

MIL-R-21931A(OS)
5 January 1977
SUPERSEDING
(See section 6)

MILITARY SPECIFICATION

RESIN, EPOXY

This specification is approved for use by the Naval Sea Systems Command, Department of the Navy, and is available for use by all departments and agencies of the Department of Defense.

1. SCOPE

1.1 This specification covers one type of thermosetting epoxy resin compound used in ordnance applications.

2. APPLICABLE DOCUMENTS

* 2.1 Issues of documents. The following documents of the issue in effect on the date of invitation for bids or request for proposals form a part of this specification to the extent specified herein. In the event of conflict between this specification and other documents referenced herein, the requirements of this specification shall apply.

SPECIFICATIONS

Federal

PPP-C-96	Cans, Metal, 28 Gage and lighter
PPP-D-729	Drums, Shipping and Storage, Steel, 55-Gallon
PPP-D-760	Drums and Pails, Metal (5 and 16.64 Gallon)

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Naval Ordnance Station, Attn: Standardization Division (611), Indian Head, Md. 20640, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC-9330

MIL-R-21931A(OS)

STANDARDS

Federal

FED-STD-406

Plastics, Organic; General Specifications,
Test Methods

Military

MIL-STD-129

Marking for Shipment and Storage

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

* 2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on the date of invitation for bids or request for proposals shall apply. In the event of conflict between this specification and other documents referenced herein, the requirements of this specification shall apply.

American Society for Testing and Materials (ASTM)

ASTM D 256

Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials

ASTM D 621

Method of Test for Deformation of Plastics Under Load

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa. 19103.)

3. REQUIREMENTS

3.1 Materials. The material shall be a thermosetting epoxy resin. The ingredients of the catalyzed resin shall be such that the cured resin shall not be corrosive to metals.

3.1.1 Uncatalyzed resin. The uncatalyzed epoxy resin shall have the physical properties listed in table I.

TABLE I. Physical properties of uncatalyzed liquid resin.

Property	Test method	Values	
		Min.	Max.
Specific gravity	4.3.1.1	1.15	1.25
Viscosity, centipoises	4.3.2	7,000	20,000
Refractive index	4.3.3	1.560	1.590
Epoxy number	4.3.4	165	200

3.1.2 Catalyzed and cured resin. The resin, when cured in accordance with 4.3.1, shall have the physical properties listed in table II.

TABLE II. Physical properties of cured resin.

Property	Test method	Values	
		Min.	Max.
Specific gravity	4.3.1.2	1.20	1.30
Tensile strength, psi	4.3.5	9,000	----
Impact strength, ft-lb/inch	4.3.6	0.40	----
Hardness, Rockwell M	4.3.7	100	120
Deformation under load, %	4.3.8	----	0.5
Water absorption, %	4.3.9	----	0.2
Peak exotherm in 6 hours, °F	4.3.10.1	----	25
Gel time, hours	4.3.10.2	6	----
Volume resistivity, ohm cm	4.3.11	5×10^{15}	----

3.2 Instruction sheet. The manufacturer shall provide, with each resin submitted for approval, two copies of an approved instruction sheet containing the following:

- a. Maximum usable storage life of the uncatalyzed resin.
- b. Any other pertinent information on storage handling of the resin such as most suitable containers and precaution to be taken in handling resin and catalyst.

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 order, the contractor may use his

MIL-R-21931A(06)

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 Quality conformance inspection.** Quality conformance inspection shall be performed on each lot of resin submitted for acceptance and shall consist of all the examinations and tests as specified herein. Inspection and test procedures shall be submitted for Government approval prior to commencement of production. Failure of the resin to meet the requirements of this specification shall be cause for the rejection of the lot represented.

* **4.2.1 Lot formation.** For the purpose of sampling, a lot shall consist of an integral quantity of the material produced in one manufacturing cycle. A manufacturing cycle shall be limited to 30,000 pounds of resin.

* **4.2.2 Sampling.** For purposes of sampling a lot or shipment in the form of package lots (cans, drums, barrels, or boxes) shall be limited to 30,000 pounds of resin. For each lot the number of individual packages to be selected at random shall be as specified in table III. The composite weight of the uncatalyzed resin removed from the selected shipping containers shall be at least 10 percent greater than that required to fulfill total test specimen needs of 4.3. The total weight is calculated from the resin density: weight equals total specimen volume times density. Withdraw equal weight portions from each selected container such that the total weight removed will meet the composite test specimen weight requirements. All of the resin required for the tests of 4.3 shall be taken from the composite sample which is representative of the lot.

