

MIL-L-758A

25 MAY 1962

SUPERSEDING

JAN-L-758

15 APRIL 1949

MILITARY SPECIFICATION

LEAD STEARATE

(FOR USE IN AMMUNITION)

This specification has been approved by the Department of Defense and is mandatory for use of the Departments of the Army, the Navy, and the Air Force.

1. SCOPE

1.1 This specification covers one grade of lead stearate intended for use in propellant powder.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids form a part of this specification to the extent specified herein.

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JAN-P-112

— Packaging and Packing for Overseas Shipment Drums, Plywood (for drums whose weight of contents does not exceed 200 pounds).

STANDARDS

SPECIFICATIONS

FEDERAL

UU-S-48 — Sacks, Shipping, Paper.

RR-S-366 — Sieves, Standard for Testing Purposes

PPP-D-723 — Drums, Fiber.

PPP-D-729 — Drums, Metal, 55 Gallon (for shipment of noncorrosive material).

FEDERAL

FED-STD-141 — Paint, Varnish, Lacquer, and Related Materials, Methods of Inspection, Sampling, and Testing.

MILITARY

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

FSC 6810

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MIL-STD-109 — Inspection Terms and Definitions.

MIL-STD-129 — Marking for Shipment and Storage.

PUBLICATIONS**ORDNANCE CORPS**

ORD-M608-11 — Procedures and Tables for Continuous Sampling by Attributes.

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

2.2 The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

UNIFORM FREIGHT CLASSIFICATION RULES

(Application for copies of these freight classification rules should be addressed to the Consolidated Classification Committee, 202 Chicago Union Station, Chicago 6, Ill.)

AMERICAN TRUCKING ASSOCIATION PUBLICATION

National Motor Freight Classification Rules and Container Specifications.

(Application for copies should be addressed to the American Trucking Association, 1424 16th Street N.W., Washington, D. C.)

3. REQUIREMENTS

3.1 The requirements for lead stearate shall comply with table I when determined as specified in the applicable paragraphs of section 4.3.

TABLE I

Tests	Requirements	Applicable paragraphs
Moisture percent, maximum (max.) percent	1.0	4.3.1
Water soluble material, percent, max.	0.5	4.3.2
Acidity and alkalinity		4.3.3
Alkalinity	None	4.3.3.1
Acidity		
(a) To phenolphthalein percent as acetic acid	0.05	4.3.3.2
(b) To methyl orange	None	4.3.3.3
Purity		4.3.5
(a) Lead content, percent	26.8 to 29.5	4.3.5.1
(b) Stearate as stearic acid, percent	70.8 to 73.5	4.3.5.2
Melting point of lead stearate		
degree Centigrade (*C.) minimum (min.)	104	4.3.4
Melting point of fatty acid acid *C., min.	55	4.3.5.2.1
Iodine number of fatty acid, max.	5.0	4.3.5.2.2

3.2 Granulation. Lead stearate shall conform to the following granulation requirements using the following U.S. Standard sieves as specified in paragraph 4.3.6.

	Percent, max.
1 Retained on number 100.....	5.0
2 Through number 100 and retained on number 200	13.0
3 Through number 200 and retained on number 325	15.0

3.3 Workmanship. The standard of workmanship shall be such as to insure the production of material meeting the requirements of this specification.

4. QUALITY ASSURANCE PROVISIONS

4.1 General quality assurance provisions. 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 examinations and tests shall be kept com-

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plete 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. Reference shall be made to Standard MIL-STD-109 in order to define the terms used herein. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.

4.1.1 Contractor quality assurance system. If the contractor desires to utilize a quality assurance system, which is at variance with the quality assurance provisions of 4.2 and 4.3 and other documents referenced herein, he shall submit a written description of the system to the contracting officer for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections, to be performed, inspection stations, sampling procedures, methods of inspection, (measuring and testing equipment), and provisions for control and disposition of non-conforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance provisions required by the provisions of 4.2 and 4.3 and the other documents referenced herein. The contractor shall not be restricted to the inspection station nor the method of inspection listed in this specification provided that an equivalent control is included in the approved quality assurance procedure. In cases of dispute as to whether certain procedures of the contractor's system provide equal assurance, the comparable procedure of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any changes to the assurance required by this specification or written procedure that effects the degree of other documents referenced herein.

4.1.2 Submission of product. At the time the completed lot of product is submitted to

the Government for acceptance, the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product submitted:

- (a) A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.
- (b) Quantity of product inspected.
- (c) Results obtained for all inspection performed.
- (d) Specification number and date, together with an identification and date of changes.
- (e) Certificates of analysis on all material procured directly by the contractor when such material is covered by referenced Government specifications.
- (f) Quantity of product in the lot.
- (g) Date submitted.

