MIL-C-46110B
January 17, 1984
SUPERSEDING
MIL-C-46110A(MR)
6 August 1973

MILITARY SPECIFICATION

COATING COMPOUND, OXIDE BLACK

This specification is approved for use by the Army Materials and Mechanics Research Center, Department of the Army, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers one grade of a coating compound for use in blackening ferrous metals to conform to MIL-C-13924, class 1. (for wrought iron, plain carbon, and low alloy steels.)

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.

SPECIFICATIONS

FEDERAL

PPP-D-723 - Drums, Fiber
PPP-D-729 - Drums, Metal, 55-Gallon (For Shipment of Noncorrosive Material)
PPP-D-705 - Drum, Shipping and Storage; steel 16 and 30 gallon capacity

MILITARY

MIL-C-13924 - Coating, Oxide, Black, For Ferrous Metals

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-SMS, 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.

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129 - Marking for Shipment and Storage
MIL-HDBK 205 - Phosphating and Black Oxide Coating of Ferrous Metals

(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.)

2.2 Other publications. The following document(s) form a part of this specification to the extent specified herein. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)
D 3951 - Standard Practice for Commercial Packaging

(Application for Copies should be addressed to the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pennsylvania 19103)

Hazardous Materials Regulations Publications including:

1) Specifications for Shipping Containers, Tariff No. 25, or current issue in effect.

(Application for copies should be addressed to Publishing Agent, 1920 "L" Street, N.W., Washington, D.C. 20036.)

2) Transportation of Hazardous Materials by Motor, Rail and Water, Tariff No. 15, or current issue in effect.

(Application for copies should be addressed to Publishing Agent, 1616 "P" Street, N.W., Washington, D.C. 20036.)

The Federal Caustic Poison Act.

(Information as to the availability of the Federal Caustic Poison Act may be obtained from the Department of Health, Education and Welfare, 330 Independence Avenue, S.W., Washington, D.C. 20201.)

AMERICAN NATIONAL STANDARDS INSTITUTE
ANSI Z129.1-76 - Chemicals, Hazardous Industrial, Precautionary Labeling of

(Application of copies should be addressed to: American National Standards Institute 1430 Broadway, New York, NY 10018.)

INTERSTATE COMMERCE COMMISSION
49 CFR 100-199 Rules and Regulations for the Transportation of Explosives and other Dangerous Articles

(Applications for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.)

(Industry association specifications and standards are generally available from libraries. They are also distributed among technical groups and using Federal agencies).

3. REQUIREMENTS

3.1 Material. The coating compound shall consist of ground flake or granular sodium hydroxide and granular or bead sodium nitrate intimately blended together as a coarse, dust-free material. The coating compound shall contain no added sulfur compounds, other than those present in the basic chemical ingredients. The particle size shall not be greater than 1/4 inch maximum for any dimension when examined visually. The coating compound shall be capable of producing a black oxide coating meeting the requirements of MIL-C-13924, class 1.

3.2 Color. The mixture shall have a white or pale yellow color when examined in accordance with 4.4.3.

3.3 Chemical requirements. The mixture shall conform to the applicable chemical requirements specified in table I when tested as specified in the corresponding test paragraphs in section 4.

Table I. Chemical requirements

Property	Percentage	Test method
Moisture	0.5 max.	4.4.4
Insoluble matter	0.5 max.	4.4.5
Chlorates (as $NaClO_3$)	0.06 max.	4.4.6
Chlorides (as NaCl)	1.0 max.	4.4.7
Sodium Hydroxide (as NaOH)	63.0 min.	4.4.9
Carbonate (as Na_2CO_3)	1.3 max.	4.4.10
Total Alkalinity (as Na_2O)	48.8 min.	4.4.11
Sodium Nitrate (as $NaNO_3$)	33.0 min.	4.4.12
Calcium (as CaO)	0.1 max.	4.4.13
Magnesium (as MgO)	0.1 max.	4.4.14
Sulfates (as Na_2SO_4)	0.5 max.	4.4.15

3.4 Workmanship. The material shall be processed in a manner that will produce a uniform mixture when examined visually (see 4.4.3).