TABLE III: Minimum number of packages to be selected for sampling.

Number of packages in the lot	Number of packages to be sampled
1 to 3	all
4 to 64	4
65 to 125	5
126 to 216	6
217 to 343	7
344 to 512	8

4.2.3 Test conditions. Standard test conditions shall be $23^{\circ} \pm 1^{\circ} \text{C}$ ($73^{\circ} \pm 2^{\circ} \text{F}$) and 50 ± 4 percent relative humidity. Specimens shall be tested after being exposed to this temperature and humidity for 2 days.

* 4.2.4 Preparation of test specimens and peak exotherm and gel time tests. Weigh to the nearest gram approximately 1400 grams of uncatalyzed resin and heat to $160^{\circ} \pm 2^{\circ} \text{F}$. Heat, in a ventilated hood, a stoichiometric amount of purified metaphenylenediamine (having a melting point of 145°F) to $150^{\circ} \pm 2^{\circ} \text{F}$. The stoichiometric amount of metaphenylenediamine is derived from the epoxy number (see 4.3.4), and quantitatively utilized in accordance with table IV. Into the melted metaphenylenediamine insert a variable speed, mechanical stirrer, and add the heated uncatalyzed resin. After the uncatalyzed resin has been added continue stirring for 90 ± 30 seconds at a speed which will minimize air entrapment. Immediately place the container with the hot resin solution into a room temperature water bath and continue stirring until the solution temperature is lowered to 105°F . Pour part of the cooled solution into a 12- by 4- by 1-inch-deep metal pan to a depth of 0.75 inch. The remainder of the solution is set aside at room temperature to be used later for the peak exotherm and gel time test (see 4.3.10) and the volume resistivity test (see 4.3.11). Allow the solution in the pan to cure for 24 hours at standard laboratory (ambient temperature) condition. Post cure for 4 hours at $194^{\circ} \pm 2^{\circ} \text{F}$. After post curing, cool the casting to room temperature, machine desired test specimens as prescribed in 4.3.1.2, 4.3.5 through 4.3.9, and 4.3.11, and condition test specimens as prescribed in 4.2.3.

4.3 Test procedures.

4.3.1 Specific gravity.

4.3.1.1 Uncatalyzed resin. The specific gravity of the uncatalyzed liquid resin shall be measured with an accurate balance. The average of three determinations shall be recorded. The specific gravity shall be expressed as the ratio of the weight of a given volume of the resin at 25°C to that of an equal volume of water at the same temperature.

* 4.3.1.2 Cast resin. The specific gravity of the cast resin shall be determined by method 5011 of FED-STD-406, on three specimens. The average of the three determinations shall be reported.

4.3.2 Viscosity. The viscosity in centipoises of the uncatalyzed resin shall be determined with an accurate viscosimeter.

MIL-R-21931A(OS)

TABLE IV. Stoichiometric amount of metaphenylenediamine at determined epoxy number required for quantity of uncatalyzed resin.

Epoxy number	Number of grams of metaphenylenediamine per 100 grams of uncatalyzed resin
165	16.39
167	16.19
169	16.00
171	15.81
173	15.63
175	15.45
177	15.28
179	15.11
181	14.94
183	14.78
185	14.62
187	14.46
189	14.31
191	14.16
193	14.01
195	13.87
197	13.72
199	13.59

4.3.3 Refractive index. The refractive index of the uncatalyzed resin shall be determined by a refractometer.

4.3.4 Epoxy number. The epoxy number of the uncatalyzed resin is the number of grams of resin containing one epoxy equivalent. It shall be determined as follows:

a. In a 125-milliliter, ground-glass stoppered Erlenmeyer flask dissolve 0.5000 gram of uncatalyzed resin in 25.00 milliliters of hydrochloric acid-p-dioxane reagent. This reagent shall be prepared by pipetting 4.00 milliliters of reagent grade concentrated hydrochloric acid into a 250-milliliter volumetric flask and filling to the mark with p-dioxane having a melting point of 11.5° C.

b. The resin solution as prepared above shall be allowed to stand for 20 minutes, with occasional shaking.

c. After standing, wash into a 250-milliliter beaker with 50 milliliters of distilled water followed with 40 milliliters of acetone.

d. To the wash add 5 milliliters of reagent grade nitric acid and 0.2 gram of gelatin.

e. With continuous stirring, the mixture in the beaker shall be titrated with 0.1000 normal silver nitrate solution, using a calomel (saturated)-silver electrode system (see figure 1) containing a 0.05-molar ammonium nitrate bridge, to detect the equivalence point potentiometrically. Reagent grade silver nitrate shall be used. The end point of the titration shall be determined by adding the silver nitrate dropwise near the equivalence point and choosing the volume where the greatest change in potential occurs.

