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SUPERSEDING
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MILITARY SPECIFICATION

CORROSION REMOVING COMPOUND, SODIUM HYDROXIDE BASE; FOR ELECTROLYTIC OR IMMERSION APPLICATION

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers corrosion removing compound, containing sodium hydroxide plus a water-soluble chelate compound or a sequestrant compound, and with or without alkali metal cyanide. Solutions of these compounds are suitable for rust removal, either by simple immersion of the articles or by electrolytic action, without causing material change in the dimensional characteristics of the treated articles (see 6.1).

1.2 Classification. Corrosion removing compounds covered by this specification shall be of the following types, as specified (see 6.2):

Type I - Sodium hydroxide plus chelate compounds or sequestrant compounds (see 6.3).

Type II - Sodium hydroxide-sodium cyanide mixture plus a chelate compound or a sequestrant compound.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications, standards, and handbooks. Unless otherwise specified, the following specifications, standards, and handbooks of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

FEDERAL

PPP-D-729 - Drums; Metal, 55-gallon (for Shipment of Noncorrosive Materials)

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Director, US Army Materials and Mechanics Research Center, ATTN: DRXMR-SSS, Watertown, MA 02172 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by
Attributes

MIL-STD-129 - Marking for Shipment and Storage

(Copies of specifications, standards, handbooks, drawings, and publications required by manufacturers in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.1.2 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS

3.1 Physical form. The raw materials used in the manufacture of these compounds shall be technical grade. They shall be intimately assembled and processed to produce a dry, fine, granular or dry, fine, flake compound. The compound shall remain uniform in composition and shall show no evidence of segregation or caking during handling and storage (see 4.4.1).

3.2 Composition. The corrosion-removing compounds covered by this specification shall conform to the requirements of table I when tested as specified in section 4.

Table I. Composition.

Ingredient	Requirement (percent by weight)		Test Paragraph
	Type I	Type II	
Sodium hydroxide	54.0 max	35.0 min	4.5.4
1/2 Trisodium salt of N-hydroxyethyl- ethylenediaminetriacetic acid (3Na ⁺ EDTAOH)	13.0 min	---	4.5.7
Chelate or sequestrant compound (EDTA tetrasodium salt dihydrate)	---	25 to 35	4.5.9
Sodium gluconate	25.0 min	---	4.5.6
Sodium cyanide	---	25 to 35	4.5.8
Phosphorus and silica, total as PO ₄ plus SiO ₂	0.25 max	0.25 max	4.5.5
Others, including foamers	7.0 max	4.0 max	---

1/2 N-hydroxyethylethylenediaminetriacetic acid may be used instead of the trisodium salt if the amount of acid when calculated as the trisodium salt meets the requirements of this specification.

3.3 Solubility (turbidity). The corrosion removing compound shall be completely soluble in water when tested as specified in 4.5.2.

3.4 Foaming characteristics. When tested as in 4.5.3, the corrosion removing compound shall show a foam head of between 1/8 and 1/4 inch.

3.5 Etching characteristics (type II only). When tested as in 4.5.10, the steel plates shall show no evidence of etching.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

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4.2 Lot. A lot of corrosion removing compound shall consist of that quantity produced by one manufacturer, in not more than 24 consecutive hours of continuous production, with no change in facilities, materials or process which would change the chemical or physical properties of the compound. In the event the process is a batch operation, each batch shall constitute a lot.

4.3 Sampling. A random sample of filled containers shall be selected in accordance with MIL-STD-105 at inspection level S-4 and acceptable quality level (AQL) equal to 1.0 percent defective to verify compliance with all requirements of this specification regarding physical form, fill, container, closure and marking. Samples shall be taken from each of the filled containers and the samples composited. Two one-gallon samples of this composite shall then be packaged in clean, dry, metal containers and sealed and marked for identification.