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.

4.2 Acceptance inspection. Conformance of the material to the requirements of this specification shall consist of an examination for acceptability of quality control methods used by the manufacturer, examining and testing the acceptance samples (4.3.3) for all the requirements, and an examination of the sample of filled containers (4.3.4) for conformance to the packaging, packing, and marking requirements.

4.3 Sampling.

4.3.1 Inspection lot. A lot shall consist of the material produced by one manufacturer under essentially the same manufacturing conditions with no change in materials and provided the operation is continuous. In the event that the process is a batch operation, each batch shall constitute a lot (see 6.3).

4.3.2 Nondestructive examination. Sampling shall be conducted in accordance with MIL-STD-105.

4.3.3 Sample for tests. Three separate 1-pound samples shall be taken from each inspection lot. When the material is produced by a batch process the three samples shall be taken at different locations within each batch or from three separate containers. When the material is produced by a continuous-run process the three samples shall be taken so as to represent, respectively, the first part, the middle part, and the last part of the run of the constituted inspection lot. The individual specimens shall be tested as specified in 4.4.

4.3.4 Examination of filled containers. A sample of filled containers shall be taken at random in accordance with MIL-STD-105, Acceptable Quality Level (AQL) equal to 2.5 percent defective and checked for defects listed in 4.3.5.1.

4.3.5 Classification of defects.

4.3.5.1 Preparation for delivery (section 5).

Critical:	None defined
Major:	AQL 2.5 percent defective
101	Unit of shipping container not as specified
102	Quantity of material per unit or shipping container not as specified

103	Closure of unit or shipping container not as specified
104	Unit or shipping container damaged
105	Evidence of sifting or leakage
106	Marking of unit or shipping container, illegible, incorrect, or incomplete

4.4 Inspection methods. Unless otherwise specified, the chemical values specified in table 1 shall apply to the average of the determinations made on the product.

4.4.1 Conformance of the mixture to the requirements for material (3.1) and workmanship (3.4) shall be determined by processing 2" x 4" x 1/8" panels of low carbon steel, cold rolled, in a prepared aqueous solution (approximately 9 pounds per gallon) operated at a rolling boil for 285 to 305°F (140-152°C) for a period of 30 minutes, maximum. The panels shall be examined and tested for compliance of the black oxide coating with class 1 of MIL-C-13924.

4.4.2 Tests. Distilled water and analytical reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections applied where significant. Tests shall be conducted as follows:

4.4.3 Color and form. The composite and individual specimens shall be visually examined for compliance with 3.2 and 3.4.

4.4.4 Moisture (see 6.4). Transfer a weighed 10 gram portion of the mixture to a previously tared shallow weighing bottle, heat in a convection oven for 2 hours at 302 ± 5°F (150 ± 3°C), cover, cool in a desiccator and weigh. Calculate the loss in weight as the percentage of moisture in the sample.

$$\text{Percent moisture} = \frac{100\ (A-B)}{W}$$

where:

A = Weight of bottle plus sample before drying in grams
B = Weight of bottle plus sample after drying in grams
W = Weight of sample in grams

4.4.5 Insoluble matter. Transfer a weighed 25 gm sample of the mixture to a beaker. Heat to boiling with 200 ml. of distilled water and decant the solution through a filtering crucible of fine porosity having a nominal maximum pore diameter of 4.5 to 5 microns which has been dried at 221 ± 9°F (105 ± 5°C) and weighed. Rinse the beaker with boiling distilled water and transfer any adhering insoluble matter to the filter. Decant the wash water from the beaker through the filter. The filtering crucible shall be dried in

an oven at 221 ± 9°F (105 ± 5°C) for three hours, cooled in a desiccator and weighed. The increase in weight shall be calculated as percent insoluble matter as follows:

$$\text{Percent insoluble matter} = \frac{100\ (A-B)}{W}$$

where:

