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NCEL Technical Note

N-1755 July 1986 By Peter J. Hearst, PhD Sponsored by Naval Sea Systems Command

Chemical Cleaning of Ship Tanks -Laboratory Investigation of Stripping Agents for Epoxy Coatings

ABSTRACT Stripping agents for epoxy coatings were investigated for use in a Ship Tank and Bilge Chemical Cleaning Hardware and Processing System (STABCHAPS). This system recycles hot solutions for the removal of contaminants, rust, and coatings. Available commercial stripping agents did not prove to be satisfactory. A very effective stripping agent, NCEL Stripping Agent H4, was developed. It consists of 77% N-methylpyrrolidone, 22% ethanolamine, and 1% Igepal C0630. However, this stripping agent is too hazardous for general use and needs to be modified.



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INTRODUCTION

The Naval Civil Engineering Laboratory has developed a Ship Tank and Bilge Chemical Cleaning Hardware and Processing System (STABCHAPS). This system sprays and recirculates hot solutions for cleaning, stripping, derusting, and passivating tanks and bilges. Typically 500 gallons of solution are sprayed at 50 gpm, 100 psi, and 180 °F through a set of nozzles. Solutions can also be sprayed by hand lance at 2,000 psi.

The STABCHAPS was developed as an alternate to sandblasting and its attendant air pollution and heavy metal water pollution problems. For removal of alkyd paints with STABCHAPS, alkaline stripping agents could be used. For removal of epoxy coatings or other chemically resistant coatings, no suitable stripping agents were available.

The results of laboratory investigations of alkaline paint stripping, citric acid removal of rust, and steel passivation with dilute aqueous amine solutions were presented in Reference 1. The chemical cleaning process based on these studies that would be used with the STABCHAPS has been described in Reference 2. The alkaline paint stripping, as discussed in Reference 1, is effective for alkyd coatings but not for epoxy coatings or other chemically-resistant coatings that are used in Navy tanks, bilges, or other spaces. Epoxy stripping agents commonly used or commercially available were toxic and chemically aggressive to equipment and transfer hoses, and were not considered suitable for spraying aboard ships with the STABCHAPS. Consequently, no experiments had been performed with such agents.

Epoxy stripping agents have now been further investigated because of the great need for such agents. The objective of this phase of the work was to find a stripping agent that could be used with the STABCHAPS, as modified to handle more aggressive materials, to remove an epoxy coating from the walls of a ship's tank. The results of this work are presented in this report.

EXPERIMENTAL WORK

Screening Tests

Samples of various commercial stripping agents were obtained for laboratory screening tests of their ability to remove epoxy coatings. Many of these agents contained volatile components and were designed to be used with floating seal layers, generally of mineral oil, to prevent evaporation losses. Where seal layers were present, these were removed before sampling.

For the screening tests, 1- by 3-inch epoxy-coated steel test panels were half immersed for 4 hours in 200-ml samples of the stripping agents contained in 400-ml beakers. The stripping agents were held at 180 °F (82 °C) in a water bath and were stirred with magnetic stirrers. The panels were inspected at hourly intervals for visual changes and were probed with a spatula to detect softening of the coating films.

Most of the commercial stripping agents had little effect on the epoxy coatings. Those agents that gave at least fair performance in the screening tests were subsequently tested in a laboratory spray apparatus. (Exceptions were agents that were not considered suitable candidates for STABCHAPS because of probable material or toxicity problems, such as those containing phenols or low-boiling components.) The results of the screening tests for these agents, which are rough estimates of the coating removal, are shown in Table 1. The screening tests are more fully reported in Reference 3.

Laboratory Spray Apparatus

The laboratory spray apparatus previously used to simulate the operation of the STABCHAPS (and described in Reference 1) was modified to handle more aggressive chemicals. The pump, which had an epoxy body, was replaced by a stainless steel gear pump, and the polypropylene spray chamber was replaced by a stainless steel spray chamber.

