

**O-N-335**

JANUARY 11, 1962

SUPERSEDING

Fed. Spec. O-S-61

May 27, 1930

(See par. 6.4)

## FEDERAL SPECIFICATION

**NICKEL SALTS, ELECTROPLATING**

*This specification was approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.*

**1. SCOPE AND CLASSIFICATION**

1.1 Scope. This specification covers electroplating nickel salts.

**1.2 Classification.**

1.2.1 *Types.* Nickel salts shall be of the following types, as specified (see 6.2):

Type I—Nickel sulphate ("single salt").

Type II—Nickel ammonium sulphate ("double salt").

Type III—Nickel chloride.

**2. APPLICABLE SPECIFICATIONS AND STANDARDS**

2.1 The following specifications and standards, of the issues in effect on date of invitation for bids, form a part of this specification to the extent specified herein:

*Federal Specifications:*

U-M-186—Medicinal Products and Clinical-Laboratory-Reagents: General Specification for Containers (Packaging and Packing).

UU-S-48—Sacks, Shipping, Paper.

PPP-D-723—Drums, Fiber.

*Federal Standards:*

Fed. Std. No. 102—Preservation, Packaging and Packing Levels.

Fed. Std. No. 123—Marking for Domestic Shipment (Civilian Agencies).

(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards, and Handbooks as outlined under General Information in the Index of Federal Specifications, Standards, and Handbooks and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C.)

(Single copies of this specification and other product specifications required by activities outside the Federal Government for bidding purposes are available without charge at the General Services Administration Regional Offices in Boston, New York, Atlanta, Chicago, Kansas City, Mo., Dallas, Denver, San Francisco, Los Angeles, Seattle, and Washington, D.C.)

(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications, Standards, and Handbooks established distribution points in their agencies.)

*Military Standards:*

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 (40" x 48" 4-Way Partial and 4-Way Pallets).

(Copies of Military specifications and standards required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

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### 3. REQUIREMENTS

**3.1 Material.** Material shall be as specified herein. The salts shall not contain more than 0.02 percent iron, 0.025 percent zinc, 0.005 percent copper, 0.10 percent of free acid, or 0.05 percent insoluble matter.

**3.2 Type I.** Type I nickel sulphate shall be as follows:

$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ , or a mixture of the above with  $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ .

Nickel sulphate shall contain not less than 21.4 percent of nickel (plus cobalt.)

**3.3 Type II.** Type II nickel ammonium sulphate shall be as follows:

$\text{NiSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ .

Nickel ammonium sulphate shall contain not less than 14.6 percent of nickel (plus cobalt).

**3.4 Type III.** Type III nickel chloride shall be as follows:

$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ .

Nickel chloride shall contain not less than 24.5 percent of nickel (plus cobalt).

**3.5 Workmanship.** The workmanship shall be first class in every respect.

### 4. SAMPLING, INSPECTION AND TEST PROCEDURES

**4.1** The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examination and tests shall be kept complete and available to the Government as specified in the contract or order. 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.

**4.2.1 Lot.** For purposes of sampling a lot

shall consist of all nickel salts of the same type manufactured as one batch.

**4.2.2 Examination of filled containers.** A random sample of filled containers shall be selected from each lot in accordance with MIL-STD-105 at inspection level I, and acceptable quality level (AQL) = 2.5 percent defective to verify compliance with all stipulations of this specification regarding fill, closure, packaging, packing, marking and other requirements not involving tests. Containers shall be examined for defects of the container and the closure, for evidence of leakage, and for unsatisfactory markings; each sample filled container shall also be weighed to determine the amount of the contents. 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 for the appropriate sampling plan of MIL-STD-105, the lot represented by the sample shall be rejected.

**4.2.3 Acceptance tests.** From each lot, two containers shall be taken at random. From each of the two containers eight-ounce specimens shall be taken and placed in separate clean, dry metal or glass containers, sealed, marked and forwarded to a testing laboratory satisfactory to the bureau or agency concerned. The sample specimens shall be subjected separately to the tests specified in 4.3. If either specimen fails to pass one or more of these tests, the lot shall be rejected.

