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TESTING OF ISOLATION BARRIER SEALING SURFACES

ABSTRACT

Isolation barrier doors are to be installed in the 105KE and 105KW basins as part of the 1994 unreviewed safety question (USQ) resolution plan to isolate the fuel storage basin from the fuel discharge chute. Included in this installation is the placement of new sealing surfaces for the barriers by affixing stainless steel plates to existing carbon steel angle bars with a specially formulated epoxy adhesive/sealant material. The sealant is a two-part component consisting of an epoxy resin (the condensation product of bisphenol A and epichlorohydrin) and a curing agent (a proprietary cycloaliphatic polyamine). The sealant is solvent free (complying with air pollution regulations) and capable of withstanding the surrounding radiation fields over an estimated 15-year service life. The epoxy sealant experiences negligible water damage partly because of its hydrophobic (water-repelling) nature. With bond tensile strengths measured at greater than 862 kPa (125 lbf/in²), the epoxy sealant is judged acceptable for its intended application. The four-hour pot life of the epoxy sealant provides sufficient time to apply the epoxy, examine the epoxy bead for continuity, and position the stainless steel sealing plates.

TESTING OF ISOLATION BARRIER SEALING SURFACES

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TESTING OF ISOLATION BARRIER SEALING SURFACES

1.0 INTRODUCTION

Isolation barrier doors are to be installed in the 105KE and 105KW basins as part of the 1994 unreviewed safety question (USQ) resolution plan to isolate the fuel storage basin from the fuel discharge chute. The sealing surfaces for these doors are to be modified as follows: (1) existing inflatable seals and hardware are to be removed from the surface of the existing carbon steel angle bar embedded at each corner of the wall between the basin and the chute; (2) each angle bar is to be mechanically prepared and cleaned to remove any loose deposits (e.g., rust, paint); and (3) a new sealing surface is to be installed by fastening a U-shaped stainless steel plate (1.5-mm [0.059-in] thick) with an epoxy adhesive/sealant to the cleaned angle bar. This document details the epoxy sealant to be used and the results of its qualification tests.

The barrier doors and the sealing surfaces will be installed by Sonsub Environmental Services, Houston, Texas. The specific epoxy sealant being used is manufactured by Thin Film Technology (TFT) Inc., also located in Houston. See Appendix A for company contact listings.

2.0 SUMMARY

The sealant is a two-part component consisting of an epoxy resin (the condensation product of bisphenol A and epichlorohydrin) and a curing agent (a proprietary cycloaliphatic polyamine [see page 3]). The sealant is solvent free (complying with air pollution regulations) and capable of withstanding the surrounding radiation fields over an estimated 15-year service life. The epoxy sealant experiences negligible water damage partly because of its hydrophobic (water-repelling) nature.

Tensile and shear tests simulating the sealant joint were performed with bonding between the stainless steel and carbon steel test pieces occurring approximately 45 minutes after epoxy application. Results showed a bond tensile strength of 883 kPa (128 lbf/in²) and a bond shear strength of 1,076 kPa (156 lbf/in²). Tensile tests in which the bond time after epoxy application is varied (10 minutes, 2, 3, and 4 hours) were also performed. The 10-minute and 2-hour samples failed at the bolt weld before the epoxy bond was broken. The 3- and 4-hour samples yielded bond tensile strengths of 1,416 kPa (205 lbf/in²) and 880 kPa (128 lbf/in²), respectively. Remote epoxy application was demonstrated November 16, 1994, at the Sonsub laboratories, showing the epoxy bead to be stable. Laying down the bead of epoxy is estimated to require 10 to 20 minutes over the total length of approximately 12 m (40 ft).

3.0 BACKGROUND - EPOXY RESINS

Epoxy resins are a thermosetting synthetic product derived from the reaction of a polyepoxide resin and a basic or acidic curing agent (hardener) (Shields 1984). The predominant types of epoxy resins are formed from the reaction of bisphenol A and epichlorohydrin (ASM 1990). This reaction of epichlorohydrin (in excess) with bisphenol A, in the presence of sodium hydroxide, is shown in Figure 1. The product is a crude diglycidyl ether of bisphenol A (DGEBA) possessing a viscosity of 11 to 15 Pa·s (110 to 150 P) at 25 °C (77 °F) (ASM 1990).

Figure 1. Diglycidyl Ether of Bisphenol A (ASM 1990).

Epoxy resins possess a unique combination of properties (Lee and Neville 1967)

- Low viscosity: The liquid resins and their curing agents form low viscosity easy-to-process (or modify) systems.
- Easy cure: Epoxy resins cure quickly and easily at practically any temperature from 5 to 150 °C (40 to 300 °F), depending on the selection of the curing agent.
- Low shrinkage: Phenolic casting resins that evolve water reveal high shrinkage, as do the acrylic and polyester resins that must rearrange and reorient considerably in the liquid and semigelled phases. Epoxy resins cure with very little rearrangement and with no volatile by-products.
- High adhesive strengths: Because of the chemical makeup, chiefly the presence of polar hydroxyl and ether groups, the epoxy resins are excellent adhesives. The resins cure with low shrinkage, so that the various surface contacts set up between the liquid epoxy-resin formulation and the adherents are not disturbed during cure.

- High mechanical properties: The strength of properly formulated epoxy resins usually surpasses that of other types of casting resins, probably in part because of their low shrinkage, which minimizes stresses that otherwise would weaken the mechanical structure.
- High electrical insulation: Epoxy resins are excellent electrical insulators.
- Good chemical resistance: The chemical resistance of the cured epoxy resin depends considerably on the curing agent used. Overall, most epoxy resins possess extremely high resistance to caustics and good to excellent resistance to acids.
- Versatility: The basic properties of epoxy resins may be modified in many ways: by blending of resin types, by selection of curing agents, and by the use of modifiers and fillers.

Because of the versatility of epoxy resins, they are used in thousands of industrial applications (Lee and Neville 1967), including:

- Adhesives for aircraft honeycomb structures
- Body solders and caulking compounds for the repair of plastic and metal boats, automobiles, etc.
- Casting compounds for the fabrication of short-run and prototype molds, stamping dies, patterns, and tooling
- Caulking and sealant compounds in building and highway construction
- Epoxy-based solution coatings for structural steel, tanks, marine applications, and as primers and varnishes
- Dental, surgical, and prosthetic applications.