An overall view of the spray apparatus is shown in Figure 1. The spray chamber serve as a reservoir for the stripping agent. It was charged with 2 liters of the agent, although only 1 liter was required to cover the cylindrical screen filter from which the liquid was drawn through 9/16-inch Teflon tubing into a pump. This 100-psi pump forced the liquid through a 1/4-inch stainless steel loop immersed in a water bath and through a moving arm, also made of 1/4-inch stainless steel tubing, at the end of which the spray nozzle (Spraying Systems #Y-TT-1510-SS) was mounted. The spray nozzle was pulled down and allowed to move back up by an eccentric attachment on a slowly moving wheel powered by a small variable speed motor in such a manner that the spray passed across the surface of the test panel 34 times per minute. The pressure drops in the stainless steel tubing were such that a pressure of 90 psi at the pump provided a measured pressure of 60 psi at the stationary end of the arm and a calculated pressure of 55 psi at the nozzle (considering the proportional additional pressure drop in the arm). The pressure at the pump was regulated by varying the speed of the 1.5 hp dc pump motor and by controlling the flow through a bypass valve. A schematic flow diagram is shown in Figure 2.

The spray chamber is shown in greater detail in Figure 3. During operation, the large viewport was partly closed by two pieces of plate glass (not shown) to leave an opening 1 inch wide and 6 inches high for the spray nozzle and spray. A polypropylene spray guard (also not shown) was mounted on the spray nozzle assembly to catch any spray that rebounded from the chamber.

The temperature of the stripping agent was measured at the nozzle assembly with a thermocouple. A temperature of 180 °F (82 °C) at the nozzle was achieved with a water bath temperature of 88 °C. The flowrate was approximately 1 gpm. This was the flowrate for water at room temperature with a pump pressure of 90 psi.

The approximately 3- by 6-inch hot-rolled steel test panels, 1/16-inch thick, were coated with a total of 10 mils of MIL-P-24441 epoxy coating (Formulas 150, 151, and 152). Two parallel scribe marks and two intersecting scribe marks were placed on the lower third of each test panel.

(These are shown to 90% scale in Figure 4 and other figures.) The panels were mounted on a magnetic holder at a distance of about 12 inches from the spray head.

At the beginning of each run, the water bath was preheated, the test panel was placed on the magnetic holder, the stripping agent was placed in the chamber, and the window and splash guard were replaced. The spray nozzle was lowered so that the spray would miss the test panel, and the stripping agent was circulated until it reached the desired temperature. Besides the 1,500 watts of heat provided for normal temperature control, an additional 2,000 watts were supplied to the water bath during this period. The test panel was sprayed for 1 hour and, while the spraying was stopped, the panel was visually examined in the test chamber with the aid of a flashlight. After reheating the circulating stripping agent, the spraying was continued for a second and, usually, a third hour. At this time, or after it had remained in the chamber overnight, the panel was removed, washed with alcohol if the run was completed, and photographed. At the time of the visual ratings, the liquid level was determined by means of the equivalent of a sightglass made with Teflon tubing.

Spray Test Results

The results obtained with the laboratory spray apparatus are summarized in Table 1. For the commercial stripping agents, the approximate compositions provided by the manufacturer are given to the extent known. Commercial Agent A was diluted with water to 50% concentration; Commercial Agents B and C were used without their mineral oil seal layers. The NCEL formulations are identified by alphanumeric designation and the compositions are given. The coating removal is described as estimated after 3 hours of spraying (except where other time periods are given) for the upper two-thirds of the panel and for the lower third of the panel that contained the scribe marks. The percent of coating removal was estimated visually. For many of the runs, the results are also shown in photographs of the test panels after 3 hours of spraying at 180 °F, or as otherwise indicated, in Figures 4 to 11.