4.4 Inspection.

4.4.1 Visual examination. Each sample-filled container, as specified in 4.3 shall be examined for defects of the container and the closure, for evidence of leakage and for unsatisfactory markings. The compound in each sample container shall be examined for uniformity, segregation and caking to determine compliance with 3.1. Any container in the sample having one or more defects, or under required fill, shall be rejected, and if the number of defective containers in any sample exceeds the acceptance number of the appropriate sampling plan of MIL-STD-105, the lot represented by the sample shall be rejected.

4.4.2 Lot acceptance tests. The composited samples shall be subjected to the tests specified in 4.5. Failure of these samples to comply with any of the tests specified herein, shall reject the lot or batch. Rejected lots shall not be resubmitted.

4.5 Test procedures.

4.5.1 Preparation of sample. Each composited sample shall be prepared for testing as specified in 4.5.1.1 or 4.5.1.2.

4.5.1.1 Type I compound. Carefully weigh 660 g of the type I compound in a 1000 mL beaker. Add, while stirring, enough distilled water to have approximately 800 mL of solution. Heat the mixture to near boiling, and stir the solution to completely dissolve all of the compound. Cool the solution to room temperature. Transfer the solution to a 1000 mL volumetric flask. Wash the beaker several times with distilled water and transfer the washings to the flask. Fill the flask to the mark with distilled water and mix well.

4.5.1.2 Type II compound. Carefully weigh 360 g of the type II compound into a 1000 mL beaker. Add to the beaker, while stirring, approximately 500 mL of distilled water. When completely dissolved, transfer the solution to a 1000 mL volumetric flask. Wash the beaker several times with distilled water and transfer the washings to the flask. Fill the flask to the mark with distilled water and mix well.

4.5.2 Solubility (turbidity).

4.5.2.1 Type I. Examine the sample solution prepared in 4.5.1.1 for turbidity and for conformance with 3.3.

4.5.2.2 Type II. Heat 500 mL of the sample solution from 4.5.1.2 to $120^{\circ}\text{F} \pm 10$ ($49^{\circ}\text{C} \pm 5.6$) and examine for turbidity.

4.5.3 Foaming characteristics.

4.5.3.1 Preparation of steel panels. The panels, each measuring 1 X 4 X 1/8 inches (25 X 100 X 3.175 mm), shall be SAE 1020 cold-rolled steel. A 3/16-inch (4.8 mm) hole shall be drilled in one corner of each panel for attaching power leads by means of a steel bolt and nut. The surfaces of the panels shall be abraded to a surface roughness not greater than 32 microinch (0.8 μm).

4.5.3.2 Test procedure. Place 500 mL of the sample solution (see 4.5.1.1 and 4.5.1.2) into a 600 mL standard form beaker. Place the two panels (see 4.5.3.1) on opposite sides in the beaker, immersing them to a depth of 2 inches (50 mm). Attach a suitable direct current source, making one panel anodic (positive) and the other cathodic (negative). (A 6-volt storage battery may be used as the supply source.) Apply a current of 4 amperes and hold steady for 30 minutes at a temperature between 80°F (27°C) and 130°F (54°C) (by means of a water bath, if necessary). After the current has been off for 2 minutes, the height of foam at the center of the beaker shall be measured. Reserve the panels for the test of 4.5.10.

4.5.4 Sodium hydroxide content.

4.5.4.1 Preparation of color blank. Immediately before performing the test for sodium hydroxide content, prepare a color blank by dissolving 1 g of sodium cyanide in 80 mL distilled water in a 125 mL Erlenmeyer flask and then adding 16 drops of LaMotte sulfo-orange indicator.

4.5.4.2 Preparation of 1N hydrochloric acid solution. A 1N HCL solution shall be prepared by mixing one volume of concentrated reagent grade hydrochloric acid (38 percent) with 11 volumes of distilled water. Standardize the solution with primary standard grade, anhydrous, sodium carbonate using methyl orange as an indicator.