Epoxies require a curing agent (hardener) if useful properties are to be obtained. The curing agents employed in the epoxy resin technology are basic (Lewis bases, inorganic bases, primary and secondary amines, and amides) or acidic (Lewis acids, organic acids, and phenols) (Lee and Neville 1967). As an amine curing agent is used in the epoxy sealant, the other curing agents will not be discussed here. Primary and secondary amines add to the epoxy ring, forming a tertiary amine, which then reacts with present hydroxyl groups to harden the epoxy resin. Aliphatic¹ amines cure epoxies at room temperatures, whereas aromatic amines require elevated temperatures. Aliphatic amines are effective in providing flexibility and cured epoxies have excellent mechanical, physical, and chemical properties (Shields 1984).

¹ Aliphatic: saturated hydrocarbon having no carbon-carbon double bonds.
Cycloaliphatic: aliphatic compound with a cyclic or ring structure.
Aromatic: unsaturated hydrocarbon with one or more benzene ring structures.

4.0 ISOLATION BARRIER SEALANT

The epoxy sealant used in attaching the stainless steel sealing surfaces is specially formulated by TFT for underwater application with the robotic extrusion equipment manufactured by Sonsub (see Appendix B for TFT product data sheets). The formulation is based on TFT's "BIO-DUR 561,"² a product manufactured as an underwater coating to steel and concrete surfaces and applied by divers using manual spreading techniques and straight-edged plastic or steel applicators. The material safety data sheets (MSDS) for the epoxy resin and curing agent appear in Appendix C.

4.1 PHYSICAL PROPERTIES

The epoxy resin used in BIO-DUR 561 is the condensation product of bisphenol A and epichlorohydrin (see Figure 1 for schematic). The resin is cured at a stoichiometric ratio with a proprietary cycloaliphatic polyamine (see Table 1 for ratios) with benzyl alcohol added as a cure accelerator (see MSDS in Appendix C). BIO-DUR 561 had satisfactory adhesive and mechanical properties; however, the Kevlar³ fiber reinforcement employed in the formulation gave a "cohesive" quality that made the freshly applied adhesive drag behind Sonsub's extrusion applicator. To eliminate this defect, TFT formulated a sealant with similar resinous ingredients but with a different thixotrope (thickener) to reduce the cohesive effect. The thixotrope used is magnesium silicate and is included in both the resin base and the curing agent.

Because the new formulation is solvent-free, it eliminates the odor problems and explosion hazards of epoxy solvents (however, it forms CO and CO₂ decomposition products). The absence of these solvents assures compliance with all current and proposed air pollution regulations. The absence of solvents also prevents solvent entrainment and subsequent shrinkage problems that otherwise would occur after application. Table 1 lists the physical properties of the sealant.

The pigmentations used for the epoxy base and curing agent are inert compounds. A synthetic red iron oxide is used for the epoxy base with white titanium dioxide in the curing agent; these yield a dark pink color when mixed. This coloring allows mixers to see at a glance whether "hotspots" of unmixed epoxy base or curing agent remain.

Before curing, the epoxy is fluid (similar to pancake batter) and sticky. It provides good fill for uneven surfaces, such as holes, scratches, or dings. With the current bead size, a surface irregularity up to 9.5 mm (0.375 in) in diameter can be bridged without affecting the epoxy's mechanical and sealant properties. The epoxy is reported to be viscous enough so as to not flow or run down the vertical surface of the carbon steel angle bar.

² BIO-DUR 561 is a registered trademark of Thin Film Technology, Inc.

³ Kevlar is a registered trademark of E. I. duPont de Nemours Company.

Table 1. Epoxy Sealant Physical Properties.

Mixing Ratio	Base/Curing agent = 3/2 by volume 1.58/1.0 by weight
Pot life (working time)	≈4 hours in 10 °C (50 °F) water*
Flash point	Over 93 °C (200 °F)
Dry time, hard	12 hours in 10 °C (50 °F) water*
Shelf life	24 months minimum (normal conditions, and in original sealed container)
Storage conditions	Normal (Covered, room temperature or refrigerated storage)
Volatile organic content (VOC)	Zero

* As measured by Sonsub

4.2 RADIATION RESISTANCE

Epoxyes are stable to a total absorbed dose of about 5×10^9 rad (Clough et al. 1991). This is the dosage required to reduce tensile elongation to 50% of the material's unirradiated value. This value was achieved at a high-dose rate (10^6 - 10^7 rad/hr) that minimizes oxygen interaction with the free radicals produced during irradiation.

However, in low-dose-rate conditions, the interaction of oxygen with the testing material produces a greater amount of damage because of the much longer times required to accumulate a specific radiation dose. Data listed for other materials in which high- and low-dose rate data were compared showed a decrease of a factor of about 50 in the total absorbed dose necessary to produce the same effect at low dose rates (Clough et al. 1991). The epoxy will be immersed in air-saturated water at a low dose rate; therefore the total absorbed dose necessary to reach the epoxy damage threshold (as defined by a 50% reduction of ductility) will be a factor of 50 less: 1×10^8 rad.

A radiological profile of the 105K East Basin discharge chute (Millsap 1994) shows a maximum dose rate of 200 rad/hr in July 1993 (radiation values in the K West Basin discharge chute are lower). The barriers are to be in place for up to 15 years; therefore the maximum dose absorbed by the sealant will be

$$200 \text{ rad/hr} \times 8760 \text{ hr/yr} \times 15 \text{ yr} = 2.63 \times 10^7 \text{ rad.}$$

This value is about 25% of the total absorbed dosage required to produce material instability as given by Clough et al. (1991) for a low dose rate. However, at most points inside the discharge chute access passageways, the dose rate is ≤ 40 rad/hr. Thus the maximum absorbed dose would be reduced to a value of 5.26×10^6 rad, or 5% of the total absorbed dosage required to produce material instability. An additional margin of safety also exists

because the barrier door, 12.7-mm (0.5-in) thick steel, and the sealing plate will provide some shielding that would further reduce the radiation field seen by the epoxy.

4.3 MOISTURE RESISTANCE

When adhesive joints are exposed to moisture, water molecules will migrate and be adsorbed preferentially into the interface region. This phenomenon occurs because joint substrates, such as metal or metal oxides, have very high surface energies, and water permeates all organic adhesives (ASM 1990). Many researchers have found that, in a typical structural joint, such as epoxy-metal, water generally enters a joint system by diffusion through the epoxy rather than by passage along the interface (ASM 1990).