**4.3 Test procedures.** Any recognized and appropriate chemical or spectro-chemical methods may be substituted for the analytical procedures specified in 4.3.2, 4.3.3, 4.3.4, and 4.3.5. In case of dispute, the methods specified in 4.3.2, 4.3.3, 4.3.4 and 4.3.5 shall be the standard referee tests.

**4.3.1 Insoluble matter.** Dissolve 25 grams of the salt in about 125 cubic centimeters of distilled water and add 2.5 cubic centimeters of normal (N) hydrochloric acid. Heat the solution to boiling, filter through a weighed Gooch crucible or glass fritted crucible, and

wash the precipitate thoroughly with hot water. Dry the crucible at 110 degrees centigrade (C.) and reweigh. Compute the content of insoluble matter from the gain in weight.

**4.3.2 Copper and zinc (see 6.3).** Saturate with hydrogen sulphide a 200 cubic centimeter portion of the solution from the graduated flask (representing 20.0 grams of the salt in a solution that is 0.01 N in free acid). Filter out the precipitate which contains the sulphides of copper, zinc, and lead if these are present, together with some nickel sulphide. Wash it with water containing hydrogen sulphide. Dissolve the precipitate and filter paper by heating with 5 cubic centimeters of concentrated sulphuric acid and successive small additions of nitric acid. Expel the excess of nitric acid by evaporating to fumes of sulphuric acid. Dilute the residue to about 50 cubic centimeters and filter out any lead sulphate that may be precipitated if lead is present. To the filtrate from the lead sulphate add 1 cubic centimeter of concentrated nitric acid and deposit the copper electrolytically. Compute the copper content from the increase in weight of the cathode. (In this electrolysis any lead that was not precipitated as sulphate, will be deposited as peroxide upon the anode. In the absence of definite knowledge regarding the effects of small amounts of lead in nickel plating baths, no maximum content is fixed by this specification. It is necessary, however, to completely remove lead if it is present in order to determine the zinc.) Evaporate the solution from which the copper was removed until fumes of sulphuric acid appear, dilute to about 50 cubic centimeters, and exactly neutralize with ammonium hydroxide, using methyl red indicator. Then add 1 cubic centimeter of normal sulphuric acid and dilute the solution to 100 cubic centimeters and saturate with hydrogen sulphide. Filter out the precipitate of zinc sulphide, wash it with hydrogen sulphide water, and ignite at as low a temperature as possible till all carbon is burned off, and then at 900° C. Weigh as zinc oxide. The factor for ZnO to Zn is 0.80.

**4.3.3 Nickel (plus cobalt).** Cool the filtrate and washings from (4.5.1) and dilute to exactly 250 cubic centimeters in a graduated flask. Dilute a 20.0 cubic centimeter portion of the solution (representing 2.0 grams of the salt) to 100 cubic centimeters, and add 25 cubic centimeters of strong ammonium hydroxide (specific gravity 0.90). Filter out any ferric hydroxide that may be precipitated; electrolyze the filtrate with platinum electrodes until at least 15 minutes after the blue color has disappeared. Then introduce about five drops of the solution into a small test tube and carefully heat it to boiling in order to expel most of the free ammonia. Then add a few drops of a 1 percent alcoholic solution of dimethylglyoxime. If a pink color appears continue the electrolysis until on repeating this test, no nickel is detected. Dry and weigh the cathode and compute the content of nickel plus cobalt from the increase in weight. (*Note.*—On any acceptable salt, with a copper content of not more than 0.02 percent, no significant error is introduced by including this copper in the weight of the nickel.)

**4.3.4 Iron.** Boil the filtrate from the sulphides of copper, lead, and zinc, to expel hydrogen sulphide, and add a few drops of nitric acid to oxidize the iron. Add an excess of ammonium hydroxide and boil and filter the solution. After washing the precipitate with hot water, ignite it and weigh as  $\text{Fe}_2\text{O}_3$ . The factor for  $\text{Fe}_2\text{O}_3$  to Fe is 0.70. If the weight of iron thus computed is above the specified maximum of 0.08 percent, fuse the precipitate with potassium bisulphate, dissolve, reduce with zinc and titrate with standardized permanganate to determine the actual iron content. (This procedure will correct for any aluminum, and also for nickel that might be included with the iron when the latter is subjected to only one precipitation.)