The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

4.1.3 Government verification. Using the contractor's written quality assurance procedure (see 4.1.1), this detail specification, and other contractual documents as a guide, the Government inspector shall verify all quality assurance operations performed by the contractor. Verification shall be in accordance with a or b as applicable, the decision being the responsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all

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quality assurance provisions of other specifications referenced in any of the contractual documents have been complied with. Deviations from prescribed or agreed upon procedures discovered by the Government inspector shall be brought to the attention of the supplier. Disposition of the product and remedial action shall be as directed by the Government inspector and, depending on the nature of the deviation, may consist of lot rejection, screening, re-sampling, re-instruction of the supplier's employees, or other appropriate action:

- (a) Verification at the point of manufacture shall be accomplished at unscheduled intervals in accordance with 4.1.3.1 and 4.1.3.2
- (b) Verification at the point of delivery shall be in accordance with 4.1.3.2.

4.1.3.1 *Surveillance*. Surveillance shall include, but is not limited to:

- (a) Observation of procedures concerning lot information and identification.
- (b) Observation of sampling procedures and application of acceptance criteria.
- (c) Determination that all required examinations and tests are performed in accordance with the procedures of this specification, or approved equivalents thereto.

- (d) Review of procedures for control and disposition of non-conforming material.

4.1.3.2 *Product inspection*. Product inspection shall consist of Government inspection of product which has been previously inspected by the contractor and found to meet the quality assurance provisions of this specification. The inspection by the Government shall be performed in order to determine that the product is of the quality required by this specification and that the contractor's records are reliable.

4.2 Inspection provisions.

4.2.1 *Lot formation*. A lot shall consist of one or more batches of lead stearate, produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each batch shall consist of that quantity of lead stearate that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogenous.

4.2.2 *Examination*. Sampling plans and procedures for the following classification of defects shall be in accordance with Standard MIL-STD-105. Continuous sampling plans, in accordance with Handbook ORD-M-608-11 may be used if approved by the procuring activity. Also, at the option of the procuring activity, AQL's and sampling plans may be applied to the individual characteristics listed using an AQL of 0.25 percent for each major defect and an AQL of 0.40 percent for each minor defect.

4.2.2.1 Drums, prior to filling (polyethylene liner).

<i>Categories</i>		<i>Defects</i>	<i>Method of inspection</i>
Critical:	None defined.		
Major:		AQL 0.25 percent	
101.	Liner cut, torn or punctured		Visual
Minor:	None defined.		

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4.2.2.1.1 Drums, prior to filling (alternate method).

<i>Categories</i>	<i>Defects</i>	<i>Method of inspection</i>
Critical:	None defined.	
Major:	AQL 0.25 percent	
101.	Laminations or coatings incomplete	Visual
Minor:	None defined.	

4.2.2.2 Drums, prior to closing (polyethylene liner).

<i>Categories</i>	<i>Defects</i>	<i>Method of inspection</i>
Critical:	None defined.	
Major:	AQL 0.25 percent	
101.	Liner not completely sealed	Visual
Minor:	AQL 0.40 percent	
201.	Markings misleading or unidentifiable	Visual

4.2.2.3 Drum, sealed (Steel).

<i>Categories</i>	<i>Defects</i>	<i>Method of inspection</i>
Major:	AQL 0.25 percent	
101.	Closing ring, damaged or not properly positioned so it does not close drum properly	Visual
Minor:	AQL 0.40 percent	
201.	Marking misleading or unidentifiable	Visual

4.2.2.4 Container, drum, sealed (fiber) or sack (paper).

<i>Categories</i>	<i>Defects</i>	<i>Method of inspection</i>
Critical:	None defined.	
Major:	AQL 0.25 percent	
101.	Weight of contents	Scale
102.	Closure incomplete or damaged to the extent that contents sifts out	Visual
Minor:	AQL 0.40 percent	
201.	Markings misleading or unidentifiable	Visual

4.2.2.5 Drum, sealed (plywood).

<i>Categories</i>	<i>Defects</i>	<i>Method of inspection</i>
Critical:	None defined.	
Major:	AQL 0.25 percent	
101.	Weight of contents	Scale
102.	Strapping missing or broken	Visual
Minor:	AQL 1.00 percent	
201.	Strapping mislocated	Visual
202.	Marking misleading or unidentifiable	Visual

4.2.3 Testing.

4.2.3.1 *Batch blending.* Where batches are blended to make a uniform lot, a random sample of 10 containers shall be selected from each lot. When lots are comprised of

10 containers, or less, each container shall be sampled.