The above results were obtained with panels that were all prepared in the same manner (Series B panels). These panels (and also an earlier set of Series A panels) had been prepared with a small hand-held airless spray apparatus. (The coatings on the Series A panels were slightly, but not significantly, more difficult to remove than those on the Series B panels, as judged by experiments with N-methylpyrrolidone and with H3C.) A new set of panels (Series C) was carefully prepared with fresh epoxy coatings. With these panels, H3C required an approximately four-fold increase in time for coating removal, as reported in Table 1.

The approximate losses of stripping agent during the spraying operation were measured by subtracting the liquid level determined at the Teflon gauge glass from the original 2 liters of stripping agent. The percent losses and the percent losses per hour for the NCEL stripping agents are shown in Table 2.

The loss of ethanolamine from stripping agent H3C remaining in the spray apparatus was determined by titrating a 10-ml aliquot of the original stripping agent and of the final stripping agent in 25 ml of water with 1N hydrochloric acid and dividing the difference by the former value. These results, and the approximate percent losses per hour, are shown in Table 3.

DISCUSSION OF RESULTS

Commercially available stripping agents for epoxy coatings are usually intended for use in hot tanks and are intended to be used with mineral oil seal layers to reduce evaporation. They generally are not intended for spray operation. Three commercial stripping agents were run in the series of spray tests reported herein. Other commercial agents were ineffective in the immersion tests, contained volatile or toxic ingredients considered unsafe for the STABCHAPS operation, or were received too late to include in these experiments.

Spray test results with the most promising commercial stripping agents, at a temperature of 180 °F (82 °C), were not encouraging (as shown in Table 1). Commercial Agent A was run in spite of poor immersion results because of claims that had been made. It had a very high evaporation rate in the spray test, and when water was added to replace evaporation losses, strong foaming resulted that filled the spray chamber in half an hour, during which no coating was removed. Commercial Agent B removed less than half the coating in three hours, and Commercial Agent C removed only small amounts of coating at the edges and scribes.

The stripping agents developed at NCEL were based on N-methylpyrrolidone, which is supplied by GAF Corporation under the trade name of M-Pyrol. This chemical had been patented for coating removal in thickened or paste form containing amines and surface active agents (Ref 4), but no formulations appeared to be available that would be suitable for spraying. N-methylpyrrolidone might also be contained in some of the commercial stripping agents tested, but because of its low toxicity this material need not be listed in safety data sheets.

The N-methylpyrrolidone by itself (Run SP7) performed almost as well as the best commercial agent that had been run. The addition of 20% diethanolamine to provide alkalinity and of two different surface active agents to provide better wetting (in Runs SP8 and SP11) did not improve the performance. The diethanolamine had been chosen because of its water solubility, which would be advantageous in subsequent rinse operations. It was also chosen in preference to ethanolamine because of its lower volatility, which would reduce any change in concentration that might occur during the spraying operation.

The use of ethanolamine, also called monoethanolamine (MEA), instead of diethanolamine markedly improved the stripping action. The most effective formulation prepared to date, H3C, removed all the epoxy coating in little more than 1 hour (in Run SP10), whereas the best of the commercial products had removed much less than half the coating from a similarly prepared panel in 3 hours.

The ethanolamine is more alkaline and, therefore, more aggressive than the diethanolamine. In aqueous solutions, at concentrations of 30% and temperatures of 25 °C, the pH values are reported to be 12.7 and 11.5, respectively (Ref 5). (The former value might rise to about 13 in more concentrated solutions, but pH values are not applicable to the essentially anhydrous stripping agent.) The ethanolamine also has a lower boiling point and greater volatility than the diethanolamine, and there is thus a change in concentration during use. Under the experimental conditions of the spray test, the loss of stripping agent was about 6% per hour, and in the stripping agent remaining in the apparatus, the concentration of the ethanolamine was reduced about 6% per hour (for example, from 20% to about 18% in 2 hours). These losses could vary greatly under other experimental conditions and especially with different types of ventilation. The loss of ethanolamine is less at lower temperature, as shown in Table 3.