A = Weight of crucible plus residue in grams
B = Weight of crucible in grams
W = Weight of sample in grams

4.4.6 Chlorates. Transfer a weighed 25 gm sample of the dried mixture to a beaker and dissolve in hot distilled water. Add sufficient HNO_3 to make the solution acid to litmus and then add 5 ml. of HNO_3 and an excess of silver nitrate solution and stir. Filter the solution to remove any chlorides which may have been precipitated. Add to the solution 10 ml. of 30 percent formaldehyde solution and heat on the water bath for 1/2 hour. Chlorate is thus reduced to chloride and precipitated by the excess silver nitrate. Filter off the precipitate on a tared filtering crucible of fine porosity having a nominal maximum pore diameter of 4.5 - 5 microns, which has previously been dried at 221 ± 9°F (105 ± 5°C). Dry the crucible and precipitate for 3 hours at 221 ± 9°F (105 ± 5°C). Cool in a desiccator and weigh. Calculate the percentage of sodium chlorate as follows:

$$\text{Precent sodium chlorate} == \frac{74.3\ A}{W}$$

where:

A = Weight of precipitate in grams
W = Weight of sample in grams

4.4.7 Chlorides. Carefully weigh a 25 gm dried sample of the mixture and transfer it to a 600 ml. beaker. The sample shall be dissolved in 300 ml. of hot (approximately 185°F) (85°C) distilled water and 35 ml. of concentrated nitric acid added. Twenty-five millilliters of approximately 0.1 N silver nitrate solution shall be added to the solution with the aid of a pipette. The solution shall be heated to boiling on a hot plate with occasional stirring and boiled moderately for approximately 2 minutes with constant stirring. The beaker and contents shall be removed from the hot plate. The precipitate shall be allowed to settle (if the precipitate does not settle rapidly it is permissible to filter the solution as specified and test the filtrate) and one drop of the supernatant liquid shall be added to one drop of a one percent solution of sodium chloride on a black porcelain spot plate. If no precipitate occurs on the spot plate, a second 25-ml. portion of silver nitrate solution shall be added to the sample, boiled as indicated above, and the spot test repeated. The addition of silver nitrate solution shall be continued and the spot test repeated as specified above until a precipitate of silver chloride is obtained. The supernatant solution shall be filtered through a tared pyrex sintered glass crucible of fine

porosity, having a nominal maximum pore diameter of 4.5 to 5 microns, and with the aid of suction, the filtrate shall be collected in a 1000-ml. filtering flask. The silver chloride precipitate shall be transferred quantitatively to the filtering crucible with the aid of five 20 ml. portions of 1:100 nitric acid solution and one 20 ml. portion of distilled water. The crucible and contents shall be dried in an oven at 221 ± 9°F (105 ± 5°C) for 3 hours. The crucible shall be cooled in a desiccator and weighed. The percent chlorides, as sodium chloride in the sample, shall be calculated as follows:

$$\text{Percent sodium chloride} = \frac{40.8\ (A-B)}{W}$$

where:

A = Weight of crucible plus precipitate in grams
B = Weight of crucible in grams
W = Weight of sample in grams

4.4.8 Preparation of solutions.

4.4.8.1 Barium chloride solution. Dissolve 10 grams of barium chloride ($BaCl_2 2H_2O$) in freshly boiled and cooled distilled water and dilute to 100 milliliters. Use this solution in 4.4.9 and 4.4.15.