**4.3.5 Free acid.** Dissolve a 20-gram sample of the original salt in 100 cubic centimeters of distilled water and add a few drops of methyl red or brom cresol purple solution. If the solution acquires an acid color, titrate

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it with 0.1 *N* sodium hydroxide to a neutral color (corresponding to a pH of approximately 6). For nickel sulphate and nickel ammonium sulphate compute the free acid as  $H_2SO_4$ ; for nickel chloride compute it as  $HCl$ .

### 5. PREPARATION FOR DELIVERY

(For civil agency procurement the definitions and applications of the levels of packaging and packing shall be in accordance with Fed. Std. No. 102.)

#### 5.1 Packaging.

5.1.1 *Level A*. Nickel salts shall be furnished in quantities specified (see 6.2) in containers as follows:

5.1.1.1 *Bottles*. Bottles shall conform to type I, class A of U-M-186. Bottle closure shall be as specified in U-M-186.

5.1.1.2 *Drums*. Fiber drums shall conform to type II or III, grade A of PPP-D-723. Drums shall be provided with a barrier lining which will not affect or be affected by the product packaged.

5.1.1.3 *Sacks*. Sacks shall conform to construction number 2X of UU-S-48.

5.1.2 *Level C*. Nickel salts shall be preserved and packaged in such a manner that will afford adequate protection against corrosion, deterioration and physical damage during shipment from supply source to the first receiving activity for immediate use. This may conform to the supplier's commercial practice, when such meets the requirements of this level.

#### 5.2 Packing.

##### 5.2.1 *Level A*.

5.2.1.1 *Bottles*. Bottles shall be packed for export shipment in accordance with U-M-186.

5.2.1.2 *Drums and sacks*. Drums and sacks

will require no additional packing. When specified (see 6.2) containers shall be palletized.

##### 5.2.2 *Level B*.

5.2.2.1 *Bottles*. Bottles shall be packed for domestic shipment in accordance with U-M-186.

5.2.2.2 *Drums and sacks*. Drums and sacks will require no additional packing. When specified (see 6.2) containers shall be palletized.

5.2.3 *Level C*. Nickel salts shall be packed in containers in a manner which will insure acceptance by common carrier and safe delivery at destination. Shipping containers or method of shipment shall conform to the carrier rules or regulations as applicable to the mode of transportation.

5.3 *Pallets*. When specified (see 6.2) drums and sacks shall be palletized in accordance with MIL-STD-147.

#### 5.4 Marking.

5.4.1 *Civil agencies*. Marking shall be in accordance with Fed. Std. No. 123.

5.4.2 *Military agencies*. In addition to any special marking required by the contract or order, unit and interior packages, exterior shipping containers and palletized unit loads shall be marked in accordance with MIL-STD-129.

### 6. NOTES

6.1 *Intended use*. Nickel salts covered by this specification is intended for use in electroplating.

6.2 *Ordering data*. Procurement documents should specify the following:

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

(b) Type of nickel salts required (see 1.2)

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(c) Type of container and selection of applicable levels of packaging and packing (see 5.1 and 5.2).

(d) When pallets are required (see 5.2 and 5.3).

6.3 The copper and zinc analyses should be known before initiating the nickel analysis, since they will deposit and cause an error if present in significant amounts. If copper and zinc are present in significant quantity they should be removed by proper  $H_2S$  precipitation prior to the electrolytic nickel determination.

6.4 This specification covers material formerly covered by Military Specification MIL-N-21967 (SHIPS), dated May 21, 1959.

6.5 Transportation description. Transportation descriptions and minimum weights applicable to this commodity are:

*Type I*

Rail: Nickel sulphate.  
Carload minimum weight  
36,000 pounds.  
Motor: Nickel sulphate.  
Motor volume minimum weight  
36,000 pounds.

*Type II*

Rail: Nickel ammonium sulphate.  
Carload minimum weight 36,000 pounds.

Motor: Nickel ammonium sulphate.  
Motor volume minimum weight  
36,000 pounds.

*Type III*

Rail: Chemicals, not otherwise indexed by name.  
Carload minimum weight 24,000 pounds, subject to Rule 34, Uniform Freight Classification.

Motor: Nickel chloride.  
Truckload minimum weight (W) 30.6 pounds, (W) subject to Rule 34, National Motor Freight Classification.

Notice: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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