In epoxy adhesives, absorbed water acts as a plasticizer, substantially reducing the tensile modulus and lowering the glass transition temperature (T_g)⁴ (ASM 1990). However, the epoxy in question is a hydrophobic (water-repelling) mixture, as shown by TFT test plots in Figures 2 and 3 (for actual data, see Appendix B). The data are for the base epoxy material immersed in room-temperature deionized water; the thickness of the epoxy film was ≈ 0.25 mm (0.01 in) with a 7-day cure time. Figure 2 shows that the water absorption levels off and the epoxy equilibrates at a weight change $\leq +1.5\%$. The effect of the uptake of this quantity of water is negligible on the epoxy's strength and sealant capabilities. This conclusion is supported by the plot in Figure 3 showing the minimal effect of water exposure on the epoxy's hardness.

Figure 2. Effects of Deionized Water on Epoxy Weight.

⁴ T_g : The approximate midpoint of the temperature range over which a polymer changes (reversibly) from, or to, a viscous or rubbery condition to, or from, a hard and relatively brittle one.

Figure 3. Effects of Deionized Water on Epoxy Hardness.

4.4 SURFACE PREPARATION

The strength of a bonded joint depends not only on the cohesive strength of the epoxy but also on the degree of adhesion to the bonding surfaces. For optimum adhesion, the surfaces must be cleaned or converted to a suitable condition before bonding by either mechanical or chemical methods. For the K basins application, cleaning the existing carbon steel angle bar by chemical treatments was not a viable option. Instead, the angle bars will be wire-brushed to remove any loose scaly material such as paint or rust deposits. The brushing will also produce a rough surface profile that the epoxy can form a mechanical interlocking bond with. The stainless steel sealing plate is to be grit-blasted (on the side to be bonded only) for the same reason. Once prepared, the treated surfaces should be bonded as soon as possible to prevent rust buildup and other surface contamination.

5.0 EPOXY SEALANT TESTING

Sonsub performed three tests on the epoxy sealant to determine its mechanical properties and approximate application time; H.O. MOHR Research & Engineering, Inc., of Houston, Texas, measured mechanical property values (see Appendix A):

- Joining the carbon and stainless steel pieces early in the sealant pot life (approximately 45 minutes after application); then measuring tensile and shear failure loads

- Joining the carbon and stainless steel pieces late in the sealant pot life (10 minutes, 2, 3, and 4 hours after application); then measuring the tensile failure loads to determine a time dependency, if any
- Applying a long line of sealant (approximately 1.8-m [6-ft] long) to determine an approximate travel speed for the extrusion applicator nozzle and to measure bead stability.

5.1 TENSILE & SHEAR TESTING - EARLY IN POT LIFE

5.1.1 Procedure

Rusted carbon steel test pieces were cut so that a vertical test surface of 10 by 10 cm (4 by 4 in.) was available for bonding. The test pieces then were wire brushed to remove deposits and rinsed clean of contaminants. The stainless steel test pieces were 7.6 by 7.6 cm (3 by 3 in.), grit blasted (side to be bonded only), and rinsed of contaminants. A bolt was welded to the center back of the stainless steel test piece for use in measuring failure loads (see Figure 2). Both pieces then were submerged in 10 °C (50 °F) water for the remainder of the procedure.

After 45 minutes underwater, the sealant was applied by extruding a narrow mound approximately 6.4-mm (0.25-in) wide and 3.2-mm (0.125-in) high down the center of the carbon steel test piece.⁵ This mound size, on the application of pressure, would produce the required 7.6-cm (3-in) -wide, 0.18-mm (7-mil) -thick sealant area. Forty-five minutes after the epoxy sealant was applied, the stainless steel test pieces were attached to the carbon steel test pieces with an initial platen pressure of 29.6 kPa (4.3 lbf/in²) and a 12-hour cure time.

The test pieces then were taken to H. O. MOHR Research & Engineering, Inc., for measuring of the tensile and shear failure loads (approximately one week after bonding). Figures 4 and 5 are schematics showing the location and direction of the applied loads for the tension and shear tests, respectively.

The 8,900-N (2,000-lbf) load cell system used by H.O. MOHR for the testing was calibrated on February 26, 1994, according to the current ASTM (American Society for Testing and Materials) Standard E-4 (ASTM 1989). The maximum error observed was 0.25% in the loading range of 890 to 8,900 N (200 to 2,000 lbf).

⁵ This amount is equivalent to a round bead approximately 4.8 mm (0.1875 in) in diameter with a cross-sectional area of approximately 16 mm² (0.025 in²).

Figure 4. Schematic of Sealant Tensile Test.

Figure 5. Schematic of Sealant Shear Test.

5.1.2 Results

The results of the tensile and shear tests are shown in Table 2. In each of the samples the epoxy formed an area approximately 76 by 25 by 3 mm (3 by 1 by 0.125 in) down the center of the test blocks. The breakout area was that area of the epoxy sealant that remained on the stainless steel test piece after it was separated from the carbon steel test piece.

Table 2. Early Pot Life Tensile and Shear Test Results, 45 Minute Bond Time. (12 hour platen pressure)

No.	Cure Method	Failure Mode	Total Area cm ² (in ²)	Breakout Area cm ² (in ²)	Failure Load N (lbf)	Bond Strength* kPa (lbf/in ²)
1	Water	Tension	19.8 (3.063)	19.8 (3.063)	1659 (373)	880 (128)
2	Air	Shear	≈27.4 (≈4.25)	20.8 (3.219)	4030 (906)	1469 (213)
3	Water	Shear	≈32.3 (≈5.0)	24.1 (3.734)	3461 (778)	1076 (156)

* Bond Strength = (Failure Load/Total Area)

The fracture surfaces of the shear and tension samples (water-cured) were examined. On the tension test sample, all of the epoxy remained attached to the stainless steel. On the shear test sample, approximately three-quarters of the epoxy remained attached to the stainless steel, leaving approximately one-quarter of the epoxy on the carbon steel. The epoxy fracture was brittle with the appearance of river markings, similar to those that appear in the brittle (cleavage) fracture of metals. Examination of the epoxy cross section showed an even distribution of voids (air pockets) within the epoxy having a diameter of less than 1.6 mm (0.0625 in).