4.4.8.2 Sample test solution. Weigh quickly to the nearest milligram into a glass-stoppered weighing bottle approximately 30 g. of the sample material (see 6.4). Transfer the weighed material to a 1-liter volumetric flask, rinsing the bottle several times with water to assure complete transfer of the material. Add sufficient freshly boiled and cooled water to half fill the flask and agitate the contents until all solid material is dissolved. Cool to room temperature and dilute with water to mark. Save this test solution for determinations 4.4.9, 4.4.12, 4.4.13, and 4.4.15. Use the above sample weight as S in calculations of 4.4.9 through 4.4.15.

4.4.9 Sodium hydroxide. Pipette 50.0 ml. of the test solution into a 250 ml. Erlenmeyer flask, add 25 ml. of a neutral 10-percent aqueous solution of barium chloride, and titrate with 0.5 N hydrochloric acid, using 3 drops of phenolphthalein solution, to disappearance of the pink color. Save this solution for the carbonate determination in 4.4.10. Calculate the percent sodium hydroxide as follows:

$$\text{Percent sodium hydroxide} = \frac{80\ VN}{S}$$

where:

V = Volume of hydrochloric acid solution used, in ml.
N = Normality of hydrochloric acid solution
S = Weight of specimen used, in g. (see 4.4.8.2)

4.4.10 Carbonate. Add 2 drops of methyl orange indicator to the solution reserved from the sodium hydroxide assay (see 4.4.9) and titrate with 0.1 N hydrochloric acid solution to a pink color, which persists for one minute. Calculate the percent of carbonate as sodium carbonate as follows:

$$\text{Percent carbonate (as } Na_2CO_3) = \frac{(212)\ (B)\ (M)}{S}$$

where:

B = Volume of hydrochloric acid solution used in ml.
M = Normality of hydrochloric acid solution used
S = Weight of specimen used, in grams (see 4.4.8.2)

4.4.11 Total alkalinity. Calculate the total alkalinity, as Na_2O as follows:

$$\text{Percent total alkalinity, as } Na_2O = \frac{62\ (VN + BM)}{S}$$

where:

V = Volume of hydrochloric acid solution used in 4.4.9 in ml.
N = Normality of hydrochloric acid solution used in 4.4.9
B = Volume of hydrochloric acid solution used in 4.4.10 in ml.
M = Normality of hydrochloric acid solution used in 4.4.10
S = Weight of specimen used, in grams (see 4.4.8.2)

4.4.12 Sodium nitrate determination. Pipette 5 ml. of the test solution (see 4.4.8.2) into a 250-ml. beaker. Add 100 ml. of distilled water and 2.0 ml. of 6 N H_2SO_4. Heat to boiling, and add 10 ml. of nitron reagent (10 gm. of nitron in 100 ml. of 50% glacial acetic acid. Keep in a dark colored bottle). Cool and place in an ice-bath for 1-1/2 hours. Filter through a weighed, fritted glass crucible of medium porosity, having a nominal maximum pore diameter of 10-15 microns, and place a piece of ice in the crucible before filtering. The solution must be cooled to reduce the solubility of the nitron compound. Use the filtrate to effect transfer of all the precipitate. Wash the collected precipitate in the crucible with four 3-ml. portions of ice water. Reboil the filtrate, and add a small quantity of nitron reagent to check the completeness of the precipitation. Dry the crucible at 221 $\pm$ 9°F (105 $\pm$ 5°C). Cool in a desiccator, and weigh. Repeat the weighing procedure until a constant weight is obtained. Calculate the percent sodium nitrate as follows:

$$\text{Percent sodium nitrate} = \frac{(4530)\ (\text{Weight of precipitate})}{S}$$

S = Weight of specimen used in grams (see 4.4.8.2)

4.4.13 Calcium oxide. By means of a pipette, transfer to a beaker 100 ml. of the test solution prepared as specified in 4.4.8.2. Make distinctly acid with 5 ml. of concentrated HNO_3. Then make distinctly alkaline with NH_4OH and heat to boiling. Precipitate the calcium by adding 10 ml. of a saturated solution of ammonium oxalate. Continue the boiling for a few minutes, filter the precipitate on #42 Whatman paper or equivalent, wash with cold distilled water containing a little NH_4OH, and determine the percentage of calcium oxide either gravimetrically or volumetrically. Save the filtrate for the magnesium determination in 4.4.14.

