	Equivalent[(a)]
Hard Chromium	Equivalent[((a)], good on contaminated and noncontaminated surfaces	Equivalent[(a)], passed, no peeling/flaking	Equivalent[(a)], passed	Equivalent[(a)]	Equivalent[(a)], good on contaminated and noncontaminated	Conventional slightly better
Electrolytic Zinc on Steel	Contaminated, conventional failed	Equivalent[(a)], passed, no peeling/flaking	Picklex® failed lifting, passed blisters and peeling	Picklex® much better on contaminated, equivalent on noncontaminated	NA[(b)]	Equivalent[((a)], marginal
ENi on Steel	Contaminated, Picklex® failed	Equivalent[(a)], passed, no peeling/flaking	Both passed blisters and peeling; only contaminated, conventional passed lifting	Equivalent[(a)], good	Equivalent[(a)], good	Equivalent[(a)], marginal
Alodine®	NA[(b)]	NA[(b)]	NA[(b)]	NA[(b)]	NA[(b)]	Equivalent[(a)], both need CrCC for excellent performance

(a) Conventional and Picklex®-pretreated panels provided the same coating performance.
(b) NA = Not applicable.
(c) cracking of powder topcoat with Picklex® and conventional

Bath Exhaustion Test Results and Conclusions

Corroded steel panels were used for the Picklex® bath exhaustion tests to provide an accelerated test. A total of 2,035 contaminated panels of C1010 steel with a surface area of 0.208 ft^2/panel (424 ft^2 total) were processed through 0.66 gal (2.50 L) of Picklex® bath (or 642 ft^2/gal) over about a one month period. Powder coating properties were tested at regular intervals. The bath condition was also monitored with respect to pH, acid components and visible spectrophotometry. Wavelengths ≥520 nm appear viable for Picklex® bath monitoring.

Based on the UV/visible results, it is concluded that the Picklex® bath is 78% active (22% depleted) after the equivalent of approximately 642 ft^2 of contaminated steel surface area per gallon of Picklex® is processed. The active ingredients in surface-finishing baths are not normally consumed 100% since the rate of action becomes too slow as the reagents become dilute. Using a linear extrapolation of the consumption of active ingredients to 50% would correspond to approximately 1,450 ft^2 of surface processed per gallon of bath. Using the measured conversion factor of 3.7 (Ferguson, 2000) to convert from contaminated to noncontaminated panels gives an equivalent of 5,400 ft^2/gal processed. Given the uncertainties, this value is on the order of the 10,000 ft^2 value claimed by the vendor for the capacity of Picklex®, especially if the bath can be consumed more efficiently (e.g., by greater care in rinse recycle, or by use of squeegees). As measured by the milliequivalents of weak acid (to reach an equivalence point), the Picklex® bath strength was 56.8% of full strength after processing 2,035 panels. This measurement is in general agreement with the visible absorption percent change. These two independent Picklex® bath monitoring methods register a similar depletion in bath chemicals, which can be extrapolated to a bath life near that claimed by the vendor. Hence, there is qualitative agreement between the methods and claims based on bath monitoring methods.

Although a determination of the chemical reactions of Picklex® was outside of the scope of the enclosed study, it was observed that very little iron accumulated in the Picklex® bath , even when processing corroded panels. Therefore, it may be in the case of Picklex®, that stripped corrosion products are in fact being incorporated into the conversion coating produced by Picklex®. Hence, Picklex® may meet the need for a combined pretreatment and conversion coating process that does not need to be purged as a waste on a regular basis, but only as needed to correct operational errors. Evidently this film forming performance would occur by first dissolving the oxide (rust) in mild acid pH 2, with phosphate complexation assistance perhaps enhanced by electron exchange via the Mn present in the formulation. Then, instead of the iron dissolving into the bulk solution, a second "conversion coating" type reaction would occur, depositing the dissolved iron and probably small amounts of bath components, such as Mn, Cr, Mg, and phosphate onto the surface. Therefore, no loose particles are formed as in the case of alkaline cleaning or phosphatizing, which avoids the need for filtration. The nonuniform appearance of the Picklex® conversion coating may reflect this variability in composition as these four metal ions are different colors. Hence, depending on the results of the PEA, and the physical and corrosion resistance properties of the final surface treatment, Picklex® may offer a significant advantage to conventional acid pickling of steel.

Picklex® Waste Disposal Assessment

Fresh, spent, and impurity-spiked spent Picklex® samples were treated (Ferguson, 2000) using the conventional pH 9 precipitation industrial waste water treatment method to produce samples for a waste disposal assessment for Picklex®. The waste solids were assessed for TCLP leachability. The treated water supernatants for discharge were also examined and found as most likely dischargeable. Actual discharge limits from industrial waste treatment plant (IWTP) operations are site-specific and are determined on a case-by-case basis with local, state, and federal regulatory agencies. Hence, no exact classification of these potential waste solutions is possible until a specific location is known. All leachates passed with respect to 40 CFR 261 (U.S. EPA, 1999). Therefore, Picklex® does not appear to present unusual waste treatment issues. Note that the manufacturer claims that solution, diluted by rinse waters, can be restored by adding a concentrate to the bath. Therefore, waste treatment may not be frequent or necessary in many processes.

Phase I Discussion

These results suggest that Picklex® can provide an effective, nearly one-step metal surface preparation operation for many, but not all, metal finishing operations. Hence, based on these limited screening test results, Picklex® appears to effectively avoid the production and use of certain hazardous/toxic chemicals in surface finishing operations, such as pickling acids, metal salt phosphatizing solutions, hot alkaline baths, gas mists from electro-cleaning, etc. It appears not to exhaust readily even when processing heavily corroded surfaces. To eliminate yet another process step, further testing might verify its effectiveness as a degreaser for many applications since Picklex® contains significant organic content and forms organic-based films.

The Picklex® bath exhaustion test results suggest that the Picklex® bath can accommodate impurities for certain surface finish properties. However, for HCr, the influence of drag in metal ion impurities on Cr plating current efficiency should be checked carefully in pilot testing before using Picklex® in this process.

Table 2 compares the materials test results for conventional vs Picklex® coatings. Although none of the processes are optimized for maximum properties, the results indicate that, in most cases, Picklex®-pretreated and/or conversion coated panels performed as well or slightly better than panels conventionally pretreated and/or conversion coated. However, as expected, Picklex® may not be applicable to all systems. For example, it may not be acceptable as a pretreatment for ENi-coated materials when bending of the parts occurs due to lack of adhesion of the plate, or for electrolytic zinc-coated materials if rubbing is involved.

Picklex® pretreatment was judged acceptable for use in hard chromium operations, although the conventionally pretreated panels provided slightly better corrosion performance. Picklex® also was judged acceptable for corrosion resistance after powder top coating. Picklex® appears to be a viable alternative to conventional pretreatments for aluminum substrates, but not as a direct substitute for CrCC as a topcoat for corrosion resistance. It appears true that in some cases the more contaminated the surface, the better Picklex® performs relative to conventional processing. Given the simpler processing and minimized waste generation with Picklex®, "same as" mechanical test results are often sufficient to justify the use of Picklex® over the conventional processes. Painting pretreatment and replacement of acid pickling/alkaline cleaning appear as excellent applications for the Picklex® technology for both steel and aluminum. Unlike conventional processes, steel and aluminum can be treated with Picklex® at the same time with the same process.