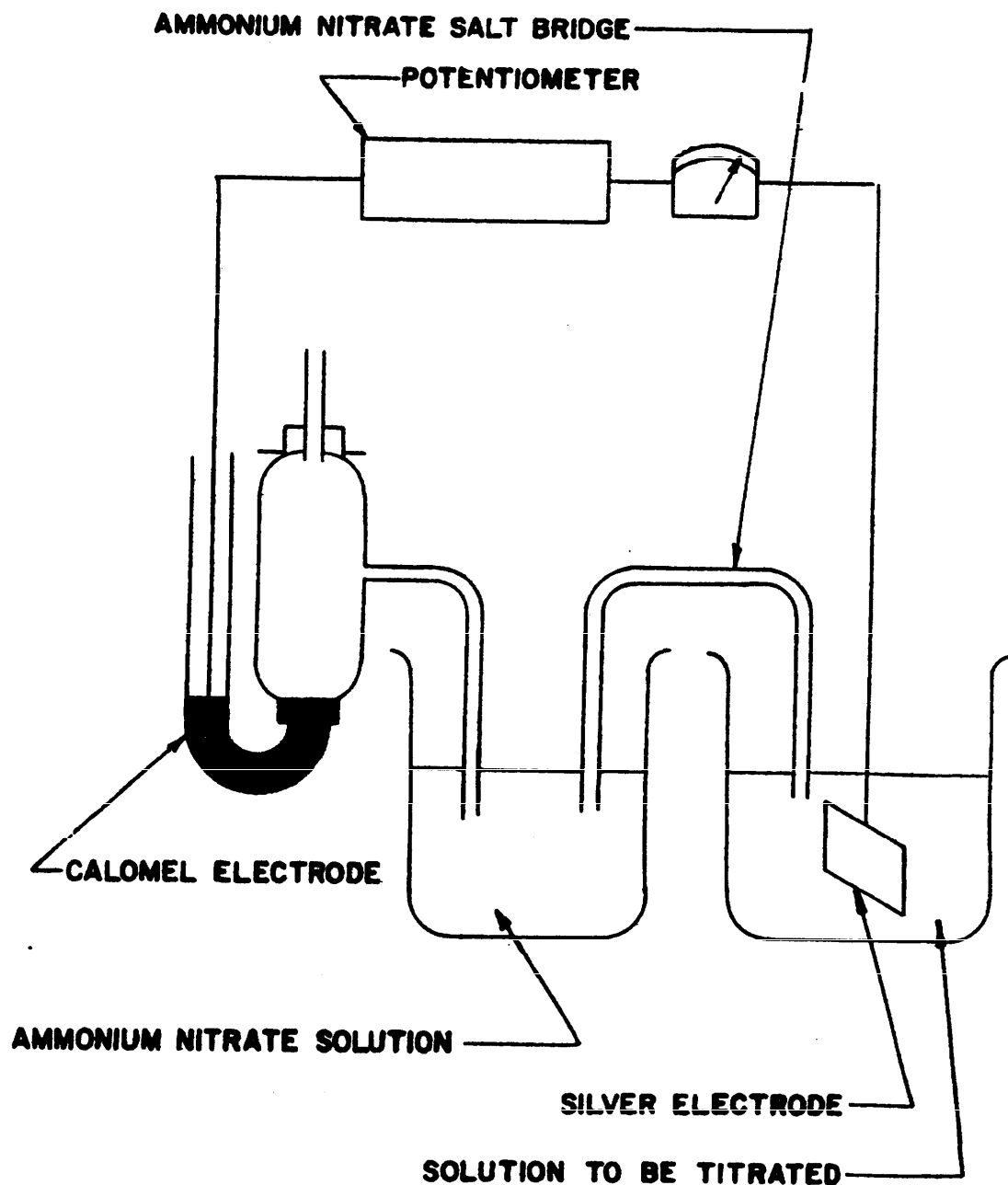


FIGURE 1. Titration assembly.