4.4.13.1 Volumetric determination. Wash the filter paper and precipitate thoroughly and then transfer paper and precipitate to a beaker containing 400 ml. hot water to which has been added 10 ml. of 18 N sulfuric acid. Disintegrate the paper by stirring vigorously with a glass rod and then titrate rapidly while hot with approximately 0.05N $KMnO_4$ solution until a pink endpoint persists for one-half minute. Calculate the percentage of calcium oxide as follows:

$$\text{Percent of calcium oxide} = \frac{28\ VN}{S}$$

where:

V = ml. of $KMnO_4$ solution used
N = Normality of $KMnO_4$ solution
S = Weight of sample in grams (see 4.4.8.2)

This method is preferred for small amounts of calcium oxide since it is more accurate.

4.4.13.2 Gravimetric determination. The precipitate and filter paper is placed in a weighed crucible and gently heated, the crucible being covered, until the water is expelled and the paper charred. Do not let paper burn or let flame occur. Transfer to a muffle furnace and heat to 1200°C for 5 to 10 minutes. It is well to remove the cover for an instant to assist escape of the CO_2 during the heating. The covered crucible and its contents are cooled in a desiccator containing concentrated sulfuric acid. The cooled CaO is weighed and the ignition repeated until the weight is constant. Calculate the percentage of calcium oxide as follows:

$$\text{Percent of calcium oxide} = \frac{1000\ (A-B)}{S}$$

where:

A = Weight of crucible plus residue in grams
B = Weight of crucible in grams
S = Weight of sample in grams (see 4.4.8.2)

This method is preferred for percentages of calcium oxide near the maximum.

4.4.14 Magnesium. Catch the filtrate and wash water from the calcium determination (4.4.13) in a beaker. Add 10 ml. of a 10 percent solution of disodium phosphate and then a volume of concentrated NH_4OH (27 to 30%) equal to approximately 1/10 volume of liquid in beaker. Mix thoroughly and allow to stand overnight. Filter the precipitate on #42 Whatman filter paper or equivalent, wash with 5-percent NH_4OH and ignite in a tared platinum crucible. If the ignition does not result in a white precipitate, cool the crucible and contents, add a few drops of nitric acid or ammonium nitrate solution, heat gradually and then ignite fully. Spattering must be avoided. Cool in a desiccator and weigh. Calculate the percentage of magnesium oxide as follows:

$$\text{Percent magnesium oxide} = \frac{362\ (A-B)}{S}$$

where:

A = Weight of precipitate plus crucible in grams
B = Weight of crucible in grams
S = Weight of sample in grams (see 4.4.8.2)

4.4.15 Sulfates. By means of a pipette transfer to a beaker 200 ml. of the test solution prepared as specified in 4.4.8.2. Add sufficient HCl to make the solution slightly acid, and heat to boiling. Slowly add 10 ml. of a 10% solution of $BaCl_2$ (see 4.4.8.1) and continue boiling for several minutes. Settle the precipitate on a steam bath and then filter in a tared filtering crucible of fine porosity having a nominal maximum pore diameter of 4.5 to 5 microns. Calculate the percentage of sodium sulfate as follows:

$$\text{Percent sodium sulfate} = \frac{304.5\ (A-B)}{S}$$

where:

A = Weight of precipitate plus crucible in grams
B = Weight of crucible in grams
S = Weight of sample in grams (see 4.4.8.2)

4.4.16 Acceptance and rejection criteria. If the individual or composite sample fails to meet the test requirements of the specification, the lot shall be rejected. When rejected lots of the material are resubmitted for acceptance inspection, samples shall be selected in accordance with the provisions of MIL-STD-105 employing tightened inspection.

