

DOD-N-51512

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SUPERSEDING

O-N-335

January 11, 1962

MILITARY SPECIFICATION

NICKEL SALTS, ELECTROPLATING (METRIC)

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

1. SCOPE

1.1 Scope. This specification covers three types of nickel salts used for electroplating.

1.2 Classification. Nickel salts shall be of the following types as specified (see 6.2):

- Type I - Nickel sulfate
- Type II - Nickel ammonium sulfate
- Type III - Nickel chloride

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. The following specifications and standards form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation.

: Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, U.S. Army Chemical Research and Development Center, ATTN: SMCCR-SPD-TS, Aberdeen Proving Ground, MD 21010-5423 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 6810

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SPECIFICATIONS

FEDERAL

PPP-C-2020 - Chemicals, Liquid, Dry, and Paste: Packaging of
 PPP-D-723 - Drums, Fiber

STANDARDS

FEDERAL

Fed. Std. No. 313 - Material Safety Data Sheets Preparation and the
 Submission Of

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by
 Attributes
 MIL-STD-129 - Marking for Shipment and Storage
 MIL-STD-147 - Palletized Unit Loads

2.1.2 Other Government documents. The following other Government documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issues shall be those in effect on the date of the solicitation.

CODE OF FEDERAL REGULATIONS (CFR)

49 CFR 171 to 179 - Department of Transportation Hazardous Materials
 Regulations

(The Code of Federal Regulations is available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Orders for the above publication should cite "49 CFR 171 to 179".)

(Copies of specifications, standards and other Government documents required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting activity.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are indicated as DOD adopted shall be the issue listed in the current DODISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS shall be the issue of the nongovernment documents which is current on the date of the solicitation.

ASTM STANDARDS

D 1193 - Reagent Water (DOD Adopted)

(Application for copies should be addressed to ASTM, 1916 Race Street, Philadelphia, PA 19103.)

(Nongovernment standards and other publications are normally available from the organizations which prepare or which distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein (except for associated detail specifications, specification sheets or MS standards), the text of this specification shall take precedence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Composition. Type I nickel salt shall be nickel sulfate, hexahydrate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$) or a mixture of nickel sulfate, hexahydrate and nickel sulfate, heptahydrate ($\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$). Type II nickel salt shall be nickel ammonium sulfate, hexahydrate [$\text{NiSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$]. Type III nickel salt shall be nickel chloride, hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$). No materials other than the specified materials shall be used. The contractor shall certify that the above requirements have been met.

3.2 Chemical characteristics. Nickel salts shall conform to the applicable chemical characteristics of table I when tested as specified therein.

TABLE I. Chemical characteristics

Characteristic	Percent by weight			Test paragraph
	Type I	Type II	Type III	
Nickel, minimum	21.4	14.6	24.5	4.2.4.1
Copper, maximum	0.005	0.005	0.005	4.2.4.2
Iron, maximum	0.020	0.020	0.020	4.2.4.3
Zinc, maximum	0.025	0.025	0.025	4.2.4.4
Insoluble matter, maximum	0.05	0.05	0.05	4.2.4.5
Free acid, maximum	0.10	0.10	0.10	4.2.4.6

3.3 Material Safety Data Sheets. Material Safety Data Sheets for the applicable type nickel salt shall be prepared and submitted by the contractor as specified in Fed. Std. No. 313 (see 6.5).

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.

4.1.1 Responsibility for compliance. All items must meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling in quality conformance does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to acceptance of defective material.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the nickel salt of one type, produced by one manufacturer, at one plant, from the same materials, and under essentially the same manufacturing conditions provided the operation is continuous. In the event the process is a batch operation, each batch shall constitute a lot (see 6.3).

4.2.2 Sampling.

4.2.2.1 For examination of packaging. Sampling shall be conducted in accordance with MIL-STD-105.

4.2.2.2 For nickel salt test. Sampling shall be conducted in accordance with table II. A representative specimen of approximately 100 grams (g) shall be removed from each sample container and placed in a suitable clean, dry, sealed container labeled to identify the lot and container from which it was taken.