Bath exhaustion tests indicated that Picklex®-pretreated panels performed better when zinc phosphate conversion coating was not applied. A projected 5,400 ft^2 of noncontaminated steel surface were processed per gallon of Picklex® bath used before signs of tape adhesion failure appeared. The actual use rate may be lower than this since heavily corroded panels were used in this accelerated test possibly exceeding the impurity deposition capacity of the Picklex® film. Therefore, data suggest that Picklex® can serve as a satisfactory alternative to conventional pretreatment and phosphatizing CC processes, even when the Picklex® bath has been repeatedly used without replenishment.

Picklex® is a clear, light-green aqueous solution that has a slight, but not unpleasant odor. Qualitative advantages of surface finishing processing with Picklex® include: slow evaporation rate, adequate draining rate, and it is easily agitated. It did not form solids in the bath during processing. Only one rinse bath was used with Picklex® and this solution also seemed to possess a long life, did not form precipitates, and could be added back to the Picklex® bath for value recovery. Operationally, the use of Picklex® is straightforward. The Picklex® treatment is dipped at ambient temperature for short periods with the usual aeration mixing. Such simple operations support the vendors' claim that labor savings may result from using Picklex®, depending on the complexity of the process and steps being replaced.

One disadvantage of Picklex® is cost. Depending on the process selected and care given to operations, spillage control, drain time, excess recovery, and rinse water recycle are important factors. Picklex® contains, acid, TOC, and manganese ions, but these are not believed to be environmental or toxicity problems. Picklex® also contains chromium (III), this is not believed to be an environmental or toxicity problem because often the solution will not have to be discharged. In the case where the solution needs to be treated, common metal ion removal in waste treatment systems will easily control this. The surface film formed by Picklex® is water soluble, hence excessive rinsing may result in loss of some or all of the beneficial properties from using Picklex®. Manufacturer expects price to drop as sales and manufacturing volume increases.

PHASE II, Field Testing

This section describes Phase II of the study performed to measure the effectiveness of Picklex® as an alternative to commercial metal surface pretreatments and conversion coats in an industrial setting. A broad laboratory evaluation of Picklex® was studied during Phase I for many processes. Then, a focused field test with Picklex® for powder coating applications on aluminum and steel was studied.

A total of 41 different combinations of substrate, degreaser, pretreatment, conversion coat, and powder coat were tested. Only noncontaminated panels and components, without corrosion products, were used. Aluminum 3105 and low carbon steel 1010 panels were used. Also aluminum die cast alloy and malleable iron casting components were processed.

Three batches of panels and/or components were processed at Mills Metal Finishing. The first batch included steel and aluminum panels and served as a process validation from the laboratory to industrial setting. The second batch included commercial components and panels. The third batch included both components and panels and focused on using Spraylat PE6639M, which is the Commercial Partner's standard powder coat material used on the aluminum components for production use.

The coatings were evaluated by a matrix of tests including adhesion, bend adhesion, impact adhesion, hardness, and corrosion resistance. The materials' testing was performed at Battelle. Results indicate that the field testing replicated the laboratory processes performed during Phase

I. Picklex® may serve as a degreaser for steel. The Picklex® processing time can be reduced relative to the laboratory tests and conventional surface preparation processes. The immersion time for aluminum can be decreased from 5 minutes to 30 seconds. Eliminating rinsing after Picklex® was found to produce undesirable results and insufficient adherence of powder coating to substrate.

Objectives and Scope

Field-produced components and panels similar to those used in Phase I testing were evaluated at a commercial metal surface treatment vendor using their equipment and personnel. These test components and panels after treatment and powder coating were then evaluated with the same coating performance test conducted in Phase I. Performance testing was limited to tape adhesion, bend, impact adhesion, and corrosion resistance.

Picklex® as a Pretreatment

The field testing replicated the results of the laboratory processes. The field testing showed slightly better results for Picklex® than conventional surface-finishing processes for bend and impact adhesion. In general, Table 3 compares results between the two phases for conventionally pretreated panels versus Picklex® pretreated panels.

Table 3. Comparison of Coating Performance Results of Conventionally Pretreated Panels versus Picklex® Pretreated Panels – Phase I & Phase II

Coating	Phase	Tape Adhesion	Bend	Impact Adhesion	Corrosion Resistance
China White Powder Topcoat on Aluminum	Phase I	Equivalent[(a)]	Equivalent[(a)] passed, while CCC Picklex® conventional cracked with no peeling/flaking	Equivalent[(a)]	Equivalent[(a)]
	Phase II	Equivalent[(a)]	Equivalent[(a)] passed	Picklex® CCC slightly better, conventional CCC had one failure	Equivalent[(a)]
China White Powder Topcoat on Steel	Phase I	Equivalent[(a)]	Equivalent[(a)] passed, cracking with Picklex® and conventional	Conventional slightly better than Picklex®	Equivalent®
	Phase II	Equivalent[(a)]	Equivalent[(a)] passed	Equivalent[(a)]	Equivalent[(a)]

(a) Conventional and Picklex® pretreated panels provided the same coating performance.
(b) Panels processed in Picklex® for 5 minutes and rinsed in DI water for 45 seconds before receiving conversion coat.

Picklex® may serve as a degreaser for steel. During Batch No. 2, one group of steel panels was processed through Picklex® without a conversion coat and skipping the degreasing step. This group produced performance results similar to those processed with Picklex® and no conversion coat, but with the degreasing included. Further testing is recommended to show replication of these results, especially with lightly pre-greased feed material.

During Phase II, the immersion time was reduced from 5 minutes to 30 seconds for aluminum and from 5 minutes to 90 seconds for steel. Table 4 shows a comparison between Batch Nos. 1 and 2 for commercial degreaser, Picklex® (with varying times), and 45-second rinses. Depending on the corrosion resistance needed for the application, shorter Picklex® immersion times might be acceptable. The 5-minute immersion time gives greater corrosion resistance than the 90-second application. The corrosion resistance on aluminum was equivalent for all other test parameters.

Table 4. Effect of Picklex® Process Time

Powder Coating	Process Time	Tape Adhesion	Bend	Impact Adhesion	Corrosion Resistance
China White Powder Topcoat on Aluminum	5 vs. 0.5 min.	Equivalent[(a)]	Equivalent[(a)]	Equivalent[(a)] passed with 5 minute application receiving slightly higher readings	Equivalent[(a)]
China White Powder Topcoat on Steel	5 vs. 1.5 min.	Equivalent[(a)]	Equivalent[(a)]	Equivalent[(a)] passed with 5 minute application receiving slightly higher readings	Depending on application needs, 5 minute application has higher corrosion resistance than 90 second application

(a) Differences in Picklex® processing time provided the same coating performance.
(b) Both rinses were at 45 sec and conversion coating step was skipped.

The vendor recommended eliminating the rinse after processing with Picklex® for powder topcoats. The field testing on panels showed that for aluminum and steel at least a quick rinse (in and out) is needed. For the non-rinsed case, the aluminum and steel surfaces appeared to be very tacky with inconsistent residual Picklex® deposit remaining after drying. This residual Picklex® did not allow the powder coat to adhere properly causing failures. For example, one group of steel panels in Batch No. 2 failed in the salt fog chamber much sooner than the group with a rinse after Picklex®.