5. PACKAGING

5.1 Preservation and packaging. Not applicable.

5.2 Packing. Packing shall be level A or C as specified (see 6.2).

5.2.1 Level A. Coating Compound, Oxide Black shall be packed in quantities of 100 and 400 pounds (45 and 180 kilograms) as specified by the procuring agency. One hundred pounds shall be packed in a fiber drum conforming to type III, grade A, of PPP-D-723 or in a steel drum conforming to type III of PPD-D-705. Net weight of the material of each drum shall be 100 pounds (45 kilograms). Four hundred pounds (180 kilograms) shall be packed in steel drums PPP-D-729, meeting the requirements of the Department of Transportation, 17H or Rule 40 of 18 gauge, with full removable head, and locking ring closure. Net weight of the material of each drum shall be 400 pounds (180 kilograms). Containers shall be provided with a 0.004 inch polyethylene liner, sealed as practicable. The exterior finish for the 400 pound (180 kilograms) drums may be a suitable commercial coating.

5.2.2 Level C. Coating Compound, Oxide Black shall be packed to afford protection against damage during direct shipment from the source of supply to the first receiving activity for immediate use. Materials shall be packed in accord with CFR 49.

5.2.3 Marking. In addition to any marking required by the contract or order, shipments shall be marked in accordance with MIL-STD-129.

5.2.4 Corrosive label. A corrosive materials label shall be affixed to each drum in accordance with the Code of Federal Reglations, Title 49, Parts 100-199.

5.2.5 Caution label. A caution label shall conform to ANSI Z 129.1 American Mutual Standard. The label shall be in bold type and securely affixed to the container as follows:

DANGER! CAUSES SEVERE BURNS TO SKIN AND EYES

Avoid contact with skin, eyes, and clothing. Do not take internally. When handling, wear goggles and face shield. While making solutions, add material slowly to surface of solution to avoid violent spattering. In case of contact, immediately flush skin with plenty of water for at least 15 minutes and get medical attention."

6. NOTES

6.1 Intended use. The material covered by this specification is intended for use in blackening wrought iron, plain carbon, and low alloy steels, listed as class 1 of MIL-C-13924. Information on procedures for blackening steel including concentration of solution, temperature of solution, and time of immersions are noted in MIL-HDBK-205.

6.2 Ordering data. Procurement documents should specify the following:

(a) Title, number and date of this specification.

(b) Level of packaging and packing required (see 5.1 and 5.2).

(c) Container capacity required.

6.3 Batch. A batch is defined as that quantity of material which has been manufactured by some unit chemical process and subjected to some physical operation intended to make the final product substantially uniform.

6.4 This material rapidly deliquesces in air and absorbs carbon dioxide. Contact with air must be avoided as much as possible in order to eliminate errors in analysis.

Custodian:

Army - MR
Air Force - 68

Preparing activity:

Army - MR

Review Activities:

Army - GS, MI, AR, ER, SM
Air Force - 68
Navy - AS

Project No. 6850-0696

User activities:

Army - AR
Navy - SH, OS, MC

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(See Instructions – Reverse Side)

1. DOCUMENT NUMBER	2. DOCUMENT TITLE
MIL-C-46110 B	

3a. NAME OF SUBMITTING ORGANIZATION	4. TYPE OF ORGANIZATION *(Mark one)*
	☐ VENDOR
b. ADDRESS *(Street, City, State, ZIP Code)*	☐ USER
	☐ MANUFACTURER
	☐ OTHER *(Specify)*: _____

5. PROBLEM AREAS

a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

6. REMARKS

7a. NAME OF SUBMITTER *(Last, First, MI)* – Optional	b. WORK TELEPHONE NUMBER *(Include Area Code)* – Optional
c. MAILING ADDRESS *(Street, City, State, ZIP Code)* – Optional	8. DATE OF SUBMISSION *(YYMMDD)*

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