The much greater effectiveness of using a sprayed solution, as compared to immersion of a sample, had been evident from previous experiments with stripping agents (Ref 1), and increased reactivity of fluids in motion is considered common knowledge. It therefore was surprising to note that considerable stripping occurred at the backs of the sprayed panels. At the back of the panel sprayed with N-methylpyrrolidone for 3 hours (in Run SP7 and as shown in Figure 12) the white epoxy topcoat curled up with adhering grey intermediate coat, while most of the green primer remained on the steel panel, also with adhering grey intermediate coat; the failure was primarily in the intermediate coat. At the back of the panel sprayed with H3C for 2 hours (in Run SP10 and as shown in Figure 13), about 70% of the white topcoat was removed, but where this was removed, the undercoats were removed also; the failure appeared to be in the primer at the primer-metal interface.

Stripping agent H3C stripped a Series B panel in little more than an hour, but six hours were required to strip a Series C panel. This illustrates the variation in chemical resistance and other performance of identically designated coating systems (in this case the nominally identical MIL-P-24441 systems) depending on the condition of the formulated coating and the method of application. The performance of a stripping agent with a coating on a panel will not necessarily relate directly to the performance with the same type of coating in the field. Furthermore, a good epoxy stripping agent may effectively remove most epoxy coatings, but not necessarily all of them.

The time required for stripping is strongly affected by the temperature. The approximately 1-1/4 hour required for stripping of a Series B panel with H3C at 180 °F was increased to about 6-1/4 hours at 160 °F. The stripping time more than doubles with every 10 °F drop in temperature, as shown by the semi-log plot of Figure 14. Thus, the temperature effect is much greater than the typical doubling of the rate of first order chemical reactions with every increase of 10 °C (18 °F). (Run SP-12, at 150 °F, required less time than Run SP-15, at 160 °F; however, this run was interrupted overnight, during which time the stripping action presumably continued.)

The time required for stripping is also strongly affected by the presence of water. The addition of 10% water to H3C almost tripled this time. It had the same effect on the stripping rate as lowering the temperature about 14 $^{\circ}$ F.

The stripping agent H3C could probably be further improved by varying the composition of the N-methylpyrrolidone and ethanolamine mixture and by changes in the surface active agent. An increase in the ethanolamine and a reduction of the more expensive N-methylpyrrolidone would reduce the initial cost of the stripping agent but might also reduce the effectiveness and increase evaporation losses. Thus, more research should be performed. However, a stripping agent is now available that is useful for demonstrating the potential effectiveness of the STABCHAPS in the removal of epoxy coatings from tanks or other spaces on ships.

FIELD TEST CONSIDERATIONS

The experimental results discussed above indicated that the following matters should be given consideration in the running of any field tests using stripping agent H3C or its equivalent with the STABCHAPS.

Availability

Both the N-methylpyrrolidone and the ethanolamine, or monoethanolamine (MEA), are readily available in drum quantities, and the Igepal CO630 is available in 50-pound pails. A very slight modification of the composition of H3C would allow the preparation of the stripping agent at the test site by mixing the premeasured quantities as supplied in the full containers. This change in composition would not be expected to affect the performance of the stripping agent. The modified agent, called NCEL Stripping Agent H4, has the following composition:

Component	Containers	Quantity (gal)	Proportion (by volume)
N-methylpyrrolidone	7 ea. 55-gal drums	385	77%
Ethanolamine	2 ea. 55-gal drums	110	22%
Igepal CO630	l ea. pail of 5.8 gal	6	1%
Total	10 containers	501	100%

The current cost of these components, excluding freight, is \$5,054, \$496, and \$84, respectively, for a total of \$5,634.

Process Factors

Effects of Temperature. Because the rate of coating removal is very dependent on the temperature, the coating surface temperature should be monitored. Measurement of the temperature of the runoff spray solution and of the tank wall would be desirable.

Effect of Water. The presence of water reduces the effectiveness of the stripping solution; therefore, the tank should be dry or nearly dry before the stripping agent is used. The approximate amount of water present could be determined by analysis of the stripping agent at the end of the stripping process.