NIL-R-21931A(OS)

f. The epoxy number shall be calculated as follows:

$$\text{Epoxy No.} = \frac{\text{weight of resin sample} \times 100}{0.0001(\text{ml of } 0.1\text{N AgNO}_3 \text{ for blank}) - (\text{ml of AgNO}_3 \text{ for (HCL)})}$$

* 4.3.5 Tensile strength. The tensile strength of the catalyzed and cured resin shall be determined by method 1011 of FED-STD-406 on test specimens constructed in accordance with figure 2. The rate of head travel for the tensile testing machine shall be not greater than 0.05 inch per minute while extensometer readings are made. The average of five determinations shall be recorded.

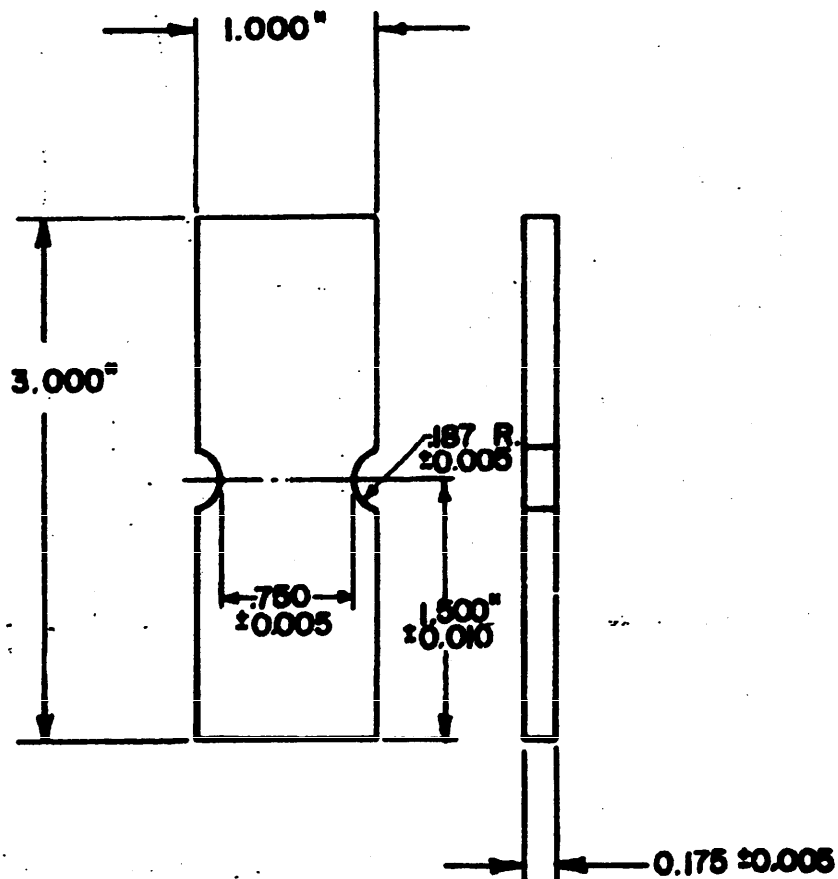


FIGURE 2. Tensile test specimen.

4.3.6 Impact strength. The impact strength of the cured resin shall be determined in accordance with ASTM D 256 on 2.5- by 0.5- by 0.25-inch specimens. The average of five determinations shall be recorded.

* 4.3.7 Hardness. Rockwell M hardness on the cured specimens shall be determined as prescribed by method 1081 of FED-STD-406. The average of four readings per casting shall be reported.

4.3.8 Deformation under load. Deformation under a 2,000-pound-per-square-inch (psi) load and $160^{\circ} \pm 2^{\circ}$ F of a 0.5-inch-cube cured resin specimen shall be determined as prescribed in ASTM D 621. The average of two measurements shall be recorded.

* 4.3.9 Water absorption. Water absorption of the cured resin shall be determined as prescribed in method 7031 of FED-STD-406. The average of three measurements shall be recorded.