Surface–Finishing Procedures

Three different degreasing conditions were used for the field testing. Certain panels in Batch No. 1 were degreased with toluene at Battelle before processing further to replicate the Phase I laboratory test results. At Mills, certain non-degreased panels were degreased with Zep I.D. Red, and certain other sets of panels were not degreased

Two pretreatment systems were used for each type of material: commercial pretreatment and Picklex® pretreatment. For aluminum, the pretreatment consisted of Mills' normal process line, a soak cleaner, two rinses, an etch, two rinses, a deoxidizer, and two rinses. For steel, each commercial pretreatment consisted of Mills' normal process line, a cleaner and then a series of rinses. For Picklex® pretreatment, Picklex® was used for a specified time and then one rinse was performed at a specified time if at all as per the test sequence. In both cases a conversion coat was followed unless this step was skipped and the Picklex® served as the conversion coat also. The two commercial conversion coatings which were reviewed during field testing are chromate for aluminum and zinc phosphate for steel. All of the components and panels completely processed at Mills were powder coated. Figure 1 illustrates the process steps for both conventional zinc phosphatizing on steel and the Picklex® process. The use of Picklex® replaces the pretreatment (electro cleaner and one rinse step) and conversion coating (conversion coat and three rinses). Figure 2 illustrates the sequential process steps for both conventional chromate conversion coating on aluminum and the alternative Picklex® process. As shown, the use of Picklex® (Al-P-R-N-N-PC) replaces both the pretreatment (alkali cleaning, 2 rinse steps, etch, 3 rinse steps, deoxidizer, 2 rinse steps) and chromate conversion coating (chromate conversion coat and three rinse steps).

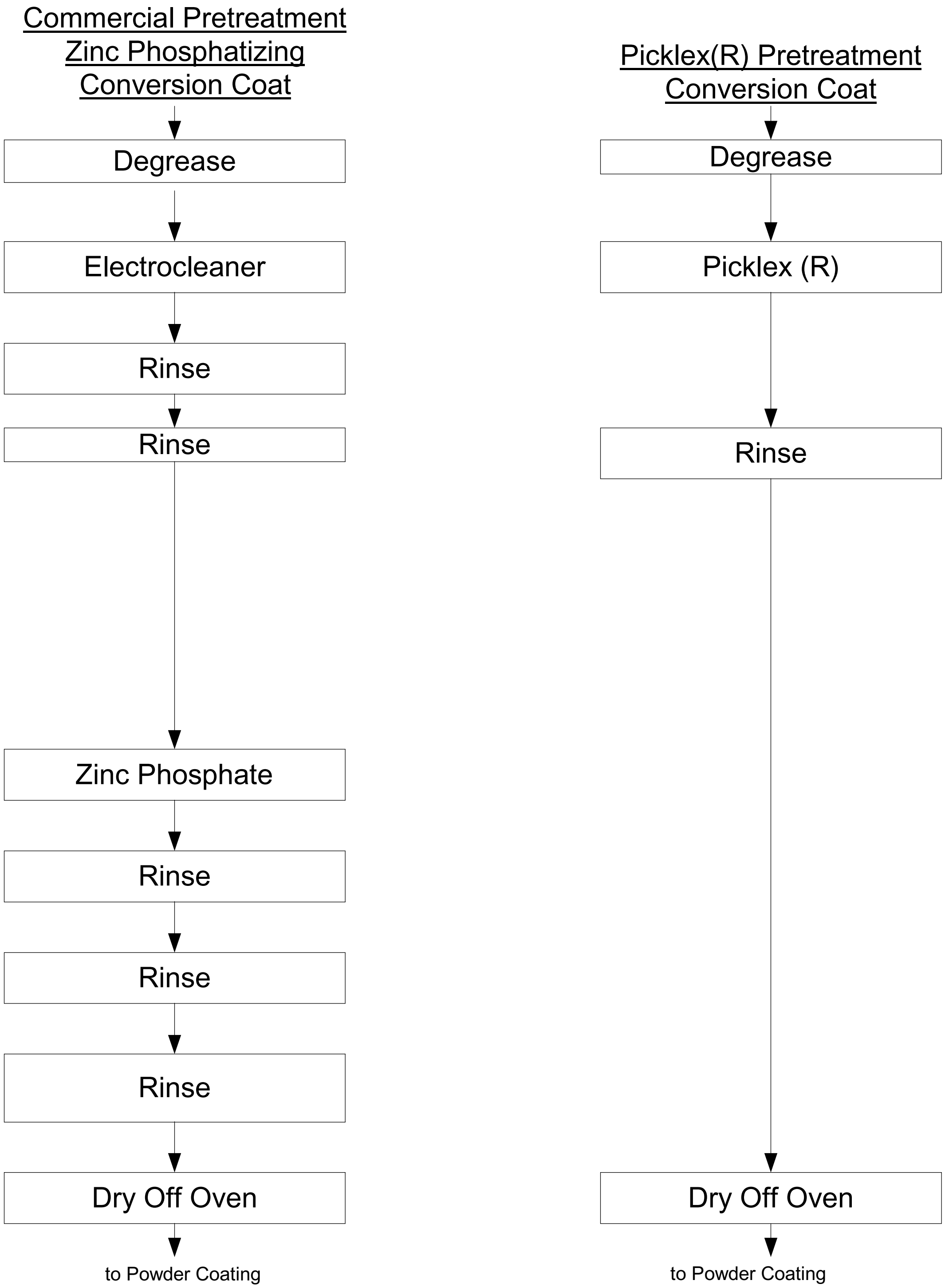

Figure 1. Processes for Commercial Pretreatment Zinc Phosphate Conversion and Picklex® Pretreatment Conversion Coatings on Steel