5.2 TENSILE TESTING - LATE IN POT LIFE

5.2.1 Procedure

This test is similar to that described in Section 5.1, except that the timing of the bonding of the stainless steel and carbon steel test pieces varied. The pieces were attached at times of 10 minutes, and 2, 3, and 4 hours after the epoxy sealant was applied to the carbon steel. The epoxy was applied with a syringe, giving a bead approximately 12.8-mm (0.5-in) wide and 6.4-mm (0.25-in) high. The stainless steel test pieces were increased in thickness to approximately 6.4 mm (0.25 in) so the weight of the pieces could be used to simulate the joining pressure. The samples then were water cured (temperature: 10 °C [50 °F]) for 48 hours before tensile testing.

These test samples also were taken to H. O. MOHR Research & Engineering, Inc., for measuring of the tensile failure loads. The location and direction of the applied load for the tension test was identical to that in the test detailed in Section 4.1 (Figure 4).

5.2.2 Results

Mixing of the epoxy yielded a consistency resembling pancake batter (not pancake syrup, as previously reported). After 4 hours in the 10 °C (50 °F) water, a sample of epoxy was removed to determine its tackiness. A small sample, pulled between the thumb and finger, showed the epoxy to remain very tacky and spreadable. Separating the thumb and finger to break the epoxy bond, individually wetting each, then resuming the pulling, resulted in no differences in tack (even after the epoxy was wiped off, the thumb and finger remained sticky).

After curing for 24 hours, the epoxy samples were examined. The height of the epoxy bond was ≤ 0.4 mm (0.015 in). The epoxy appeared to have spread uniformly between the steel plates. A fingernail pushed into excess epoxy on the carbon steel (not covered by stainless steel) made only a minor indentation (comparable to that made in old, hard chewing gum).

The results of the tensile tests appear in Table 3. In the 10-minute and in the 2-hour test samples (samples #6 and #5, respectively), the weld attaching the bolt to the stainless steel plate failed before the epoxy bond did (see Figure 6). At approximately 7½ hours after the epoxy application, it was discovered that the sample #4 stainless steel plate had slid off the carbon steel and epoxy surface. The sample again was placed on the still soft epoxy and pressed down to reattach it.

Table 3. Late Pot Life Tensile Test Results, Various Bond Times.
(12 hour platen pressure)

Sample # & Time to Bond	Total Area cm ² (in ²)	Area 1† cm ² (in ²)	Area 2‡ cm ² (in ²)	Failure Load N (lbf)	Bond Strength* kPa (lbf/in ²)
6 (10 min)	N/A	N/A	N/A	5080 (1142)	1124 minimum (163)**
5 (2 hr)	N/A	N/A	N/A	5382 (1210)	1207 minimum (173)**
7 (3 hr)	43.4 (6.719)	24.1 (3.743)	19.2 (2.976)	6139 (1380)	1416 (205)
4 (4 hr)	39.5 (6.125)	37.2 (5.766)	2.3 (0.359)	3479 (782)	880 (128)

† Area of epoxy remaining on stainless steel

‡ Area of epoxy remaining on carbon steel

* Bond Strength = (Failure load/Total Area)

** Bolt failed before epoxy bond. Bond strength calculated assuming that total area of epoxy was approximately 45 cm² (7 in²).

Figure 7 shows the fracture surfaces of samples #4 and #7 (the upper angle bar is carbon steel; the two smaller plates are stainless steel). For sample #4, with a 4-hour bond time, 94% of the epoxy remained attached to the stainless steel plate. For sample #7, with a 3-hour bond time, only 56% of the epoxy remained attached to the stainless steel plate.

5.3 TEST OF NOZZLE TRAVEL SPEED

The purpose of this test was to determine an approximate travel speed for the extrusion applicator. As nozzle speed will need to be adjusted manually by the operator to maintain a uniform bead size, this value is only an approximation. This speed can be used to estimate a sealant application time for each door opening.

5.3.1 Procedure

In Sonsub's 4.9 m (16 ft) deep mockup tank, a uniform sealant mound of the required size (6.4 mm [0.25 in] wide and 3.2 mm [0.125 in] high, minimum) was remotely applied over a length of 1.8 m (6 feet) to measure the speed of application (WHC had requested that a sample corner and a joining of two beads also be done). The sealant was applied with Sonsub's remote applicator nozzle developed for field application in the discharge chutes. A video recording of the test was made to document the results.

5.3.2 Results

During the demonstration of remote epoxy application, the operator's ability to lay a continuous bead of epoxy, traverse a 90° corner, and reposition the applicator to fill a gap in the epoxy bead was observed. The operator was sufficiently skilled in maneuvering the applicator tool to lay a corner bead and fix any gaps in the epoxy efficiently. This same operator will be performing the field epoxy application. The demonstration suggests that there will not be a problem in applying enough epoxy to the carbon steel. If anything, too much epoxy might be applied; it then could seep from the carbon steel-stainless steel joint edges when the cure pressure was applied. The plastic applicator head lays a bead approximately 19 mm (0.75 in) wide and 9.5 to 12.7 mm (0.375 to 0.5 in) high in the center and 6.4 mm (0.25 in) high at the edges.

Minor mechanical difficulties with the video camera system, made it impossible to estimate travel speed. The original estimate given by Sonsub of 10 to 20 minutes to lay the epoxy bead over a distance of approximately 12 m (40 ft) appears valid. This estimate was based on Sonsub trial runs using a substituted material similar in consistency to the epoxy sealant.

Although the epoxy had the consistency of pancake batter in an air environment, it appeared somewhat thicker in the underwater application. This was due to the temperature of the water (approximately 10 °C [50 °F]), which increases the epoxy's viscosity. The epoxy bead appeared stable (resistant to running) except when significant excess (much greater than the application size) epoxy was present.

Figure 6 Epoxy Bonded Plate Samples #5 (2 Hours) and #6 (10 Minutes).

Figure 7 Epoxy Bonded Plate Samples #4 (4 Hours) and #7 (3 Hours).

The demonstration indicates that the Sonsub apparatus will be capable of traversing the whole length of the door frame in one pass. It was thought that the epoxy applicator would be able to do only one side and one-half of the bottom, then would need to be repositioned to complete the application.