4.3.10 Peak exotherm and gel time. The same sample shall be used for the peak exotherm and the gel time measurements.

4.3.10.1 Peak exotherm. The peak exotherm shall be determined as follows: 300 ± 5 grams of the catalyzed resin as prepared in 4.2.4, and cooled to 105° F, shall be poured into a standard number 1 (211×4.00) can immediately. A thermocouple shall be mounted securely and oriented in the exact center, vertically and horizontally of the liquid resin. The temperature of the reaction mixture shall be measured periodically every 5 to 15 minutes over a 6-hour period. The peak exotherm shall be reported as the maximum temperature rise above 105° F. The ambient room temperature shall be $75^{\circ} \pm 2^{\circ}$ F.

4.3.10.2 Gel time. The gel time shall be reported as the time required for the sample in 4.3.10.1 to become stringy as determined by the lowering and raising of a thin wire into the reaction mixture.

MIL-R-21931A(OS)

4.3.11 Volume resistivity. The volume resistivity measurement is made as follows:

4.3.11.1 Apparatus.

- a. Strip electrode assembly (see figure 3)
- b. Top cover plate (see figure 4A)
- c. Strip electrode spacer (see figure 4B)
- d. Standard number 1 can
- e. Keithley model 2008 shunt attachment (see figure 5)
- f. 45-volt standard battery.

4.3.11.2 Monitoring device (fabrication and assembling).

- a. The strip electrode assembly shall be fabricated as specified in figure 3 and shall consist of two brass strip electrodes, two Teflon strip electrodes, two Teflon strip electrode spacers (see figure 4B), and a lucite or polystyrene top cover plate (see figure 4A).
- b. The strip electrode assembly is placed in the standard number 1 can with the cover plate being supported by the top of the can, and two terminals connected to the current monitoring device as shown in figure 5. The overall monitoring device consists of a Keithley model 2008 decade shunt attachment and a 45-volt battery.

4.3.11.3 Resin preparation. The catalyzed resin, as prepared in 4.2.4 and cooled to 105° F shall be poured into the number 1 can to a depth of at least 1 centimeter above the electrode strips. The catalyzed resin shall be cured for 24 hours at standard conditions. Postcure the casting in the can by reheating to a temperature of 194° ± 2° F throughout the resin and holding this temperature for 4 hours. Cool the resin to standard condition before testing.

4.3.11.4 Procedure. The monitoring device shall be turned on and allowed to warm up for about one-half hour. The resistivity shall be measured at a field strength of approximately 200 volts per centimeter. The voltage drop across the sample and the current in amperes shall be read directly from the electrometer and the volume resistivity, P, calculated from the following equation:

$$P = \frac{A(E_b - E_d)}{LI}$$

MIL-R-21931A(OS)

where:

- A = surface area in cm^2 of the electrode
- L = current path length in centimeters of the material
- A/L = 86 for electrode described
- E_b = battery voltage
- E_d = voltage drop across the sample
- I = current in amperes.

* 4.4 Examination of packaging. The packaging of the resin shall be examined for conformance to the requirements of section 5.

4.5 Resubmission and retest. Resin which has been rejected may be reworked or replaced to correct the defects and resubmitted for acceptance. Before resubmitting, full particulars concerning previous rejection and the action taken to correct the defects found in the original shall be furnished to the procuring activity. Resin rejected after retest shall not be resubmitted without the specific approval of the procuring activity.

* 5. PACKAGING

* 5.1 Packaging and packing. Packaging and packing shall be level A or C, as specified in the contract (see 6.2).

5.1.1 Level A. Unless otherwise specified, the uncatalyzed epoxy resin shall be packaged or packed in 5-pound, 5-gallon, 120-pound steel containers, or 55-gallon steel open head drums as designated by the procuring activity. Five-pound containers shall conform to type V, class 2, of PPP-C-96 and shall be packed for the level of shipment in accordance with the appendix thereto. When containers are manufactured from electrolytic tin plate, the containers shall be made from 0.50-pound material. Interior and plan B exterior coatings shall be required. Five-gallon and 120-pound steel containers shall conform to type II (class 2, 5-gallon lug cover pails) and type III, class I (120-pound (16.64-gallon) lug cover drums) of PPP-D-760, respectively. Packing for shipments will not be required. Fifty-five gallon drums shall conform to type III or IV of PPP-D-729 and shall have a full removable head with bolted ring or level locking devices. Drums shall not require overpacking for shipment.