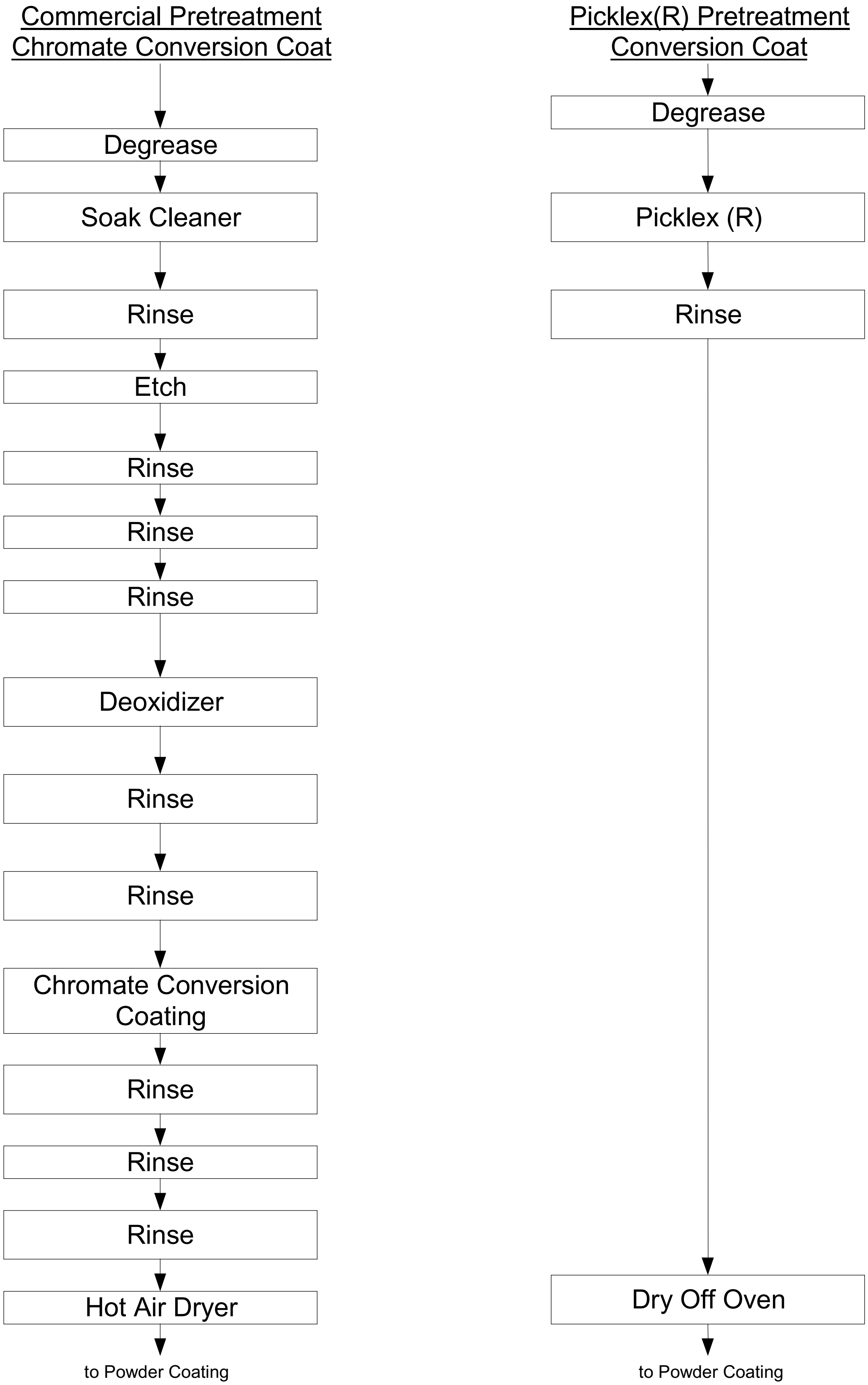

Figure 2. Processes for Commercial Pretreatment Chromate Conversion and Picklex® Pretreatment Conversion Coatings on Aluminum

Test Panels

Following Phase I protocol metal test panels were obtained from Q-Panel Laboratory Products (Cleveland, OH).(1) Surface treatments were applied to aluminum 3105 and steel 1010 because the two alloys were tested in Phase I and represent large commercial usage. A minimum of two panels was used for each surface finish evaluation parameter

Figure 3. Powder Coating Operation

Three powder coatings were used to coat test panels in the field trials, Vista SH-2004 china white from Ferro Powder Coatings Division and PE6639M and PEB1867C from Spraylat Corporation. China white powder coat material, which was obtained and used during Phase I, was used for all batches in Phase II to replicate the powder coat material used during Phase I. Two Spraylat products supplied by the Commercial Partner No. l, Spraylat PEB1867C and PE6639M, were also used in Phase II. The Spraylat PEB1867C was used for Batch Nos. 1 and 2. Spraylat PE6639M, was used for Batch No. 3. The Spraylat PE6639M was applied to Batch No. 3, aluminum panels and components because this powder coat material is the standard material for the Commercial Partner No. 1's die cast aluminum parts

Technical Performance

This section summarizes the technical performance evaluation by the various coating performance parameters and addresses the economic and overall feasibility issues in engineering assessment.

Powder Coating Film Thickness

Dry powder-coating film thickness was measured on coated panels by ASTM D 1186 and ASTM D 1400 as described in Appendix D, averaging six readings for each panel. Table 5 shows the thickness range for Batch Nos. 1, 2, and 3 compared with the manufacturer's recommendations for applied thickness.

Table 5. Dry Film Thickness of Powder Coatings on Test Substrates

Batch	**Substrate**	**Powder Coating**	**Thickness Range (mils)**	**Target Thickness (mils)**
BATCH No. 1	Steel	China White	1.65 to 2.88	2.0±1.0
	Aluminum	PEB1867C	1.24 to 2.14	2.0 ±1.0
	Aluminum	China White	1.24 to 2.24	2.0 ±1.0
BATCH No. 2	Steel	China White	0.93 to 1.74	2.0±1.0
	Aluminum	China White	1.14 to 2.29	2.0±1.0
	Aluminum	PEB1867C	1.03 to 1.99	2.0±1.0
BATCH No. 3	Steel	China White	2.88 to 3.29	3.0±1.0
	Aluminum	PE6639M	2.74 to 3.39	3.0±1.0

Adhesion by Tape Test

Adhesion by tape was determined by ASTM D 3359. The ratings can be summarized as:

- 5B The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
- 4B Small flakes of the coating are detached at intersections; less than 5% of the area is affected.
- 3B Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15% of the lattice.
- 2B The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35% of the lattice.
- 1B The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65% of the lattice.
- 0B Flaking and detachment was worse than Grade 1B.

Table 6 lists the adhesion by tape ranges for each group of panels. All of the steel panels coated with China White and tested for adhesion by tape were rated 5B except one panel. In Batch No. 3 panel GGG16 was rated 4B for adhesion by tape.

Adhesion by tape results for aluminum panels coated with PEB1867C in Batch No. 1 and Batch No. 2 were rated 5B. Aluminum panels coated with China White in Batch No. 1 were rated 5B except one panel (Z22), which was rated 4B. In Batch No. 3 the aluminum panels were coated with PE6639M and adhesion by tape ratings include one panel at 2B, one panel at 3B, and four panels at 4B. Adhesion for Batch No. 3 aluminum panels is not as good as panels coated in earlier batches. Adhesion could be less for the PE6639M coating than the others, or there may be environmental process, or operator differences during the third batch of processing and coating relative to the first two batches.

In Batch No. 1 and Batch No. 2, adhesion by tape did not reveal any discernible differences between the different treatment scenarios. Batch No. 1 Group A, conventional pretreatment and chromate conversion coat; Group B, Picklex® pretreatment and chromate conversion coat; Group J, conventional pretreatment and zinc phosphate conversion coat; and Group K, Picklex® pretreatment and zinc phosphate conversion coat, served as a replication for the processes used during Phase I. Table 1 shows equivalent results between the two different processes compared to those from Phase I.   Supporting the use of Picklex® as a degreaser for steel, group CC from Batch No. 2, which was not degreased, achieved the same tape adhesion results as group BB from Batch No. 2, which was the exact same process plus the degreasing step. Tape adhesion results for certain groups showed that the Picklex® immersion time can be reduced from 5 minutes to 30 seconds for aluminum and 90 seconds for steel. The aluminum groups were the following for aluminum: group F from Batch No. 1, with a 5 minute immersion time and group S from Batch No. 2, with a 30 second immersion time. The steel groups were the following: group M from Batch No. 2, with a 5 minute immersion time and group BB with a 90 second immersion time.