4.2.3.2 *Sampling lot not batch blending.* When batches are not blended to make a uniform lot, a random sample of 10 cartons

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shall be selected from each batch. When a batch is comprised of 10 containers, or less, each container shall be sampled.

4.2.3.3 Preparation of composite. Primary sample of approximately two ounces of lead stearate shall be removed from each of the 10 containers in order to equal 20 ounces. If there are less than 10 containers, equal primary samples in sufficient quantity to equal 20 ounces, shall be removed from each container. The individual primary samples shall then be combined in order to form a homogeneous composite sample of 20 ounces and subjected to the tests specified in 4.3. If the composite sample batch or lot sample fails to comply with any of the requirements specified, the lot shall be rejected.

4.3 Test methods and procedures.

4.3.1 Moisture. Transfer a portion of about 10 gram (gm.) of the material to a tared glass or other suitable dish about 5 centimeters (cm.) in diameter and 2 cm. deep and accurately weigh the dish and contents. Place in an oven maintained at 95° to 100°C. for 2 hours, cool in a desiccator and weigh. Calculate the loss in weight as percent moisture as follows:

$$\text{Percent moisture} = \frac{A - B}{A - C} \times 100$$

where:

C = weight of empty dish, gm.

A = weight of dish and sample before drying, gm.

B = weight of dish and sample after drying, gm.

4.3.2 Water soluble material. Transfer an accurately weighed portion of about 5 gm. of the sample to a 250 milliliter (ml.) beaker. Thoroughly wet the sample with not more than 5 ml. of 95 percent ethyl alcohol and break up any lumps which remain with a stirring rod. Add 100 ml. of distilled water

to the beaker and stir until a uniform suspension of the lead stearate is obtained. Cover the beaker with a watch glass and boil the solution for 15 minutes. Bring the volume of the solution back to 95 to 100 ml. by adding distilled water and allow the beaker and contents to cool to 25 plus or minus 2°C. and filter using a Whatman No. 41, or equivalent, filter paper, catching the filtrate in a tared 150 beaker. Wash the filter paper and residue with two 25 ml. portions of water at 25° plus or minus 2°C. Evaporate the filtrate and washings to dryness, first using a hot plate until the volume is reduced to about 5 ml. and then using a steam bath to complete the evaporation. Dry the beaker and residue in an oven at 95° to 100°C. for one-half hour, cool in a desiccator and weigh. Calculate the increase in weight as percent water-soluble material as follows:

$$\text{Percent water-soluble material} = \frac{(A - B) \times 100}{C}$$

where:

C = weight of sample, gm.

B = weight of empty beaker, gm.

A = weight of beaker plus residue, gm.

4.3.3 Acidity and alkalinity.

4.3.3.1 Alkalinity. Transfer a weighed portion of about 20 gm. of the sample to a 400 ml. beaker and add 40 ml. of 95 percent ethyl alcohol previously neutralized to phenolphthalein. Stir the mixture and break up any lumps in the material with a stirring rod. Add 200 ml. of cold, freshly boiled distilled water to the beaker and stir the mixture vigorously for about 2 minutes. Filter the mixture, by means of a Buchner funnel, containing a No. 41 Whatman, or equivalent, filter paper. Wash the insoluble matter and filter paper with two 50 ml. portions of distilled water, make up the combined filtrate and washings to 500 ml. in a volumetric flask and then divide it into two equal portions. To the first portion add five drops of

phenolphthalein indicator and note the color of solution. Consider the alkalinity to be none if the solution does not turn pink on addition of phenolphthalein. If the solution does not turn pink on the addition of phenolphthalein, determine the acidity phenolphthalein as specified in 4.3.3.2.

4.3.3.2 Acidity to phenolphthalein. If the solution to which phenolphthalein has been added as specified in 4.3.3.1 remains colorless, titrate with 0.05 Normal (N) sodium or potassium hydroxide solution to a light pink end point. Calculate the percent acidity as follows:

$$\text{Percent acidity to phenolphthalein (as acetic acid)} = \frac{6.0 \text{ VN}}{W}$$

where:

V = ml. of 0.05 N sodium hydroxide or potassium hydroxide solution used.

N = normality of sodium or potassium hydroxide solution.

W = gm. of sample represented by the aliquot taken.

4.3.3.3 Acidity to methyl orange. To the second portion of the solution prepared as specified in 4.3.3.1 add a few drops of methyl orange indicator and note if acidity to methyl orange is absent as indicated by the solution turning yellow in color.