MIL-R-21931A(OS)

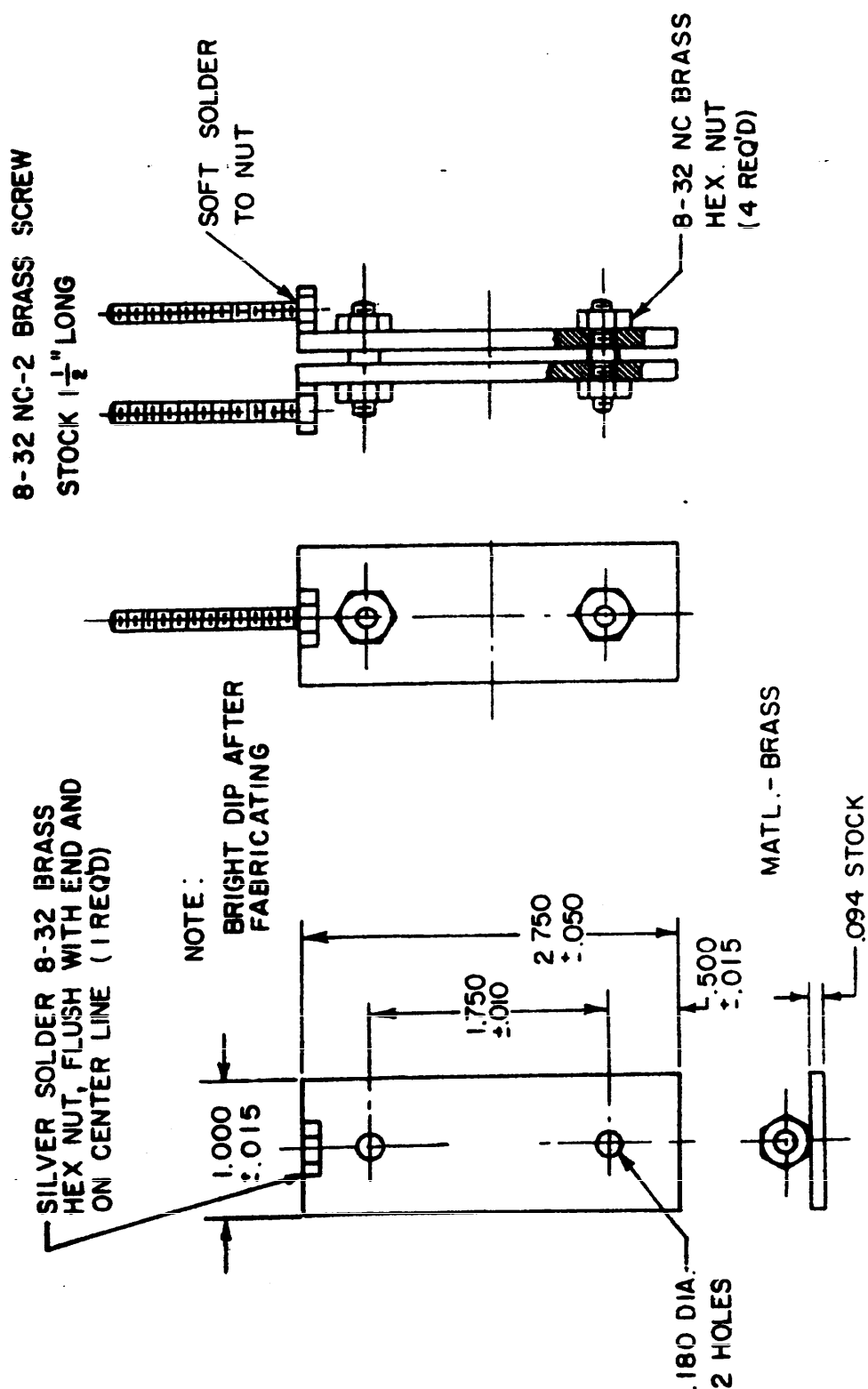


FIGURE 3. Strip electrode and strip electrode assembly.