Test System Designation

Each test system was identified by a combination of 9 abbreviations that referenced the substrate material, the degreaser, the pretreatment system, the rinse after pretreatment, the water break test, the conversion coating, the rinse after conversion coating, the dryer, and the powder coat material. The abbreviation code used in the report is shown on table 6.

Table 6. Summary of Results from Materials Testing

Batch	Group	Test ID[a]	Dry Film Thickness Range	Adhesion by Tape Range	Impact Adhesion: Average Impact (foot – pounds)	Impact Adhesion: Sample I.D. of Failures	Sample I.D. of Bend Adhesion Failures	Salt Fog: Exposure Time (hours)	Salt Fog: Adhesion by Tape Range[c]	Salt Fog: Creepage Range[d]
Aluminum										
1	A	Al-T-C-R-n-Cr-R-A-CW	1.27-1.89	5B	40	6		1760	5B	
1	B	Al-T-P5M-R-w-Cr-R-A-CW	1.91-2.24	5B	54			1760	5B	
1	C	Al-T-P5M-R-w-N-N-O-CW	1.85-2.18	5B	59			1760	4B	
1	D	Al-C-C-R-n-Cr-R-A-CW	1.86-2.07	5B	64			1760	5B	
1	E	Al-C-P5M-R-w-Cr-R-A-CW	1.93-2.57	5B	65			1760	4B	
1	F	Al-C-P5M-R-w-N-N-O-CW	2.04-2.68	5B	66			1760	3-4B	
1	G	Al-T-P5M-R-w-Cr-R-A-SB	1.63-2.04	5B	4	13, 10, 3, 18	12	1760	5B	
1	H	Al-C-P5M-R-w-Cr-R-A-SB	1.24-1.83	5B	4	11, 2, 8, 18		1760	5B	
1	I	Al-C-P5M-R-w-N-N-O-SB	1.85-2.14	5B	4	1, 4, 10, 17		1760	3-4B	
2	Q	Al-C-P30S-R-w-Cr-R-A-CW	1.23-2.28	5B	60			1277	5B	
2	R	Al-C-P30S-N-w-N-N-O-SB	1.42-1.77	5B	4	6, 19, 26, 35	3,14,31	1277	3-4B	
2	S	Al-C-P30S-R-w-N-N-O-CW	1.39-1.55	5B	46			1277	4B	
2	T	Al-C-P30S-R-w-N-N-O-SB	1.08-1.58	5B	4	2, 13, 27, 34	5,23,31	1277	5B	
2	U	Al-C-C-R-w-Cr-R-A-CW	1.14-1.30	5B	60			1277	5B	
2	V	Al-C-C-R-w-Cr-R-A-SB	1.29-1.99	5B	4	1, 15, 24, 36	5,22,31	1277	5B	
2	X	Al-C-P30S-R-w-Cr-R-A-SB	1.05-1.42	5B	5		2,9,22	1277	5B	
2	Y	Al-C-P30S-N-w-N-N-O-CW	1.53-2.29	5B	4	8, 17, 25, 29	3,9,32	1277	2-4B	
2	Z	Al-C-P30S-N-w-N-N-O-CW	1.31-1.77	4-5B	4	9, 13, 26, 27	3,16,32	1277	0-2B	
2	RR	Al-C-P30S-QR-w-N-N-O-SB	1.03-1.38	5B	4	1, 6, 8, 11	3,7,10	1277	2-4B	
2	SS	Al-C-P30S-QR-w-N-N-O-CW	1.35-1.82	5B	52			1277	4B	
3	AAA	Al-C-P30S-R-w-Cr-R-A-SA	2.88-3.39	2-4B	27		NA[e]	NA		
3	BBB	Al-C-P30S-R-w-N-N-O-SA	2.75-2.96	3-4B	35		NA	NA		
3	CCC	Al-C-P30S-QR-w-N-N-O-SA	2.74-3.00	4B	37		NA	NA		

Table 6. Summary of Results from Materials Testing

Batch	Group	Test ID[a]	Dry Film Thickness Range	Adhesion by Tape Range	Impact Adhesion		Sample I.D. of Bend Adhesion Failures	Salt Fog		
					Average Impact (foot – pounds)	Sample I.D. of Failures		Exposure Time (hours)	Adhesion by Tape Range[c]	Creepage Range[d]
Steel										
1	J	Fe-T-C-R-w-ZnP-R-O-CW	1.95-2.0	5B	20			1760, J2 @1376		7 for J2
1	K	Fe-T-P5M-R-w-ZnP-R-O-CW	1.65-2.20	5B	25			1760		
1	L	Fe-T-P5M-R-n-N-N-O-CW	2.13-2.35	5B	160			1376		6
1	M	Fe-N-P5M-R-w-N-N-O-CW	2.14-2.88	5B	160			1376		5-6
1	N	Fe-C-C-R-w-ZnP-R-O-CW	1.88-2.11	5B	7	8,18		1376		6-7
1	O	Fe-C-P5M-R-w-ZnP-R-O-CW	1.68-2.82	5B	32			1760		
1	P	Fe-C-P5M-R-n-N-N-O-CW	2.04-2.40	5B	18			1376		5-6
2	AA	Fe-C-P90S-R-w-ZnP-R-O-CW	0.93-1.34	5B	4	25	21,32,34	893, AA16 @ 608		7-8
2	BB	Fe-C-P90S-R-w-N-N-O-CW	1.22-1.74	5B	155			893		5-6
2	CC	Fe-N-P90S-R-w-N-N-O-CW	1.46-1.58	5B	158			893		4-5
2	DD	Fe-C-P90S-N-w-N-N-O-CW	1.11-1.22	5B	160		22	608		3-4
2	EE	Fe-C-C-R-w-ZnP-R-O-CW	1.22-1.69	5B	26		7,16,20	893		6-7
3	GGG	Fe-C-P90S-N-w-N-N-O-CW	2.88-3.29	4-5B	30			(b)		

(a) First abbreviation is basis metal: Al=aluminum panel, Fe=steel panel, AlC=Aluminum component, FeC=steel component
Second abbreviation is degreaser: T=toluene, C=Mill's degreaser, N=No degrease step
Third abbreviation is pretreatment system: C=conventional (Mill's pretreatment), P5M=Picklex® for 5 minutes, P30S=Picklex® for 30 seconds, P90S=Picklex® for 90 seconds
Fourth abbreviation is rinse step after pretreatment system: R=rinse, N=no rinse step after pretreatment
Fifth abbreviation is water break step: W=water break test, N=no water break test to be performed
Sixth abbreviation is conversion coating system: Cr=chromate, ZnP=zinc phosphate, N=none
Seventh abbreviation is rinse after conversion coating system: R=rinse, N=none
Eighth abbreviation is drying system: O=dry off oven, A=hot air dry off box
Ninth abbreviation is powder coating system: CW=china white, SA=standard powder coat material for aluminum Spraylat PE6639M used during Batch 3, SB=Spraylat PEB1867C used during batches 1 and 2
NA=not applicable
(b) Sample still in salt fog chamber.
(c) Adhesion by Tape test performed after salt fog exposure.
(d) Creepage rating performed only on steel panels which had loss of coating adhesion on scribe.
(e) NA = not applicable, test not performed

Superior results were obtained when comparing adhesion of Commercial Partner No. 1's production parts versus Picklex® processed parts. Commercial Partner No. 1 provided 12 aluminum components for Batch No. 3 testing to be processed with Picklex®, and finished with the standard powder coat material for aluminum, Spraylat PE6639M. Upon receipt of processed components, Commercial Partner No. 1 measured for paint thickness and adhesion by tape following the ASTM D 3359 method described in Appendix D. See Table 7 for adhesion by tape results. The adhesion by tape results were superior for all three variations using Picklex® compared to production samples from Commercial Partner No. 1. Further testing is recommended to validate these results on a production scale.