4.5.4.3 Test procedure. Pipette 10 mL of the sample solution (see 4.5.1.1 and 4.5.1.2) into a 250 mL conical flask and add 10 mL distilled water, 1 g of sodium cyanide and 16 drops of LaMotte sulfo-orange indicator. Titrate with the standardized 1N HCL solution to the identical color of the blank.

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4.5.4.4 Calculation. For type I compound, calculate the sodium hydroxide content by the following formula:

$$\text{Percent NaOH} = 0.606 \times \text{mL of 1N HCL used.}$$

For type II compound, calculate the sodium hydroxide content by the following formula:

$$\text{Percent NAOH} = 1.11 \times \text{mL of 1N HCL used.}$$

4.5.5 Phosphorus and silica content. The combined phosphorus and silica content shall be determined by spectrographic analysis.

4.5.6 Sodium Gluconate Content (type I only).

4.5.6.1 Reagents.

4.5.6.1.1 Silylating solution. Trimethylsilylimidazole in a concentration of 1.5 milliequivalents per mL of pyridine solution. This reagent is available in ready-to-use form from various distributors of chromatographic supplies.

4.5.6.1.2 Internal standard. Accurately weigh approximately 0.5 g of L-ascorbic acid, dissolve in pyridine, and dilute with pyridine to 100 mL in a volumetric flask.

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4.5.6.2 Test procedure. Dissolve approximately 10 g, (weighed to the third decimal), of the corrosion removing compound with water and dilute to volume in a 500 mL volumetric flask with water. Pipette 1 mL of the thoroughly-mixed solution to a 2 mL vial. Add six drops of concentrated HCL, and evaporate to loss of chlorine odor at 149°F (65°C) with a gentle flow of nitrogen. Pipette 1 mL of the internal standard solution (see 4.5.6.1.2) into the vial. Close the vial with a teflon-coated septum and heat at 149-158°F (65-70°C) for 30 minutes. After settling, decant the liquid into a second vial, and evaporate at 140°F (60°C) under a flow of nitrogen. Close the vial and, with a syringe, add 500 µL of the silylating solution (see 4.5.6.1.1) through the septum. Heat for 20 minutes at 149-158°F (65-70°C). Inject 1 µL of the solution into a gas-liquid chromatograph equipped with a 6 foot X 1/8 inch stainless steel column packed with 5 percent silicone OV-225 on 80-100 mesh chromosorb W-HP. Allow the chromatogram to develop for 30 minutes under the following conditions:

Injection port	446°F (230°C)
Column oven	329°F (165°C)
Flame ionization detector	572°F (300°C)
Carrier (helium) flow	60 mL per minute

Calculate the percentage of sodium gluconate in the corrosion removing compound as follows:

$$\text{Sodium gluconate} = 500 \frac{A_X W_{IS} P}{A_{IS} W_S}$$

where A_X = area of the gluconic acid derivative peak.
 A_{IS} = area of the L-ascorbic acid derivative peak.
 W_{IS} = weight of L-ascorbic acid per 100 mL of solution.
 W_S = weight of sample of corrosion removing compound per 500 mL.
 P = correction factor determined with known concentrations.

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4.5.7. Trisodium Salt of N-hydroxylethylenediamine-triacetic Acid (3 Na·EDTAOH) Content (type I only).

4.5.7.1 Reagents.

4.5.7.1.1 Preparation of ZnSO₄·7H₂O solution. Dissolve approximately 0.5 g, accurately weighed, of ZnSO₄·7H₂O in distilled water and dilute to 100 mL in a volumetric flask.

Indicator. Dissolve 0.56 g of hydroxylamine hydrochloride and 0.025 g of erichrome black T in 25 mL of methanol.

4.5.7.2 Test procedure. Add 10.g, weighed to the third decimal, of the well-mixed corrosion removing compound to a 100 mL volumetric flask. Add 50 mL of distilled water and swirl to dissolve. Dilute with methanol to the 100 mL volume mark and mix thoroughly. Pipette 10 mL of solution to a 250 mL round distillation flask and add 50 mL of methanol. Evaporate the solution on a 158°F (70°C) bath under a gentle flow of air. Then evaporate to dryness at 158°F (70°C) under vacuum.