6.0 CONCLUSIONS

The epoxy manufactured by Thin Film Technology is solvent free, capable of withstanding the basin/discharge chute radiation levels (≤ 200 rad/hr), and is water resistant. Samples that replicated the epoxy sealant's water environment and adherends were tested for bond strength. With bond tensile strengths measured at greater than 862 kPa (125 lbf/in²) (see Tables 2 and 3), the epoxy sealant is judged acceptable for its intended application. This judgement is based on three major considerations:

- The weight of the stainless steel sealing plate is less than 23 kg (50 lbm) (Anderson 1994), which, over the large surface area of the plate (0.95 m² [1,475 in²]), imparts only a minimal strain on the epoxy joint. Using the shear and tension values from Table 2 and the plate's surface area, it would require a force of 1,023 kN (230 kips) in shear or 840 kN (189 kips) in tension to remove the sealing plate.
- Once installed, the isolation barrier doors will be mechanically closed (using Z-brackets and tie-rods), thus holding the stainless steel sealing plate in position (until door removal). At this time the epoxy's sealant properties become more important than its strength (adhesive) properties. As long as a continuous bead (no gaps) of epoxy is applied on the carbon steel, the cure pressure will induce flow of the epoxy between the carbon and stainless steel and result in a uniform, leak-resistant joint. A bead with minimum dimensions of 19 mm (0.75 in) wide and 6.4 mm (0.25 in) high is judged to be acceptable.
- Seismic evaluations (Lindquist 1994), assuming a 0.24 g lateral seismic load, calculated a 0.33 mm (0.013 in) displacement between the vertical sealing surface on the center island divider wall and the vertical sealing surface on the adjacent basin wall. This seismic load is in addition to the inertial and hydrodynamic loads considered. Further calculations (Lindquist 1994) show that the isolation barrier doors are structurally capable of withstanding this displacement without twisting (which could cause leakage through the epoxy seal).

Bonding of the carbon and stainless steel pieces should occur as soon as possible after the carbon steel angle bar surface preparation. However, with a corrosion rate for uncoated carbon steel less than 0.05 mm/yr (2 mil/yr) (Emory 1994), the K basin water is relatively benign. Therefore a delay of up to 72 hours between surface preparation and epoxy application is acceptable.

Varying the time between applying epoxy and joining of the carbon steel and stainless steel surfaces produced strength values that were sufficiently high to indicate that a 4-hour delay in joining the sealing surfaces is acceptable. The 4-hour pot life for the epoxy sealant allows sufficient time to apply the epoxy, examine the epoxy bead for continuity, and position the stainless steel sealing plate.

7.0 REFERENCES

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APPENDIX A: Company Contacts

Sonsub Environmental Services

10905 Metronome
Houston, Texas 77043

(713) 984-9150
(713) 984-1810 FAX

Contact: Bill Bath, Engineer
Harry Tate, Engineer

Thin Film Technology, Inc.

P.O. Box 580669
Houston, Texas 77258-0669

(713) 333-9415
(713) 333-9406 FAX

Contact: Jeff Longmore, Director

H. O. MOHR Research & Engineering, Inc.

Northwoods Industrial Park West
12237 FM 529
Houston, Texas 77041-2805

(713) 466-1527
(713) 896-6807 FAX

Contact: David Gwyn, Project Manager

APPENDIX B: Sealant Data Sheets

Ms. Carolyn Graves
WESTINGHOUSE HANFORD Co.
Mailstop H5-67
Richland, WA 99352

26th October, 1994

Re: SONSUB Underwater Epoxy Adhesive - 1/022/1a & 1/022/1b

Dear Carolyn:

Enclosed with this are the Product Data and Statement of Shelf Life you requested. If there is more that we can add please give a call and we shall see what we can do. Also enclosed for your general interest is a Product Data Sheet describing our BIO-DUR 561 which is effectively the parent formulation from which the SONSUB material was derived.

As I mentioned during our conversation we manufacture a fair number of high performance materials for difficult applications. Our latest by the way is an epoxy based encapsulating and reinforcing system for application to hot (250°F) surfaces which has been hydro tested at over 1,800 psi over a 3/8" hole after an overnight cure. We still enjoy the challenge of oddball applications and look forward to hearing from you if you come up against a problem we can help with.

With thanks for your interest,

Sincerely:

Jeff Longmore

CC: Harry Tate - SONSUB

Encls: PDS561; PDS 1/022/1ab; 1/022/1ab shelf life

JL/fs

B-2

Thin Film Technology, Inc. P.O. Box 580669, Houston, TX 77258-0669
Voice: (713)333-9415; Fax: (713)333-9406; Mobile: (713)254-3714

PRELIMINARY DATA SHEET

SONSUB UNDERWATER ADHESIVE:
BASE & CURING AGENT 1/022/1a & 1/022/1b

THIN FILM TECHNOLOGY, Inc.
HIGH PERFORMANCE COATINGS

DESCRIPTION:

SONSUB Underwater adhesive 1/022/1a & 1/022/1b was specially formulated for underwater application to cleaned steel surfaces using robotic extrusion equipment manufactured by SONSUB. The formulation is based on the TFT "BIO-DUR 561" product manufactured for underwater application to steel and concrete surfaces by divers using manual spreading techniques and straight-edged plastic or steel applicators. Initial examination of the BIO-DUR 561 as a candidate adhesive for SONSUB showed that this material had satisfactory adhesive and mechanical properties however the fiber reinforcement employed in the formulations gave a "cohesive" quality which made the freshly applied adhesive drag behind the applicator. To eliminate this defect a product was formulated with similar resinous ingredients but employing a different thixotrope to lessen the cohesive effect.

The formulation is solvent-free to avoid the objectionable odor and explosion hazards of epoxy solvents. Absence of solvents also assures compliance with all present and proposed air pollution regulations and assures the absence of solvent entrainment and shrinkage problems which would otherwise occur after application.

The epoxy resin used is a condensation product of bisphenol A and epichlorhydrin. This resin is cured at stoichiometric ratio using a proprietary non-aromatic polyamine.

Pigmentation is inert and consists of synthetic red iron oxide in the epoxy base component with titanium dioxide in the curing agent to yield a dark pink upon mixing. Magnesium silicate is included in both components to assist in the development of a "short", non-cohesive thixotropic gel structure.