Table 7. Comparison of Commercial Partner No. 1's Production Samples versus Picklex® Treated Samples(3)

Sample Type	Adhesion Rating
Production Samples from Commercial Partner No. 1	0B-2B
DDD Picklex® Rinse (45 sec) Chromate Conversion Coating	4B
EEE Picklex® Rinse (45 sec)	3B-4B(a)
FFF Picklex® Quick Rinse	4B

(a) Only one sample received 3B rating. The remaining samples received 4B rating.
(b) Reference Appendix C for raw data provided by Commercial Partner No. 1.

Pencil Hardness

ASTM D3363 *Standard Test Method for Film Hardness by Pencil Test* describes a procedure for rapid, inexpensive determination of the film hardness of an organic coating on a substrate. It was used here as a quality control check much like dry film thickness. The powder coatings used in this study were in the hardness range of HB to 3H with no discernable differences from substrate to substrate or scenario to scenario. This supports the hardness being a function of the coating itself and not the surface preparation or test substrate.

Impact

Impact was another way to evaluate adhesion of a coating to a substrate. The field testing replicated the laboratory tests through processing and evaluating groups A and B of Batch No. 1 for aluminum and J and K of Batch No. 1 for steel (all defined in adhesion by tape section). Phase II achieved additional promising results than Phase I for both aluminum and steel. For Aluminum, Phase I found impact adhesion between conventionally pretreated panels and Picklex® pretreated panels to be equivalent. Table 6 shows the average impact results for each group and the panels, which failed impact adhesion. Group A had one failure, A6, showing that Picklex® pretreated panels have slightly higher impact resistance than conventionally pretreated panels for aluminum. Impact resistance also showed that Picklex® can serve as at least a mild degreaser for steel, comparing group BB's average of 155 to group CC's similar average of 158 (both groups are defined in adhesion by tape section). Also, the comparison between F and S for aluminum and M and BB for steel show that the results are consistent.

Certain groups of aluminum panels from Batch No. 1 and 2 failed the impact adhesion test at 4 foot-pounds. All of the groups that were powder coated with Spraylat PEB1867C, which is not the standard powder coat material for aluminum, failed the test except for Group X which received very low passing results. These results suggest that the powder coat material and not the pretreatment or conversion coating caused the failures. Also groups Y and Z which did not receive a rinse after Picklex® failed the impact adhesion test at 4 foot-pounds. These results show that at least a quick rinse is needed after processing in Picklex®.

Bend

The mandrel bend test was provided as another way to evaluate adhesion of a coating to a substrate. Results for bend tests for Batch Nos. 1, 2, and 3 are listed in Table 6. In Phase II, pretreatment rather than top coating was being investigated; therefore, ASTM D 522 was modified in the definition of pass/ fail. In this study, visible cracking was not a "fail" unless coating was removed by pressure-sensitive tape at the cracking site. Failure was defined as loss of coating adhesion.

In Batch No. 1, only one aluminum panel, G12, failed this test while two panels with identical process steps passed. G12 (Al-T-P5M-R-w-Cr-R-A-SB) was powder coated with Spraylat PEB1867C, which is not the standard powder coat material for aluminum. All panels from Group B (Al-T-P5M-R-w-Cr-R-A-CW) passed the bend adhesion test. The only difference between Group G and Group B's test sequences were the different powder coat material demonstrating that G12 most likely failed because of the powder coat material and not the pretreatment or conversion coating. Certain groups of aluminum panels from Batch No. 2; Groups V, X, and RR; were powder coated with Spraylat PEB1867C and failed the bend adhesion test. These results also support the powder coating material causing the bend adhesion failures.

The overall results for bend adhesion demonstrate that Picklex® can be used as both a pretreatment and conversion coat for steel and the processing time in Picklex® can be reduced to 90 seconds. In Batch No. 2, steel panels having zinc phosphate conversion coating, Groups AA and EE, failed the bend test. Steel panels, from groups BB and CC, processed in Picklex® for 90 seconds, rinsed in DI water for 45 seconds, dried, and then powder coated (skipping zinc phosphate conversion coating) passed the bend adhesion test. These results demonstrate that zinc phosphate conversion coating is not needed to achieve passing bend adhesion results for steel. In Batch No. 1, all steel panels passed the bend adhesion test. Comparing groups AA and EE to Group O from Batch No. 1, the only difference is that the Picklex® process time was reduced from 5 minutes in Batch No. 1 to 90 seconds in Batch No. 2. Both groups received zinc phosphate conversion coating. The bend adhesion results show when applying a zinc phosphate conversion coating, the process time in Picklex® needs to be greater than 90 seconds.

The field test (Phase II) bend results for groups A versus B (aluminum panels) and groups J versus K (steel panels) verify the results from previous laboratory test (Phase I) when comparing conventionally pretreated panels to Picklex® pretreated panels. This result demonstrates that equivalent results were obtained between the two tests. The bend results for group CC (no degreasing step on steel panels) compared to group BB (panels were degreased) give equivalent passing results and show superior results over the conventionally processed panels in Batch No. 2. The equivalent results between the two groups are another indication that Picklex® can serve as a degreaser for steel. Group DD had one failure, which showed cracking during the bend test demonstrating the need for steel to be rinsed after processing with Picklex® and before powder coating. Group GGG, Figure 4, is a replicate of group DD. Note the inconsistent surface. Groups Y and Z, aluminum panels that were processed in Picklex® and not rinsed before drying also failed the bend test. These results also support the need for a rinse after Picklex®.

Figure 4. Oven Dried Steel Panels with No Rinse after Picklex®

Corrosion

Corrosion testing used exposure in a salt fog chamber following ASTM B 117, Standard Practice for Operating Salt Spray Apparatus. Powder coated panels were scribed with an X, protected by tape on the edges, and exposed in a standard salt spray cabinet for periods up to 1760 hours. All panels were inspected at 200 hours for signs of corrosion and returned to the salt spray cabinet for continued exposure. Salt fog data are included in Appendix A. Table 6 shows the results. Length of salt fog exposure was determined by time of processing and project end date. Batch No. 1 test panels were observed to 1760 hours. Batch No. 2 test panels were observed to 1277 hours. Batch No. 3 panels were observed to 144 hours. The failure point was defined for this study as loss of adhesion. Pressure-sensitive tape was placed over the scribed area on the dried panel and removed. If coating was removed with the tape, the panel failed.

Aluminum panels from Batch Nos. 1, 2, and 3 did not display loss of adhesion due to corrosion within the time frame of this study. Steel panels from Batch No. 1 did not show loss of adhesion until 1376 hours compared with similar panels from Batch No. 2, which showed loss of adhesion by 608 hours.