TABLE II. Sampling for nickel salt test

: Number of containers in batch or lot :	Number of sample containers :
:	:
: 2 to 25 :	: 2 :
: 26 to 150 :	: 3 :
: 151 to 1,200 :	: 5 :
: 1,201 to 7,000 :	: 8 :
: 7,001 to 20,000 :	: 10 :
: Over 20,000 :	: 20 :
:	:

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. The sample unit shall be one filled unit, intermediate, or shipping container, as applicable, ready for shipment. Sample unit, intermediate, and shipping containers shall be examined for the following defects using an AQL of 2.5 percent defective:

- (a) Contents per container not as specified
- (b) Container not as specified
- (c) Container closure not as specified
- (d) Container damaged or leaking
- (e) Unitization not as specified (when required)
- (f) Marking incorrect, missing, or illegible

4.2.3.2 For nickel salt test. Each sample specimen taken in 4.2.2.2 shall be tested as specified in 4.2.4. Failure of any test by any specimen shall be cause for rejection of the lot represented.

4.2.4 Tests. Water in accordance with ASTM D 1193 and 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:

4.2.4.1 Nickel. Weigh to the nearest 0.1 milligram (mg) 1.5 to 1.7 g of the specimen, dissolve in water, and dilute with water to 200 milliliters (mL). Transfer 50 mL to a 500-mL beaker and dilute to 200 mL with water. Add 1 g of sodium citrate, heat to boiling, then add to the boiling solution, with stirring, a solution of 0.6 g of dimethylglyoxime in 100 mL of warm ethanol; follow with 5 mL of concentrated ammonium hydroxide, and let stand overnight. Filter on a Gooch crucible, wash with hot water, then with 50-percent ethanol, and dry to constant weight at 110°C. Calculate the percent by weight nickel as follows:

$$\text{Percent nickel} = \frac{81.3A}{W}$$

where: A = Weight of final precipitate in grams and
W = Weight of specimen in grams.

4.2.4.2 Copper.

(a) Standard copper solution. Dissolve 131 g of cupric sulfate, pentahydrate in water and dilute to 1 liter with water.

(b) Procedure. Dissolve 2.0 g of specimen in 2 mL of concentrated hydrochloric acid and sufficient water to make 50 mL of solution. Add 2 mL of 1-percent mercuric chloride solution and saturate the solution with hydrogen sulfide. Filter through a small filter, wash with hydrogen sulfide water, and ignite the filter precipitate in a porcelain crucible. Dissolve the residue by

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warming it with 0.5 mL of concentrated nitric acid and a few drops of water, dilute to 10 mL with water, and filter if necessary. Dissolve 1 g of ammonium acetate in the solution; then add 5 drops of freshly prepared potassium ferrocyanide solution. Any red color produced shall be no darker than that produced in a control made with 3 mL of the standard copper solution prepared in (a) and the same quantities of concentrated nitric acid, ammonium acetate, and potassium ferrocyanide used in the specimen procedure in the same final volume as in the specimen procedure.

4.2.4.3 Iron.

(a) Standard iron solution. Dissolve 86.3 mg of ferric ammonium sulfate, $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, in 2 mL of 10-percent sulfuric acid and dilute to 1 liter with water.

(b) Procedure. Dissolve 0.10 g of the specimen in 20 mL of water and add 2 mL of concentrated hydrochloric acid and 5 drops of concentrated nitric acid. Bring the solution to a boil and add sufficient ammonium hydroxide to dissolve the precipitate first formed. Filter and wash with 2.5-percent ammonia until the washings are colorless. Dissolve any precipitate on the filter with 5 mL of hot 20-percent hydrochloric acid and dilute to 40 mL with water. Add 1 mL of concentrated hydrochloric acid and dilute to 50 mL with water. Add 30 mg of ammonium persulfate and 3 mL of ammonium thiocyanate and mix. Any red color produced shall be no darker than that produced in a control containing 1 mL of the standard iron solution prepared in (a), 2 mL of concentrated hydrochloric acid, 3 mL of ammonium thiocyanate and 30 mg of ammonium persulfate in the same final volume as the specimen.

4.2.4.4 Zinc.

(a) Standard zinc solution. Dissolve 124 mg of zinc oxide in a mixture of 10 mL of water and 1 mL of concentrated sulfuric acid and dilute to 1 liter with water.

(b) Procedure. Dissolve 2.0 g of specimen in 10 mL of water and 2 drops of 10-percent sulfuric acid. Heat the solution and slowly add it with stirring to 10 mL of 10-percent sodium hydroxide solution. Boil for 2 minutes, cool to room temperature, filter, and dilute the filtrate to 40 mL with water. Neutralize 30 mL of the filtrate with concentrated sulfuric acid for types I and III nickel salts or with concentrated hydrochloric acid for type II nickel salt, add 1 mL excess of the applicable acid, and dilute to 40 mL with water (this is solution A). To the remaining 10 mL of the filtrate, add 2.5 mL of the standard zinc solution prepared in (a), neutralize with concentrated sulfuric acid for types I and III nickel salts or with concentrated hydrochloric acid for type II nickel salt, add 1 mL excess of the applicable acid, and dilute to 40 mL with water (this is solution B). To both solutions A and B, add 2 mL of freshly prepared potassium ferrocyanide solution, stir well, and allow to stand for 10 minutes. Any turbidity produced in solution A shall not exceed that produced in solution B.