PHYSICAL PROPERTIES:

Vehicle Type	Bis A epoxy/Polyamines
Pigmentation	Inert
Colors	Red Oxide base, White curing Agent
Cleaner	Lacquer Thinner or similar
Mixing Ratio	Base/Curing Agent :: 3/2 (volume)
	" " " 1.58/1.00 (wt)
Pot Life	Approx 35'/77°F
Flash Point	over 200°F
Solids by Volume	100%
Dry Time, (Touch)	5 hrs/77°F
Dry Time, (Hard)	12 hrs/77°F
Application Method	SONSUB specialized equipment
Shelf Life	24 months minimum
Storage Conditions	Normal
VOC	Zero

SONSUB UNDERWATER ADHESIVE 1/022/1a & 1/022/1b

STATEMENT OF SHELF LIFE:

This product was specially formulated for a SONSUB application. The formulation was built around the resin system used in a standard TFT product designated BIO-DUR 561 which has been seen to be stable on storage in excess of 24 months.

Based on previous experience and the knowledge of the raw materials in the formulation we would state that the SONSUB underwater adhesive 1/022/1a & 1/022/1b has a minimum shelf life of 24 months when stored in normal conditions in the original sealed containers.

Note:

The epoxy resin used in this formulation is a very pure material, low viscosity product. Purified epoxy resin is a definite organic compound rather than the loose mixture of homologs and different polymer weight materials found in other resins such as acrylics, vinyls and the like. Pure epoxy resin may crystallize to a solid material after storage of several months especially when subjected to fluctuations in temperature. This crystallization shows itself as either a "granulated sugar" like material dispersed through the formulation or, in extreme cases, the entire material will solidify. If crystallization is seen it may easily be removed by gently heating the product in ordinary, hot domestic water at about 130°F for an hour or so. Once the product is brought up to temperature the crystals will dissolve and the material will remain stable for many more months. No chemical change is involved and once reconstituted the original properties are fully restored.

NOTE: PRODUCTION BATCHES MANUFACTURED 03AUG94

BIO-DUR**561**100% PROTECTION
WET OR DRY

BIO-LINE COATINGS

PRODUCT DATA SHEET

BIO-DUR 561 is based on a unique blend of liquid epoxy polymer and aliphatic polyamine curing agents which is able to displace water from wet surfaces in order to make a permanent bond. The formulation is solvent free to ensure safety and maximum technical performance. Kevlar* fibers are incorporated for reinforcement and viscosity management to achieve high application rates - even underwater!

BIO-DUR 561 provides permanent protection even under the most adverse conditions. It uses advanced low toxicity curing agents and is available in all colors including bright white.

* trademark E.I. DuPont de Nemours Co.

APPLICATION NOTES

ANTICORROSIVE COATING: Splash zone, spot repairs above and below water.

REPAIR COMPOUND: Patching, leak sealing etc. under all conditions.

FAIRING COMPOUND: Rough steel and concrete exposed to erosion.

ADHESIVE MORTAR: in dry, wet or submerged conditions.

WASTE WATER TREATMENT: Exc. adhesion to wet concrete, exc. acid resistance.

PHYSICAL PROPERTIES

VEHICLE TYPE	Epoxy/Aliphatic Polyamines
PIGMENTATION	Color/Inert/Fibrous reinforcement
COLORS	Splash Zone Green, Red Oxide, Black, White
FINISH	Semi-gloss
THINNER	Not Required
CLEANER	TFT solvent 009-00000 or standard epoxy thinner
MIXING RATIO	1.0/1.0 (Volume)
INDUCTION TIME	Not Required
POT LIFE	Approx. 40 minutes/80°F
FLASH POINT	Over 100°F (Closed Cup)
SOLIDS BY VOLUME	100%
REC. DRY THICKNESS	No Limits
THEO. SPREAD RATE	80 sq.ft./Gal. @ 20 mils; 53 sq.ft./Gal. @ 30 mils
DRY TIME (TOUCH)	Approx 4 Hrs/80°F
DRY TIME (HARD)	Approx 14Hrs/80°F
APPLICATION METHOD ...	Spatula, straight-edged spreader, mitts etc.
SHELF LIFE	12 Months minimum
STORAGE CONDITIONS ...	Normal, Freezing OK
VOC	Essentially zero

APPLICATION NOTES

SURFACE PREPARATION: Remove marine biological settlement and corrosion by high pressure water jetting with or without abrasive or by air/abrasive blasting at shallow depths. Hand held power tools such as grinders or needle guns will give good results if applied conscientiously in small areas but will be inadequate in large areas. Plan to apply the BIO-DUR 561 within 30 - 45 minutes of surface preparation to minimize rerusting or initial settlement of fouling which can interfere with initial adhesion.

MIXING PROCEDURE: BIO-DUR 561 is supplied in two gallon kits of one gallon each of epoxy and curing agent. Whenever possible these components are made in contrasting colors for example yellow base plus black curing agent to yield an olive green mixture. This system allows the mixers to see at a glance if "hotspots" of unmixed epoxy base or curing agent are still present. Remove equal quantities of base and curing agent from their cans and place them side by side on a flat sheet of plastic, fiberboard etc. Mixing is easily accomplished by folding the components into each other using a spatula or piece of wood. Once mixing begins there will be about 40 minutes of working time available at 80°F. This time can be extended by keeping the components and mixture cool, send mixed material underwater as soon as possible rather than leaving it on a hot deck.

APPLICATION: Take the mixed BIO-DUR 561 underwater in a can or bucket, it will be found helpful to have some method of holding the container such as a hook or magnet on the structure especially if visibility is poor and a lantern must also be carried. Applicators such as broad putty knives or plastic straight edged glue spreaders work well on most surfaces. Painters mitts work well on small pipes when used with a smearing motion.

Dip the applicator or gloves into the mixed BIO-DUR 561 and transfer to the surface. Applicators are used to spread out the coating by "buttering" to a uniform thickness. Thicknesses up to 1/4", (250 mils), are easily possible on vertical surfaces however experience has shown that 25 - 35 mils gives excellent protection on steel splash zones.

BIO-DUR 561 is different from traditional "Splash Zone" coatings in that it has a strong tendency to stick securely to underwater surfaces. Contact with divers equipment should be minimised by protecting with plastic bags, inexpensive rainsuits etc.

NOTE: Temperature has a considerable influence on the rate of hardening of chemically cured materials such as BIO-DUR 561. In broad terms expect each 10°C, (18°F), rise or fall in temperature to half or double drying times and potlife.

BIO-DUR 561 may be applied in temperatures as low as 45°F - curing will be slow however the viscosity of the material will still be workable.