Comparing groups A versus B and J versus K (all groups from Batch No. 1), the salt fog results show that the laboratory process test results were verified in the field. During Phase I and II, equivalent corrosion resistance was noted when comparing commercially pretreated panels versus Picklex® pretreated panels. In Phase II Batch No. 1, both A and B groups of panels showed no corrosion at 1760 hours yielding equivalent corrosion resistance for aluminum also. For steel, both conventionally pretreated and Picklex® pretreated panels had corrosion on scribe and no undercut noted at 1760 hours when the samples were removed. One panel, J2, had loss of coating adhesion on scribe at 1376 hours and was removed. China White's process specification states corrosion resistance for 1000 hours. Group CC, in which Picklex® also replaced degreasing, showed the same amount of corrosion resistance as group BB, which was degreased, with loss of coating adhesion on scribe at 893 hours and sample was pulled from test.

The equivalent level of corrosion resistance is another factor in favor of using Picklex® as the degreaser for steel. When comparing group BB to M, it is found that higher corrosion resistance is achieved with a longer immersion time for Picklex® on steel, 893 hours for 90-second immersion versus 1376 hours for 5 minute immersion. For aluminum, the same amount of corrosion resistance was achieved when the immersion time was decreased shown by comparing Group F and S. Both groups of aluminum panels remained in the salt fog chamber for the duration of the test, 1760 and 1277 hours, respectively.

Batch No. 1 aluminum panels were pulled from the salt fog chamber at 1760 hours with no corrosion noted. Batch No. 2 aluminum panels were pulled from the salt fog chamber at 893 hours with no corrosion noted. After the salt fog test samples were removed from the exposure cabinet, the aluminum samples which showed no corrosion were tested for adhesion by tape to reveal possible loss of adhesion in the corrosive environment. Most of the aluminum panels retained high adhesion values of 5B and 4B equivalent to initial adhesion by tape values. Groups which were processed with Picklex® serving as the pretreatment and conversion coat, rinsed at least with a quick rinse, dried, and then powder coated with china white received at least a 4B rating. Two sets showed significant loss of adhesion. Z (Al-C-P3OS-N-w-N-N-O-CW) and Y (Al-C-P30S-N-w-N-N-O-CW) had panels receiving the lowest adhesion by tape ratings ranging from 0B to 2B. Both groups Y and Z were <u>not rinsed</u> before drying. This suggests that at least a quick rinse is needed after processing aluminum panels through Picklex® before drying them. Figure 5 shows panel Z 25 which received a 0B rating.

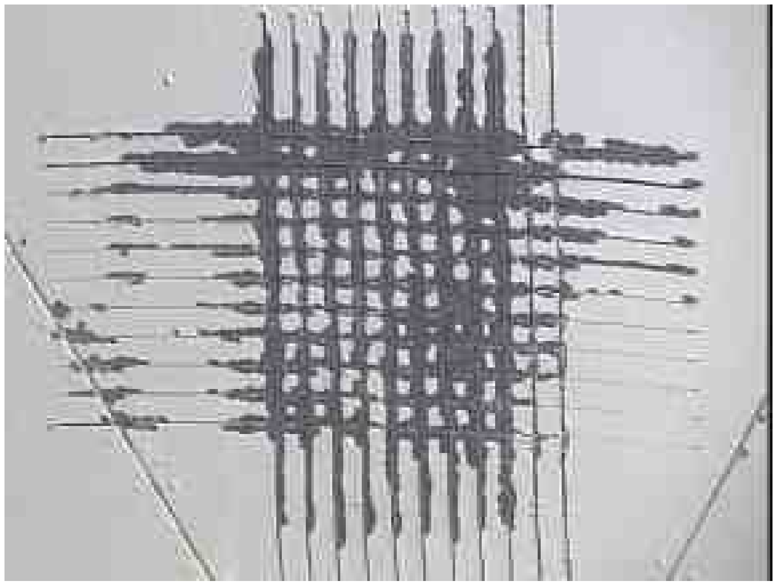

Figure 5. Panel Z 25 after Adhesion by Tape after Salt Fog Exposure

Engineering Assessment

Each Picklex® application was evaluated for the economic impact and cost advantages relative to the conventional processes in parallel to the laboratory testing provided above. Both the engineering design information and the economic evaluation are preliminary to guide subsequent Picklex® test program development and application priority, and especially, to address the most significant cost elements.

The objective of this assessment is to evaluate a preliminary engineering design and economics to guide the subsequent development and potential commercial implementation. Both capital and operating costs are estimated as incremental costs relative to the conventional processes. The capital cost estimates are order of magnitude and include 50% contingency because of the limited design detail available and the uncertainties of a general evaluation rather than site and application specific. A more detailed evaluation using site specific economic data will improve the accuracy and significance of the results in the future. Based on the commercial operation information obtained at Mills the engineering assessment has been revised to include commercial practice and potential labor savings. Several assumptions are included about consumption, make-up, and waste disposal rates that need to be verified during the technology development to produce a comprehensive evaluation.

A preliminary engineering assessment for each of the following surface-finishing applications are evaluated below relative to the Picklex® process based on the available test data.

- Chromate conversion coating on aluminum
- Zinc phosphatizing on steel.

Design Basis

A standard 500-gallon tank (36 inches by 72 inches by 48 inches) was used for all process steps to accommodate an assumed size piece to be handled. This simplified the preliminary design assessment and economics while recognizing that some operations may require different capacities and production rates to be addressed in subsequent iterations of the Engineering Assessment.

The following assumptions were used in this evaluation:

1. Metal surface treatment shop operates 8 hours per day and 5 days per week for 2,080 hours per year.
2. The use of Picklex® does not require changing the composition or operation of the plating bath(s) or subsequent surface finishing operations.
3. Comparable product quality is achieved with each comparison of conventional processing and the Picklex® alternative process.
4. The following chemical costs are used:
   - Hydrochloric acid, 37% HCl, \$72/ton
   - Sodium hydroxide, USP pellets, \$1.70/lb
   - Alumiprep® 33, as used, \$10/gallon (Henkel Surface Technologies, 2000)
   - Chromate conversion coating, \$10/gallon (Henkel Surface Technologies, 2000)
   - Picklex® as used, \$40/gallon (ICP, 2000)
5. The chemical consumption and make-up rates are as follows: Alkali cleaner is replaced every 6 months (2,500 lb NaOH/yr); Picklex® is replaced every 12 months (500 gal/yr); pickling solution is replaced every 6 months (10,000 lb HCl/yr); rust remover is replaced every 6 months (10,000 lb/yr); Alumiprep® 33 is replaced every 6 months (1,000 gal/yr).
6. Use of Picklex® solution can replace both the alkali cleaning and the chromate conversion coating in the surface preparation of aluminum for powder coating applications.
7. Waste disposal cost of replacing the chemical baths to averages \$0.50/lb.
8. Only one operator is needed for each line.
9. Operator's wage is \$20/hour.
10. Workload is consistent throughout year.