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4.2.4.5 Insoluble matter. Weigh to the nearest 0.1 g approximately 25 g of specimen and dissolve in 125 mL of water and 2.5 mL of 1N hydrochloric acid. Heat on a steam bath for 1 hour. Filter through a tared filter crucible and wash thoroughly with hot water. Dry the crucible and residue at 105°C, cool to room temperature in a desiccator, and reweigh. Calculate the percent by weight insoluble matter as follows:

$$\text{Percent insoluble matter} = \frac{100A}{W}$$

where: A = Weight of residue in grams and
W = Weight of specimen in grams.

4.2.4.6 Free acid. Weigh to the nearest 0.1 g approximately 20 g of specimen and dissolve in 100 mL of water. Add a few drops of methyl red or bromcresol purple indicator. If the indicator color is in the acid range, titrate the solution with 0.1N sodium hydroxide solution to neutral color (corresponding to a pH of approximately 6). Calculate the percent by weight free acid as sulfuric acid for types I and II nickel salts and as hydrochloric acid for type III nickel salt as follows:

$$\text{Percent free acid} = \frac{ABC}{W}$$

where: A = Milliliters of sodium hydroxide solution used in the titration,
B = Normality of the sodium hydroxide solution,
C = 4.9 for types I and II nickel salts and 3.65 for type III nickel salt, and
W = Weight of specimen in grams.

5. PACKAGING

5.1 Preservation. A unit quantity of nickel salt as specified (see 6.2) shall be unit packed level A or C as specified (see 6.2).

5.1.1 Level A.

5.1.1.1 Bottle. Bottles shall be preserved and intermediately packed level A in accordance with the level A requirements of PPP-C-2020 for type I, class 1 glass bottles.

5.1.1.2 Drums. Drums shall conform to type II or III, grade A of PPP-D-723. Drums shall be provided with a barrier lining which shall neither affect nor be affected by the nickel salt. Closure shall be in accordance with PPP-D-723.

5.1.1.3 Sacks. Sacks shall conform to the level A requirements of PPP-C-2020 for type IV, class 4 paper sacks.

5.1.2 Level C. Nickel salt shall be unit packed in accordance with applicable Department of Transportation (DOT) regulations.

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

5.2.1 Level A.

5.2.1.1 Bottles. Bottles shall be packed level A in accordance with the applicable level A packing requirements in PPP-C-2020.

5.2.1.2 Drums and sacks. Unit packs of drums and sacks shall serve as the shipping container.

5.2.2 Level C. Nickel salt unit packed as specified in 5.1 shall be packed for shipment in accordance with applicable DOT regulations.

5.3 Unitization. When specified (see 6.2), containers shall be palletized in accordance with the applicable requirements of MIL-STD-147.

5.4 Marking. All containers and unitized loads shall be marked in accordance with MIL-STD-129 and DOT regulations.

6. NOTES

6.1 Intended use. Nickel salts are intended for use in electroplating.

6.2 Ordering data. Acquisition documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Type of nickel salt required (see 1.2).
- (c) Unit quantity required (see 5.1).
- (d) Level of preservation and packing required (see 5.1 and 5.2).
- (e) If unitization is required (see 5.3).

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

6.4 Significant places. For the purpose of determining conformance with this specification, an observed or calculated value should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM E 29.

6.5 Material Safety Data Sheets. Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with Fed. Std. No. 313. The pertinent mailing addresses for submission of data are listed in appendix B of Fed. Std. No. 313.

6.6 Supersession data. This specification supersedes and includes the requirements of O-N-335, Nickel Salts, Electroplating, dated January 11, 1962.

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Custodian:

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Review activities:

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Preparing activity:

Army - EA

Project No. 6810-B521

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STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions - Reverse Side)

1. DOCUMENT NUMBER
DOD-N-515122. DOCUMENT TITLE
NICKEL SALTS, ELECTROPLATING (METRIC)

3a. NAME OF SUBMITTING ORGANIZATION

4. TYPE OF ORGANIZATION (Mark one)

☐ VENDOR☐ USER☐ MANUFACTURER☐ OTHER (Specify): _____

b. ADDRESS (Street, City, State, ZIP Code)

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 Code) - Optional

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