Remove the flask from the bath, add 50 mL methanol, and attach an 18-inch, water-cooled condenser. Reflux 1/2 hour using a 158°F (70°C bath). Filter through medium filter paper, transferring and washing filter paper 3 times with 5 mL of methanol each washing. Add 50 mL of water to the filtrate and mix. Using a pH meter, adjust the filtrate to a pH of about 8.5 with 3N HCL and then to pH 7.0 with 0.1N HCL. Add 5 g of Na₄Cl and dissolve. Add 4 drops of indicator (4.5.7.1.2) and titrate with ZnSO₄·7H₂O (4.5.7.1.1) to a purple end point. Calculate the percent concentration of hydrated 3Na·EDTAOH from the equation:

$$C = 1.35 \frac{AB}{W}$$

where A = weight of ZnSO₄·7H₂O per 100 mL.

B = mL of ZnSO₄·7H₂O.

W = weight of test sample.

4.5.8 Sodium cyanide content (type II only).

4.5.8.1 Preparation of 0.1N silver nitrate solution. Dissolve 16.9890 g of reagent grade silver nitrate in approximately 750 mL of distilled water. Dilute with distilled water to exactly 1 L.

4.5.8.2 Test procedure. Pipette a 5 mL portion of the sample solution (see 4.5.1.2) into a 250 mL flask. Add 100 mL of distilled water, 5 mL of 10 percent potassium iodide solution and 15 mL of ammonium hydroxide. Titrate with the silver nitrate solution (4.5.8.1) until a faint yellow turbidity is produced which will not redissolve with stirring. Calculate the sodium cyanide content by the following formula:

$$\text{Percent NaCN} = 0.544 \times \text{mL AgNO}_3 \text{ used in titration.}$$

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4.5.9 Chelating or sequestering agent content (type II only).

4.5.9.1 Preparation of ammonium oxalate indicator solution. Dissolve 5 g of reagent grade ammonium oxalate in 50 mL of distilled water in a 100 mL volumetric flask. Fill to the mark with distilled water and mix well.

4.5.9.2 Preparation of 0.2M calcium chloride solution. Dissolve exactly 22.2 g of dried reagent grade calcium chloride in distilled water in a 1 L volumetric flask. Dilute to exactly 1 L with distilled water.

4.5.9.3 Preparation of tetrasodium salt of ethylenediamine tetra-acetic acid dihydrate solution. Prepare a standard 90 g per liter solution of EDTA tetrasodium salt dihydrate as follows: Dissolve exactly 80.420 g of reagent grade ethylenediamine tetra-acetic acid disodium dihydrate in 2N sodium hydroxide solution in a 1 L volumetric flask. Fill exactly to the mark with 2N sodium hydroxide and mix well. Titrate a 50 mL portion of this solution with the calcium chloride solution (see 4.5.9.2) and record the mL of CaCl_2 required.

4.5.9.4 Test procedure. Pipette 50 mL of the sample solution (see 4.5.1.2) into a 300 mL conical flask. Add 150 mL of distilled water and 10 mL of the ammonium oxalate indicator solution (4.5.9.1). Titrate with the standard CaCl_2 solution (4.5.9.2) to the first faint turbidity which lasts for at least 30 seconds. Calculate the chelating or sequestering agent content by the following formula:

$$x = 25 \frac{V}{S}$$

Where:

X = percent chelating or sequestering agent.

V = mL of 0.2M CaCl_2 solution required for titration of the sample.

S = mL of the 0.2M CaCl_2 solution required for titration of the EDTA solution (4.5.9.3).

4.5.10 Etching characteristics (type II only). The panels used in the test for foaming characteristics (4.5.3) shall be rinsed with water, dried and examined for any evidence of etching. Check for compliance with 3.5.