Another reason for keeping the water content low is the fact that aqueous monoethanolamine solutions are corrosive to carbon steel. In the short contact time of the STABCHAPS process, this effect should not be critical. Effect of Stagnant Stripping Agent. Coating removal by immersion without agitation was not as effective as coating removal by spray. Therefore, stagnant stripping agent at the bottom of a tank may produce less coating removal than the spray on the walls of the tank. Any stagnant stripping agent should be removed as completely as possible.

The comparative effect of spray versus immersion is indicated in Table 1. However, the results in the immersion tests are variable and the coating removal indicated is very approximate. The coating under a stagnant layer of stripping agent may still be strongly affected to allow easy removal by mechanical methods, such as water spray or scraping.

Loss of Stripping Agent

In the laboratory spray experiments, the air turbulence caused by the spray resulted in considerable loss of stripping agent (about 6% per hour), probably by a combination of evaporation and aerosol formation. The vacuum recycling procedure of the STABCHAPS, which is a type of air lift, would be expected to cause greater evaporation and atomization losses. If these losses are too great, it may be necessary to recycle the stripping agent by direct pumping of the solution, which is an alternative STABCHAPS procedure.

Loss of Ethanolamine

Ethanolamine is more volatile than N-methylpyrrolidone, and its loss in the laboratory spray experiments was more rapid than the loss of the total stripping agent. Thus, the loss of the ethanolamine concentration in the stripping agent during a run was again as great as the total loss of stripping agent, resulting in a total loss in 2 hours of about one-quarter of the ethanolamine charged into the system. This is of some concern because the ethanolamine, which is the only hazardous component in the stripping agent, has a very low threshold limit value (TLV) of 3 ppm.

The vapor pressure of ethanolamine at the stripping temperature of 180 °F (82 °C) is about 18 mm Hg (Ref 5). In a 20% solution of ethanolamine in the less volatile N-methylphrrolidone (which has a vapor pressure of about 12 mm Hg at 180 °F according to Reference 4), the effective vapor pressure of the ethanolamine might be as low as 3.6 mm, but it is likely to be higher. If the vapor pressure is 7.6 mm, it is one-hundredth of the atmospheric pressure of 760 mm. In an equilibrium condition, 1% by volume of the air in contact with the stripping agent would consist of ethanolamine. This is 10,000 ppm.

If, in the vacuum recycling of the STABCHAPS, the air is discharged at 150 °F (where the vapor pressure of ethanolamine is about 7 mm Hg), and if all suspended aerosol particles are removed, the discharge air could still contain about 3,000 ppm of ethanolamine. This would be 1,000 times the TLV.

The loss of stripping agent and the discharge of ethanolamine could be reduced by using a high efficiency cooling device. However, a 50 °F drop in temperature would not reduce the vapor pressure of the ethanolamine to much less than one fifth its former value, and the discharge at 100 °F could still be 200 times the TLV. Again, it appears that recycling in a closed system would be necessary instead of using the vacuum recycling.

Other Safety Considerations

Ethanolamine, besides providing toxic vapors, is also a very caustic material. Thus, all precautions necessary in the handling of strongly alkaline solutions must be taken in the handling of ethanolamine.

If the system is overheated, there could be a fire or explosion hazard. However, operation below the flash points of ethanolamine and N-methylphrrolidone, of 195 °F and 205 °F, respectively, should present no hazard.

Analytical Requirements

Changes in the composition of the stripping agent during use and the amount of ethanolamine discharged into the atmosphere should be determined during field trials.

The main changes in composition of the stripping agent would be the loss of ethanolamine and the addition of water from any process residues in the STABCHAPS. The change in ethanolamine concentration can be determined by titration of an aqueous solution with hydrochloric acid. The ethanolamine concentration could also be determined by gas chromatography. The addition of water, which would be soluble in the stripping agent, could be determined by the Karl-Fisher method.