MIL-R-21931A(OS)

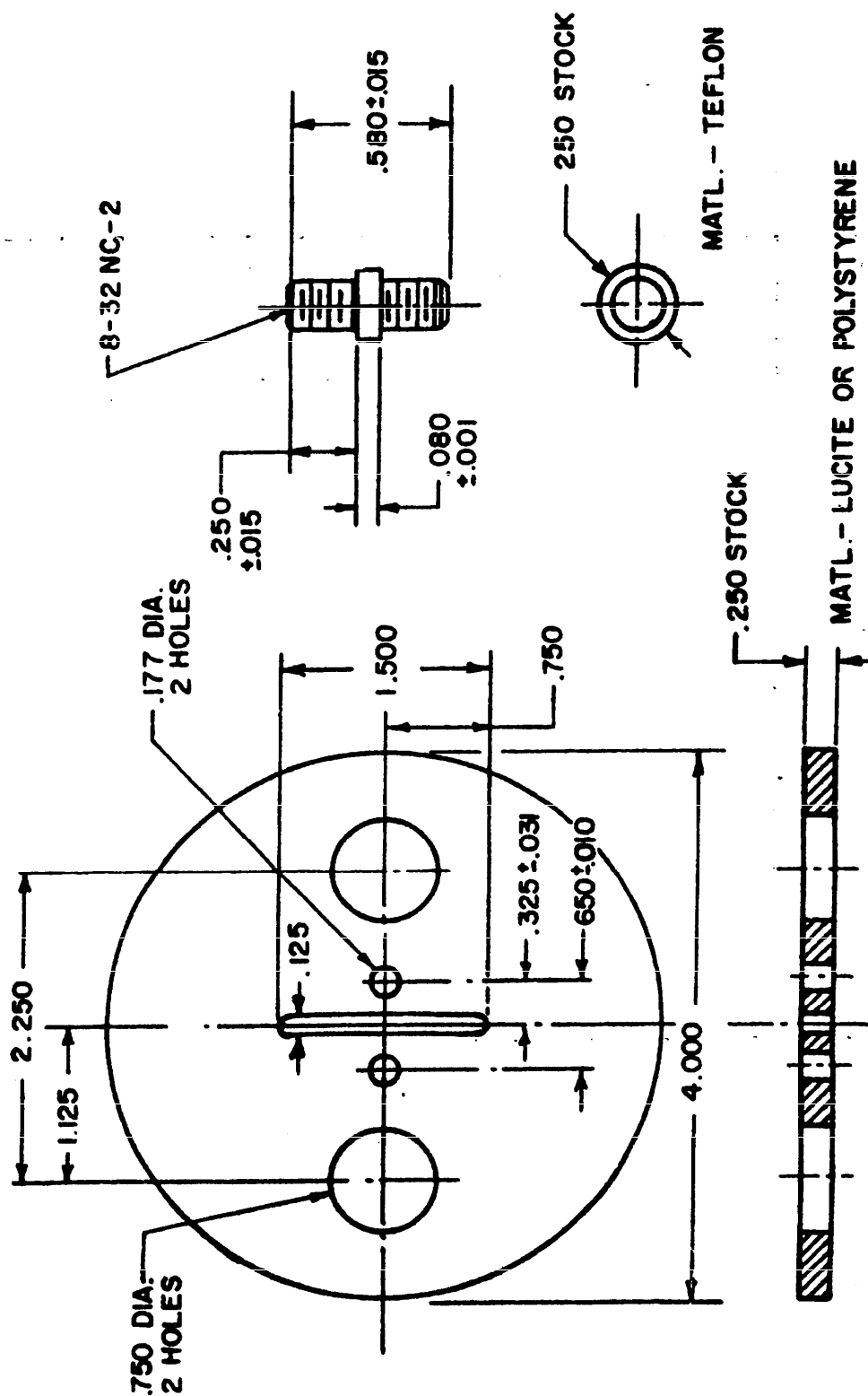
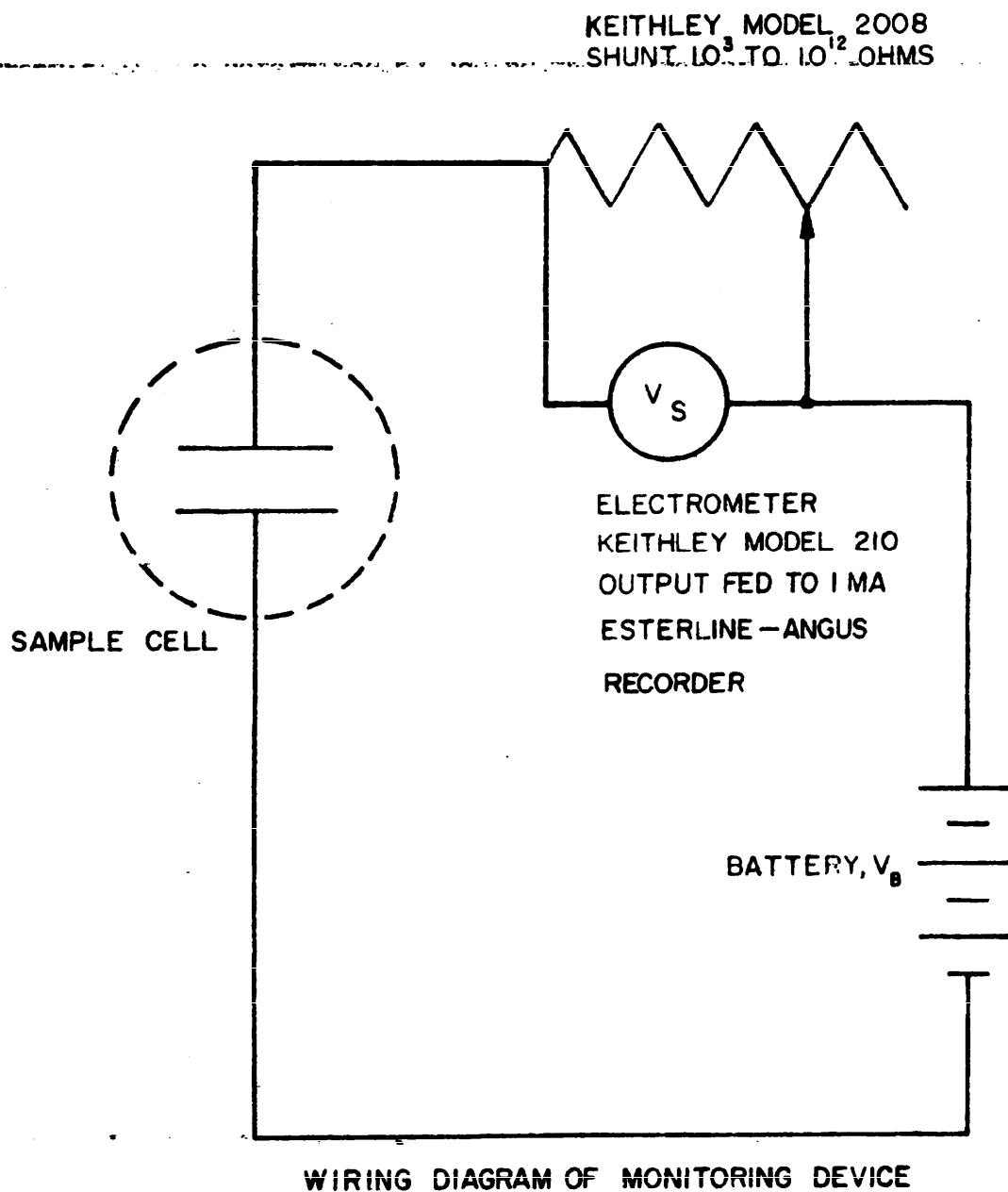