5. PREPARATION FOR DELIVERY

5.1 Packaging.

5.1.1 Level A. The corrosion removing compound shall be packaged in 55-gallon steel drums conforming to type IV of PPP-D-729. The volume of the drum shall not exceed the volume of the contents by more than 5 percent.

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5.1.2 Level C. The corrosion removing compound shall be packaged in steel drums in the size and kind specified in the contract or order. Protection shall be such as to prevent deterioration during shipment and to insure safe delivery at destination.

5.2 Packing. Unless otherwise specified, overpacking of the containers is not required.

5.3. Marking. Containers shall be marked in accordance with MIL-STD-129. In addition, each container shall be durably and legibly marked with the manufacturer's instructions for mixing and use and with the following warning notice:

"WARNING

Causes severe burns to skin and eyes. Wear goggles or face shield when handling. Avoid dust and fumes. Keep away from food products. In case of eye or skin contact, flush immediately with plentiful amounts of water for at least 15 minutes and get immediate medical attention."

5.3.1 In addition to the marking of 5.3, each container of type II corrosion removing compound shall be durably and legibly marked with the following warning:

"POISON

Contains sodium cyanide. Do not mix with or allow to come in contact with acids or acid solutions. Contact with acid liberates poisonous cyanide gas. Keep container closed and in a dry place."

6. NOTES

6.1 Intended use. The corrosion removing compounds covered by this specification are used for removing rust and scale from iron and steel with or without attendant paint, grease or other surface deposits by either simple immersion of the articles or by electrolytic action. The compounds may be used for cleaning some nonferrous metals; however, its effect on the metal to be cleaned should be checked before use for cleaning items. These compounds have been found to attack aluminum, copper, brass and zinc. These corrosion removing compounds should not be used in washing machines because of their attack on some metals. Treatment for rust removal need not be extended beyond the point of effectiveness in removing the surface deposits. Articles should be thoroughly rinsed in clean hot water and dried immediately after removal from the corrosion removing compound. Unless immersion is to be promptly followed by surface finishing, articles should be coated with a light water displacing preservative oil such as VV-L-800.

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6.1.1 Type I compound. Type I compound is to be used at a concentration of 5 pounds per gallon of water. Type I corrosion removing compound contains no cyanide and no electrolytic action is needed. However, the removal of surface deposits is slow and a long soaking period is required to do a satisfactory job. This type of compound is most effective when used at a rolling boil. Agitation of the bath reduces the soaking time.

6.1.2 Type II compound. Type II compound is to be used at a concentration of 2 to 3 pounds per gallon of water. Type II solution uses both cyanide and electrolytic action. The removal of surface deposits is more rapid than with Type I compound and the soaking period is shortened. The use of type II compound is recommended where a source of direct current and methods for cyanide disposal are available. In order to reduce the loss of cyanide, the type II solution should not be used at a bath temperature higher than 130°P (54°C).

6.2 Ordering data. Procurement documents should specify the following:

- (a) Title, number and date of this specification.
- (b) Type of compound (see 1.2).
- (c) Level of packaging required (see section 5).
- (d) Size and type of container if Level C is specified (see 5.1.2).

6.3 Definitions. The following definitions apply to terms used in this specification:

Chelate compound - Pertaining to or designating a compound which by means of 2 valences (principal, residual, or both) attaches itself to a central metallic atom so as to form a chelate group or ring.

Sequestrant compound - Sequestering agents function in the same manner as chelating compounds except that they do not form a ring.

Military custodians:

Army - MR
Navy - AS
Air Force - 68

Preparing activity:

Army - MR

Project Number 6850-0627

Review activities:

Army - MI
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DOCUMENT IDENTIFIER (Number) AND TITLE

MIL-C-14460C, Corrosion Removing Compound, Sodium Hydroxide Base;
for Electrolytic or Immersion Application, Proj. No. 6850-0627

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