Chromate Conversion Coating on Aluminum

Figure 2 illustrates the sequential process steps for both conventional chromate conversion coating on aluminum and the alternative Picklex® process. As shown, the use of Picklex® (Al-P-R-N-N-PC) replaces both the pretreatment (alkali cleaning, 2 rinse steps, etch, 3 rinse steps, deoxidizer, 2 rinse steps) and chromate conversion coating (chromate conversion coat and three rinse steps).

The revised capital cost savings associated with eliminating these process steps and associated equipment are estimated to be $254,000 because of the decreased number of process steps. Using the same factored cost estimate of Phase I that includes piping, installation, electrical, instrumentation and controls, utilities and other services the cost savings was calculated. Table 8 provides detailed cost elements that were estimated in the engineering assessment.

- **Table 8. Capital Cost Savings**

Cost Element	Commercial Pretreatment and Chromate Conversion Coating on Aluminum	Commercial Pretreatment and Zinc Phosphatizing on Steel
Tank	$27,000	$15,000
Spill Containment	$1,500	$750
Pump	$26,000	$26,000
Filter	$7,200	$7,200
Mixer	0	0
Heater	$8,000	$6,000
Power Supply	0	$8,000
Subtotal PEC	$69,700	$62,950
Installation (30% PEC)	$21,000	$19,000
Piping (30% PEC)	$21,000	$19,000
Instrumentation (10% PEC)	$7,000	$6,300
Electrical (10% PEC)	$7,000	$6,300
Utilities		
Engineering (33% PEC)	$23,000	$21,000
Contingency (50% PEC)	$35,000	$32,000
Working Capital		
Total Capital Savings	$254,000	$230,000

Empty cells indicate cost element was not estimated.
PEC = purchased equipment cost.

Additional savings are not included for incremental cost of building/floor space, water treatment, and ventilation. These additional savings could be significant for a new facility (Greenfield site) or a total facility refurbishment that included these ancillary components, especially if the freed-up space enabled additional production capacity to be brought on line.

Overall, the operating costs for conventional pretreatment of aluminum are $46,000 higher than the alternative Picklex® process. Table 13 presents the estimated operating cost savings, including the direct labor costs based on the process time needed at Mills. The assumptions included only one operator is needed, the hourly wage is $20, and the workload is consistent throughout the year. The process time not including degreasing, drying, and powder coating (times are the same for both commercial and Picklex® processes) is 20 minutes for pretreatment and chromate conversion coating on aluminum and 1 minute for processing with Picklex® as the pretreatment and conversion coat. The chemical costs remained the same as Phase I. Consistent with Phase I, both conversion coating processes would have the same powder top coating operations and costs.

Zinc Phosphatizing on Steel

Figure 1 illustrates the process steps for both conventional zinc phosphatizing on steel and the Picklex® process. The use of Picklex® replaces the pretreatment (electro cleaner and one rinse step) and conversion coating (conversion coat and three rinses).

The capital cost savings, shown in Table 8 is estimated to be $ 230,000 using the same factored cost estimate as Phase I which includes piping, installation, electrical, instrumentation and controls, utilities, and other services. The revised capital cost savings results from the reduced number of steps between Mills' process and the Picklex® alternative.

As previously stated, additional savings are not included for incremental cost of building/floor space, water treatment, and ventilation.

The operating cost savings, presented in Table 9 include the direct labor costs. Using the assumptions described in the chromate conversion cost estimate, the direct labor costs for using zinc phosphate are $36,600 higher than using Picklex®. The estimated time for processing one load through the zinc phosphate line is 25 minutes compared to the 2 minutes process time for Picklex® with steel.

▪ **Table 9. Operating Cost Estimate**

Cost Element	Unit Cost	Conventional Process		Picklex® Process	
Chromate Conversion Coating on Aluminum					
Alkali Cleaner	$1.70/lb	2,500 lb	$4,250/yr		
Aluminprep® 33	$10/gal	1,000 gal	$10,000/yr		
Chromate Conversion Coating	$10/gal	1,000 gal	$10,000/yr		
DI Water	$0.05/gal	10,000 gal	$500/yr	500 gal	$25/yr
Picklex®	$40/gal			500 gal	$20,000/yr
Waste Treatment	$0.50/lb	12,000 lb	$6,000/yr	5,000 lb	$2,500/yr
Direct Labor	$20/hr	2000 hr	$40,000/yr	100 hr	$2,000/yr
Maintenance					
Operating Supplies					
Utilities					
Analytical Lab					
Fixed Costs					
Indirect Costs (OH)					
Subtotal			$70,750/yr		$24,525/yr
Indirect Costs (OH)					
Subtotal			$70,750/yr		$24,525/yr
Chemicals		Quantity	Annual Cost	Quantity	Annual Cost
Zinc Phosphatizing on Steel					
Aeroclean DN 30	$4.91/gal	1,000 gal/yr	$4910/yr		
HCl	$72/ton	5 ton/yr	$360/yr		
Aerocote #3	$7.05/gal	1,000 gal/yr	$7050/yr		
DI Water	$0.05/gal	10,000 gal	$500/yr	500 gal	$25/yr
Picklex®	$40/gal			500 gal	$20,000/yr
Waste Treatment	$0.50/lb	10,000 lb	$5,000/yr	5,000 lb	$2,500/yr
Direct Labor	$20/hr	2000 hr	$40,000/yr	170 hr	$3400/yr
Maintenance					
Operating Supplies					
Utilities					
Analytical Lab					
Fixed Costs					
Indirect Costs (OH)					
Subtotal			$58,000/yr		$26,000/yr

Empty cells indicate that cost element does not exist.

Results and Discussion

Several qualitative processing advantages of Picklex® were found during Phase I, which include adequate draining time, easy agitation, and slow evaporation rate. An extended bath use period test was performed during Phase I, which showed that the bath did not form solids with use, and that Picklex® operates at ambient temperature. These advantages of Picklex® were confirmed during Phase II.

The field testing at Mills showed that the use of Picklex® reduced the number of process steps considerably compared to commercial processes. The optimum Picklex® process for aluminum and steel that receives a powder coat finish may consist of degreasing, Picklex® immersion dip (30 seconds for aluminum and 90 seconds for steel), and a quick rinse step (in and out). Then the processed parts may be dried and powder coated. The commercial processes for both pretreatment and chromate conversion coating for aluminum and pretreatment and zinc phosphate conversion coat on steel include many more process steps (11 more steps for aluminum and 5 more steps for steel).

A disadvantage of Picklex® is that the near term unit cost is $40/gallon; however the remaining operating and capital cost savings may offset the difference in chemical costs. Also Picklex® has a slow evaporation rate and high tolerance to contamination and so does not need to be replaced on a regular basis.

The revised engineering assessment showed the economic impact of using Picklex® as a replacement for chromate conversion coating and zinc phosphatizing. Both the capital cost savings and the annual operating cost savings are summarized in Table 10.

Table 10. Summary of Cost Reductions of Using Picklex® Instead of Conventional Pretreatments

Cost Reduction	Savings Relative to CCC on Aluminum	Zinc Phosphatizing on Steel
Capital Cost Savings	$254,000	$230,000
Annual Operating Cost Savings	$ 46,000	$ 36,600

Recommendations

Steel and aluminum components were evaluated during Phase II. The Picklex® treated aluminum components provided improved adhesion over the current production technique. Based on the positive test results, an application specific field test on aluminum components is recommended.

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