WE URGE YOU TO READ THE MATERIAL SAFETY DATA SHEET (MSDS) BEFORE USING AND TO CALL THIN FILM TECHNOLOGY FOR ADVICE OR INFORMATION REGARDING APPLICATION PROCEDURES.

Thin Film Technology, Inc. • P.O. Box 580669 • Houston, TX 77258-0669
(713) 333-9415 • Fax: (713) 333-9406 • Mobile: (713) 254-3714

SAFETY: This is a hazardous material if misused. Read and understand the Material Safety Data Sheet (MSDS) before use.
WARRANTY DISCLAIMER: The technical data given herein has been compiled for your help and guidance and is based upon our experience and knowledge. However, as we have no control over the use to which this information is put, no warranty, express or implied is intended or given. We assume no responsibility whatsoever for coverage, performance or damages, including injuries resulting from use of this information or of products recommended herein.

TELEPHONE CONFERENCE MEMORANDUM

Company: Westinghouse Hanford Company

Address: P.O. Box 1970
Richland, WA 99352

☐ INCOMING

☒ OUTGOING

DATE: November 30, 1994

TIME: 12:30 pm

WITH: Jeff Longmore, Director

OF: Thin Film Technology

PHONE: (713)333-9415

Copies to:

Name

Address

W. F. Brehm *WFB*
R. J. Kuhta
D. L. Cooley

H5-67
X3-76
X3-76

Subject: Water Degradation of Epoxy Sealant

Materials Engineering

Department

C. E. Graves 12/01/94
C. E. Graves

376-5545

Telephone #

Summary of Conference

On November 30, 1994, I telephoned Jeff Longmore of Thin Film Technology for the purpose of obtaining additional information on the [isolation barrier door] epoxy sealant's resistance to water damage. The following test data was given for the base epoxy used in our formulation. Mr. Longmore stated that the water absorption levels off and the epoxy equilibrates at a weight change of $\leq +1.5\%$ producing negligible material degradation. He also stated that the epoxy mixture is hydrophobic (water repelling) and that similar mixtures have been used in underwater structural applications for over 15 years with satisfactory results.

Chemical Exposure Test Data for Epoxy Sealant

Time	% Weight Change	Hardness (Shore D)
3 hours	+ 0.14%	78
24 hours	+ 0.35%	78
3 days	+ 0.63%	78
7 days	+ 1.00%	78
30 days	+ 1.46%	77
90 days	+ 1.31%	77

Test Conditions: Deionized Water (Room Temperature)
Thin Film of epoxy (≈ 10 mils)
7 day cure time for epoxy

APPENDIX C: Material Safety Data Sheets

READ THIS MATERIAL SAFETY DATA SHEET BEFORE USING
THIS PRODUCT. ASK YOUR SUPERVISOR FOR ASSISTANCE
IF YOU DO NOT UNDERSTAND ANY PART OF IT.

THIN FILM TECHNOLOGY, Inc.
P.O.Box 580669 HOUSTON, TX 77258-0669
Tel: (713)333-9415 Fax: (713)333-9406

THIN FILM TECHNOLOGY, Inc.

MATERIAL SAFETY DATA SHEET

PRODUCT INFORMATION

Product Name.....: SONSUB Epoxy Base - Red Oxide Chemical Name.....: N/A
CAS Number.....: N/A
Product Code.....: lab ref 1/021/2a Chemical Family...: Epoxy coating component

2 HAZARDOUS INGREDIENTS

Material	CAS Number	Contents %w/w	Hazard Data
Epoxy Resin liquid polymer	025085-99-8	55 - 85%	PSL: Not Established TLV: Not Established

***** DOES NOT CONTAIN CARCINOGENS *****

3 PHYSICAL DATA

Appearance: Red Oxide Paste Vapor Pressure: N/A Vapor Density: N/A
Solubility in water: Negligible Odor: Sl. sweet characteristic epoxy resin.
Specific Gravity: 1.20 Density at 20°C: 10.0lbs/gallon Viscosity: Heavy paste
Boiling Point: N/A Freezing Point: N/A % volatiles by volume: 0.5% max

4 FIRE AND EXPLOSION DATA

Flash Point: >250°F TCC Autoignition Temp: N/A Flam limits in air, % v/v: LEL : N/A; UEL : N/A
Extinguishing Media: Water, Spray, Foam, Dry Chemical. Fire or Explosion
Hazards: generation of toxic products. Special firefighting procedures: wear full protective equipment including self-contained breathing apparatus.

5 REACTIVITY DATA

Stability: Stable Hazardous Polymerization: Will not occur Incompatible materials: none known
Conditions to avoid: Mixing large volumes with epoxy curing agents such as BIO-DUR lab ref 1/021/2b. Volumes of one gallon are safe however expect a significant exotherm within 30 - 40 minutes at 70°F. Hazardous decomposition products: CO, CO2, unspecified others.

MSDS, SONSUB Epoxy Base - Red Oxide, LAB REF 1/021/2a -2-

6 HEALTH HAZARD DATA

Skin Irritation - Sl. irrit., possible sensitizer Inhalation Tox - Unknown, see below.
DOT Corrosivity - Not corrosive Eye Irritation - Slight irritant.
Oral Toxicity - Unknown Dermal Toxicity - Unknown

SKIN AND EYE CONTACT: Sl. irritating, possible sensitizer. INGESTION: Considered slightly toxic.

INHALATION: Overexposure to mist or vapor is extremely unlikely however inhalation of spray mist might cause irritation of respiratory tract. Prolonged or repeated exposure may cause an asthmatic reaction.

7 FIRST AID RECOMMENDATIONS

SKIN AND EYE CONTACT: Immediately flush eyes with fresh water holding lids apart for 15 minutes, washing within one minute is essential to achieve maximum effectiveness. Remove from skin using soap and water. Remove contaminated shoes and clothing. Call a physician.

INGESTION: DO NOT induce vomiting! Prevent aspiration (breathing) liquid into lungs. Get medical attention. INHALATION: Immediately move to fresh air. If breathing is difficult give oxygen. Call a physician.

8 SPILL PROCEDURES & WASTE DISPOSAL

Spill and Leak Procedures: Scrape or wipe up spilled material. Place in container for recovery or disposal. Avoid contact with skin and eyes - wear recommended protective clothing.

9 PERSONAL PROTECTION

Ventilation Requirements: Mechanical local exhaust at point of contaminant release if conditions warrant.