FIGURE 4A. Top cover plate.

FIGURE 4B. Strip electrode spacer.

MIL-R-21931A(OS)

FIGURE 5. Wiring diagram of monitoring device.

MIL-R-21931A(OS)

* 5.1.2 Level C. The epoxy resin shall be packed in a manner which will provide protection from damage during direct shipment from the contractor or supply source to the first receiving activity for immediate use. The type of containers used shall conform to applicable carrier rules and regulations.

5.2 Marking. In addition to any special marking required by the contract or order, all marking shall be in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. This epoxy resin is intended for use in ordnance applications.

6.2 Ordering data. Procurement documents should specify the following:

- a. Title, number, and date of this specification
- b. Lot and code number
- c. Quantity required
- d. Size of container
- e. Level of packaging and packing.

* 6.3 Supersession data. This specification includes the requirements of MIL-R-21931(Wep), dated 17 February 1959, and NAVORD OS 8095, dated 2 October 1957.

* 6.4 The margins of this specification are marked with asterisks to indicate where changes (additions, modifications, corrections, deletions) from the previous issue were made. This was done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in these notations. Bidders and contractors are cautioned to evaluate the requirements of this document based on the entire content irrespective of the marginal notations and relationship to the last previous issue.

Review activity:

ARMY - EA

Preparing activity:

NAVY - OS
(Project No. 9330-N773)

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL		OMB Approval No. 22-R255
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DOCUMENT IDENTIFIER AND TITLE		
MIL-R-21931 Resin, Epoxy		
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1. HAS ANY PART OF THE DOCUMENT CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?		
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