The amount of ethanolamine in air could be determined by scrubbing a known amount of air in aqueous acid or in water, followed by titration of the reduced acidity or increased basicity, respectively. This assumes that ethanolamine is the only alkaline constituent present.

Reuse or Recovery

If care is taken to prevent contamination with water, it appears likely that the stripping agent can be used many times by adjustment of the composition. If the coatings that are stripped come off as flakes (as was the case in the epoxy stripping experiments) rather than being largely dissolved, they can be removed by filtration. The loss in ethanolamine concentration could be adjusted by the addition of the appropriate amount of ethanolamine, and the overall loss of stripping agent could be made up with new stripping agent. Accumulated water could possibly be removed by a drying agent, but this has not been investigated.

From spent solutions that could not be reused with the above adjustments, the major constituents could probably be reclaimed by commercial distillation. In such a distillation water would first be removed, the ethanolamine would be obtained, and the N-methylpyrrolidone would then be distilled.

FIELD TEST RESULTS

A pilot test for stripping of epoxy coatings with the STABCHAPS using NCEL Stripping Agent H4 was performed at the Puget Sound Naval Shipyard in June 1985. The test tank was a coated sludge tank, about 8 by 4 by 4 feet. The results are summarized here and are reported in more detail in another report (Ref 6). The test was started with the normal STABCHAPS procedure of recycling the process fluid with a vacuum airlift. But after 6 minutes of operation with the test solution at about 180 °F, it was clear from the visible cloud of vapors produced by the vacuum module exhaust that this recycling method was not appropriate. Titration of the stripping agent after this short operation showed that the ethanolamine concentration was reduced from 22.0% to 20.2%. Thus, about 8% of the ethanolamine was lost.

The test was resumed on the following day with a diaphragm pump attached to the outlet of the test tank for the recycling of the stripping agent. In this essentially closed system, there was no further appreciable loss of ethanolamine in the 5 hours of operation after restarting the test, as shown in Table 4. (The ethanolamine content was now closer to that of H3C, rather than that of H4, but no difference in performance would be expected for these two compositions.)

At the end of the 5-hour run, there were large areas in the direct path of the spray that had about 75% of the coating removed. (The coating removal in these areas was about 40% in 4 hours, and the coating removal might have been completed in the areas of direct spray if the test had been continued for another hour, or a total of 6 hours.) One support member of the tank, which originally had only 3 mils of coating, was completely clean at the end of the run. Other accessible areas of the tank originally had about 9 to 11 mils of coating. In some of these areas the coating was very soft at the end of the run, whereas in others it was still firm.

The MIL-P-24441 epoxy coating in the test tank had been cured by baking to provide better chemical resistance and therefore a worst-case situation for chemical stripping. When a black MIL-P-24441-epoxy-coated steel section from a submarine was subjected to the stripping operation in the test tank for 1 hour, about 50% of the coating was removed. The remainder was very soft and could probably have been removed with a water jet. The typical epoxy coating that would be stripped in a shipyard would thus be more easily removed than that of the test tank.

The temperature of the stripping agent during that test was approximately 170 °F, as shown in Table 4. The tank wall temperatures shown in the same table were measured on the outside of the coated test tank, and the temperature of the inside surface was probably appreciably warmer than the average measured temperature of about 150 °F. As discussed earlier and shown in Figure 13, the coating removal rate would be expected to double with a 10 °F rise in temperature. The results of the test show that NCEL Stripping Agent H4 can be effective in removing epoxy coatings in a closed system with recirculation at 180 °F, or at slightly lower temperatures.

CONCLUSIONS

1. An NCEL formulated stripping agent based on N-methylpyrrolidone, ethanolamine, and a surface active agent is considerably more effective in removing an epoxy coating by spray application than any environmentally-acceptable commercial agent tried to date.

2. NCEL Stripping Agent H4 can be effective in removing epoxy coatings with the Ship Tank and Bilge Chemical Cleaning Hardware and Processing System (STABCHAPS).