Respiratory protection: It is extremely unlikely that harmful concentrations of volatile materials will be released during normal applications by spreaders, trowels or similar tools in open areas. Wear organic vapor cartridge respirator or fresh air hood if working for extended periods in enclosed spaces with minimal ventilation.

Eyes: Chemical resistant goggles if eye contact possible.

Skin - Impervious gloves, wear long sleeved shirts, coveralls as required to prevent skin contact. Discard contaminated gloves, footwear etc.

10 SPECIAL PRECAUTIONS & STORAGE

Precautionary Statements: Avoid contact with eyes, skin and clothes. Wash after handling and before eating, drinking or smoking.

Store in a cool, dry area in closed containers.

Date of Prep: 27JUL94

Preparer: J.Longmore

Refs: SUZ:cti nvSRS

READ THIS MATERIAL SAFETY DATA SHEET BEFORE USING
THIS PRODUCT. ASK YOUR SUPERVISOR FOR ASSISTANCE
IF YOU DO NOT UNDERSTAND ANY PART OF IT.

TRIM FILM TECHNOLOGY, INC.
P.O. Box 580669 HOUSTON, TX 77258-0669
Tel: (713)333-9415 Fax: (713)333-9406

MATERIAL SAFETY DATA SHEET

1. PRODUCT INFORMATION

Product Name.....: SONSUB Curing Agent - White Chemical Name.....: Polyamine solution.
CAS Number.....: N/A see below Chemical Family...: Cycloaliphatic polyamine
Product Code.....: Lab ref 1/021/2b

2. HAZARDOUS INGREDIENTS

Material	CAS Number	Contents %w/v	DOT CLASSN:	OSHA PEL	OTHER LIMITS RECOMMENDED
Benzyl Alcohol	100-51-6	12 - 24%)	Mixture: UN2735	Not Est	Not Est
Remainder Resinous are are Trade Secret		20 - 50%)	UN1719		

DATA: Alkylamines NOS: Class 8; Pack Group III

***** DOES NOT CONTAIN CARCINOGENS *****

3. PHYSICAL DATA

Appearance: White paste Vapor Pressure: 13mmHg/130°F Vapor Density: N/A Solubility in water: Very Slight
Odor: Ammonia Specific Gravity: 1.10 Density at 20°C: 9.2lbs/gallon Viscosity: light gel
Boiling Point: 222°C Freezing Point: N/A % Volatiles by volume: 0% @ 70°F

4. FIRE AND EXPLOSION DATA

Flash Point: >200°F TCC Autoignition Temp: N/A Flammable limits in air, % w/v: LEL : N/A; UEL : N/A
Extinguishing Media: Water, Spray, Foam, Dry Chemical. Fire or Explosion Hazards: Generation of toxic products.
Special firefighting procedures: wear full protective equipment including self-contained breathing apparatus.

5. REACTIVITY DATA

Stability: stable Hazardous Polymerization: Will not occur Incompatible materials: none known
Conditions to avoid: Mixing large volumes with epoxy based materials such as SIO-BUR Epoxy base. Volumes of one
gallon are safe however expect a significant exotherm within 25 - 50 minutes at 77°F
Hazardous decomposition products: CO, CO2, unspecified others.

MSDS, SONSUB Curing Agent - White, lab ref 1/021/2b

-2-

6 HEALTH HAZARD DATA

INHALATION: NOTICE - DO NOT INHALE PRODUCT. MAY CAUSE IRRITATION TO UPPER RESPIRATORY TRACT UPON PROLONGED OR REPEATED INHALATION.

EYE AND SKIN CONTACT: DANGER! CAUSES SEVERE SKIN AND EYE IRRITATION OR BURNS. MAY CAUSE PERMANENT VISUAL IMPAIRMENT. WEAR PROTECTIVE CLOTHING AND GOGGLES.

CAUTION: Preexisting eye, skin and respiratory disorders may be aggravated by exposure to this product. exercise caution in handling. Remove from skin using liquid soap or detergent - always avoid using solvents to remove skin contamination.

7 FIRST AID RECOMMENDATIONS

EYE CONTACT: Immediately flush eyes with fresh water holding lids apart for 15 minutes, washing within one minute is essential to achieve maximum effectiveness.

SKIN CONTACT: Remove contaminated shoes and clothing. Avoid using solvents on skin, use liquid soap and water to remove from skin. Except in case of minor or superficial damage cover burns with sterile dressings and seek medical attention. DO NOT APPLY GREASES OR OINTMENTS. Victims of a major skin area contact should remain under medical observation for at least 24 hours due to possible delayed effects.

INGESTION: In the event of ingestion administer 3-4 glasses of milk or water. DO NOT INDUCE VOMITING. Obtain medical care and hospital treatment immediately.

INHALATION: In normal use overexposure by inhalation is extremely unlikely. If breathing has stopped or is labored give mouth-to-mouth assistance. prevent aspiration of vomit, move to fresh air. call a physician.

Note to physician: This product is highly injurious to all tissues, similar to that of ammonia. Chemical pneumonitis, pulmonary edema, laryngeal edema and delayed scarring of the airway or other affected tissue may occur following exposure. Clinical management is based on supportive treatment which is similar to that for thermal

8 SPILL PROCEDURES & WASTE DISPOSAL

Spill and Leak Procedures: Scrape or wipe up spilled material. Transfer liquid to containers for recovery or disposal. Transfer disposable material to proper containers for disposal. Keep spills and runoff out of sewers and open bodies of water. Observe all Federal, State and Local laws regarding health and the environment.

9 PERSONAL PROTECTION

RESPIRATORY PROTECTION: Not required under normal conditions. Organic chemical cartridge respirator or NIOSH/MSHA approved self contained breathing apparatus if vaporization occurs or is anticipated.

VENTILATION REQUIREMENTS: mechanical local exhaust at point of contaminant release if conditions warrant.

EYES - Chemical splash resistant goggles if eye contact possible, contact lenses should not be worn.

SKIN - Impervious gloves, wear long sleeved shirts, coveralls as required to prevent skin contact. Discard contaminated gloves, footwear etc.

10 SIGNS AND SYMPTOMS OF EXPOSURE

Possible Longer Term Effects: Repeated and/or prolonged contact with the skin may cause primary skin irritation and dermatitis. Adverse respiratory effects such as coughing, tightness of the chest or shortness of breath may also occur.

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