3. For use of Stripping Agent H4 with the STABCHAPS, recycling in a closed system is required and careful monitoring is important because of some of the physical, chemical, and hazardous properties of the ethanol-amine.

4. Stripping Agent H4 is probably not suitable for other than experimental use because the vapors generated would contain more ethanolamine than the allowable threshold limit value (TLV) of 3 parts per million.

5. Epoxy coatings may have differing resistances to stripping agents, even if they are of the same specification, because of differences in composition or application. Thus, a stripping agent that is shown to be effective in removing epoxy coatings may not be effective for all epoxy coatings.

RECOMMENDATIONS

1. Stripping agents for chemically resistant coatings should be further investigated to find agents that are less hazardous than NCEL Stripping Agent H4.

2. The effectiveness of alternative stripping agents should be investigated more systematically by determining the softening of the coating and the loss of adhesion that are produced.

3. If any further use is made of Stripping Agent H4, its corrosiveness should be determined, and if necessary, potential corrosion inhibitors should be investigated.

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REFERENCES

1. Naval Civil Engineering Laboratory. Internal Memorandum M-52-83-05: Chemical cleaning of ship tanks - Laboratory investigation, by Peter J. Hearst. Port Hueneme, Calif., Nov 1982.

2. ______. Internal Memorandum M-52-83-04: Chemical cleaning process for tanks, by Peter J. Hearst and Adolph Bialecki. Port Hueneme, Calif., Nov 1982.

3. ______. Internal Memorandum M-71-____: Screening tests of stripping agents for epoxy coatings, by Jennie Koff et al. Port Hueneme, Calif. (in preparation).

4. GAF Corporation. M-Pyrol Handbook. New York, N.Y., 1972.

2200022

5. Dow Chemical Company. The Alkanolamines Handrook. Midlands, Michigan, 1981.

6. Naval Civil Engineering Laboratory. Internal Memorandum M-71-85-27: Field test results of epoxy stripping agent, by J. Domingo and J. Zimmerle. Port Hueneme, Calif., Aug 1985.

Stripping			Coating Remov	Coating Remova	
Agent	Run	Composition	At Undamaged Area	At Scribed Area	by Immersion ^b
Commercial Agent A ^C	SP1	50% water, 38% furfuryl alcohol, 6.3% KOH, and other unspecified ingredients	no effect (in 0.5 hr)	no effect (in 0.5 hr)	no removal (slight lifting at edges)
Commercial Agent B ^C	SP3	An amine and other unspecified ingredients	15% of total coating	85% of total coating	100% removed
Commercial Agent C ^C	SP4	30% ethanolamine, 5% KOH, and other unspecified ingredients	negligible	3% at edges and and scribes	95% blistered
NMP	SP7	N-methylpyrrolidone	10% of total; 60% of topcoat	30% of total; 90% of topcoat	15% of total; 75% of topcoat
HIA	SP8	79.2% NMP, 19.8% diethanolamige, 1% Igepal CO210	10% of total (at edges); 5% pitting of toproat	20% of total; slight pitting of topcoat	20% removed
H1C	SP11	79.2% NMP, 19.8% diethanolamine, 1% Igepal CO630	8% of total (at edges); 10% pitting of topcoat; many small blisters	25% of total; 7% pitting of topcoat	5% removed at edges
НЗВ	SP9	79.2% NMP, 19.8% ethanolamine, 1% Igepal CO520	98% (in 2 hr)	98% (in 2 hr)	80% removed
НЗС	SP10	80% NMP, 20% ethanolamine, 1% Igepal CO630	100% (in 1.25 hr)	100% (in 1.5 hr)	100% removed ^e
90% H3C in water	SP13	90% H3C plus 10% water	85% of total [100% in 3.5 hr]	95% of total [100% in 3.5 hr]	25% removed
H3C at 170 °F	SP14	'	100% of total	100% (in 2.75 hr)	f
H3C at 160 °F	SP15		2% of total; 1% pitting of topcoat [100% in 6.25 hr]	15% of total [100% in 6 hr]	at edges only
H3C at 150 °F	SP12		3% pitting of topcoat; some blisters [100% in 5.5 hr ^R]	15% of total [100% in 5.5 hr ⁸]	at edges only
НЗС	SP19 ^h		,7% of total (at edges) [100% in 6 hr]	85% of total [100% in 6 hr]	i

Table 1. Epoxy Coating Removal With Stripping Agents

^aVisually estimated coating removal after 3 hr of spraying at 180 °F, except as otherwise noted. Other time periods are shown to the nearest quarter hour.

^bEstimated coating removal at the portion of a test panel immersed in the stirred stripping agent at 180 °F for 4 hr, except as otherwise noted.

 $^{
m C}$ The name of the commercial product is available to qualified requestors.

 d A product of GAF Corporation, New York, NY 10020.

^e90% removed in 2-1/2 hr, 100% in 3-1/2 hr.

 $^{\rm f}$ Variable results, from 2 to 35% removed.

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⁸The coating was removed in 3 hr at 150 °F on one day and an additional 2-1/2 hr at 150 °F during the next day. h

Using a Series C panel, instead of the Series B panels of the other runs, as described in text.

No immersion tests peformed with Series C panels.

Stripping Agent	Run No.	Time (hr)	Volume Loss (1)	% Loss	% Loss Per Hour
H1A	SP8	3	0.15	7.5	2.5
H1C	SP11	3	0.25	12.5	4.2
НЗВ	SP9	2	0.25	12.5	6.3
НЗС	SP10	2	0.25	12.5	6.3
H3C + 10% H ₂ O	SP13	3.5	0.55	27.5	7.9

Table 2. Volume Losses of Various Stripping Agents

^aSee Table 1 for composition. Runs at 180 °F.

^bOf the 2-liter stripping agent. Values are only approximate.

Table 3. Loss	of Ethanolamine	From Stripping	Agent H3C
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Temperature (°F)	Run No.	Time (hr)	% Ethanolamine Retained ^a	% Lost	% Lost Per Hour
180	SP9	2	87	13.0	6.5
180	SP10	2	88	12.0	6.0
180 ^b	SP13	3.5	89	11.0	3.1
170	SP14	3	89.9	10.1	3.4
160	SP15	6	84.9	15.1	2.5
150	SP12	5.5	86.3	13.7	2.5

^aComparative amount of hydrochloric acid required for titration to neutrality of aliquots of the final and original stripping agent dissolved in water.

^b90% H3C with 10% water.

Time ^a	Ethanolamine Content	Coating Lo	ss (%)	Temperature (°F)			
(hr)	(%)	Spray Area	Overall	Tank Wall	Solution		
Start	20.2						
1	20.2			162	170		
2	20.1			142	165		
3			10	150	165		
4		40	30	160	185		
5	20.1	75	40				

Table 4. Field Test Results

^aSpraying time on second day (after 6 minutes of spraying on first day).

^bAs analyzed, based on original ethanolamine content of 22.0% at beginning of first day.



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Figure 1. Spray apparatus.

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- 1. Coated test panel
- 2. Oscillating spray head
- 3. Thermocouple well
- 4. Eccentric drive
- 5. Spring

- 6. Moveable arm
- 7. Pressure gauge
- 8. Heat exchange coil
- 9. Pressure gauge

- 10. Variable speed pump
- 11. Bypass valve
- 12. Cylindrical screen filter
- 13. External teflon sight glass
- 14. 500-watt constant heater
- 15. Two 500-watt controlled heaters
- 16. Four 500-watt auxiliary heaters
- 17. Thermister for temperature controller (not shown)
- 18. Water reservoir for constant level water bath

Figure 2. Flow diagram for spray apparatus.









Figure 6. Coating removal with N-methylpyrrolidone.



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Figure 7. Coating removal with stripping agent H1A.





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Figure 12. Coating removal on reverse side of panel with N-methylpyrrolidone.





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Figure 14. Effect of temperature on stripping rate of H3C.

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