4.3.4 Melting point.

4.3.4.1 Apparatus. Set up a melting point bath equipped with a mechanical stirrer and a source of heat that can be easily regulated. A beaker of about 1 liter capacity about three-fourths full of clean colorless paraffin oil is recommended for the melting point bath. Suspend an accurately calibrated thermometer which includes the range of 90° to 120°C. and is graduated in 0.2°C. divisions

in the bath so that the bulb is not less than 1½ inches from the bottom of the bath. If the mercury column is not completely immersed at the temperature of the observed melting point, suspend another thermometer at about one-half inch away from the first thermometer with its bulb near the middle of the exposed column of the first thermometer.

4.3.4.2 Determination. Grind a small portion (about 2 gm.) of the material so that it will pass a number 200 U.S. Standard sieve, Fed. Spec. RR-S-336, and spread it over the bottom of a glass dish which is about 2 inches in diameter. Dry the material in an oven maintained at 70°C. for 2 hours or in a vacuum desiccator over sulfuric acid for at least 16 hours. Use a thin-walled capillary tube long enough to extend above the top of the bath, having an inside diameter of about 1 millimeter and sealed at one end. Fill the tube with the dried, ground sample to a depth of about 5mm., compacting the sample by tapping. Fasten the tube to the standardized thermometer so that the lower end of the tube is in contact with the bulb of the thermometer. Start the stirrer and heat the bath rapidly to about 15°C. below the prescribed melting point, then adjust the heat source so that the rise in temperature does not exceed 1°C. per minute. Observe and record the temperature at which the material in the tube first appears to become completely liquified. When a total immersion thermometer is used and part of the liquid column is not immersed, add the following correction to the observed melting point:

$$\text{Correction, } ^\circ\text{C.} = N(T - t) 0.00016$$

where:

N = number of degrees in the exposed mercury column.

T = uncorrected melting point.

t = average temperature of the exposed column as indicated by the second thermometer.

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4.3.5 Purity.

4.3.5.1 *Total lead content.* Transfer an accurately weighed sample of approximately 2 gm. of the material to a 250 ml. beaker. Thoroughly wet the sample with 95 percent ethyl alcohol and with a stirring rod break up any lumps which remain. Add to the beaker 100 ml. of distilled water and then 10 ml. of concentrated nitric acid. Stir the mixture for about 2 minutes and transfer to a 250-ml. separatory funnel with greaseless fittings, using a stream of cold distilled water to make certain that all solid matter is transferred to the funnel. To the separatory funnel, add 50 ml. of ethyl ether. Stopper the funnel and shake it vigorously for a few seconds, then invert the funnel and release the pressure which has been built up in the funnel by carefully opening the stopcock. Close the stopcock and again shake the funnel vigorously releasing the pressure as described herein. Continue shaking the funnel and releasing any pressure which builds up in the funnel until all the lead stearate in the mixture appears to have been dissolved. Place the funnel upright and allow the two layers to separate. Draw off the lower aqueous layer into a 400 ml. beaker, being careful not to allow any of the ether layer to run into the beaker. Add 10 ml. of distilled water to the funnel and without shaking it, draw off the aqueous layer. Repeat this procedure and then add 25 ml. of distilled water. Stopper the funnel as directed above. Draw off the aqueous and shake vigorously, releasing the pressure layer and repeat the washing with 25 ml. of distilled water. Retain the ether layer for the determination of total stearate (see 4.3.5.2). Combine all the washings with the original aqueous layer in the 400 ml. beaker and add 30 ml. of 1 : 1 sulfuric acid. Evaporate the solution on a hot plate under a hood until dense white fumes of sulfur trioxide appear. Allow the mixture to fume for about 10 minutes, then remove the beaker and contents from the hot plate and allow to cool. Cautiously dilute the mixture in the beaker with about 100 ml. of distilled water,

washing down the sides of the beaker. Stir the mixture thoroughly and bring it to a boil on the hot plate. Allow the beaker and contents to cool and add about 25 ml. of 95-percent ethyl alcohol. Stir the contents and allow the precipitate of lead sulfate to settle for about 2 hours. Filter the mixture using a tared, ignited Gooch crucible. Transfer the precipitate to the Gooch crucible, using a stream of 8-percent sulfuric acid solution, then wash the precipitate five times with 8-percent sulfuric acid solution and three times with 95-percent ethyl alcohol. Aspirate the crucible until no alcohol odor is detected, then dry the crucible and contents in an oven at about 100°C. Ignite the crucible and contents in a muffle furnace at 500° to 600°C. for 15 minutes. Cool in a desiccator and weigh. Calculate the gain in weight to percent total lead as follows:

$$\text{Percent total lead} = \frac{68.33A}{W(1 - M)}$$

A = gm. of ignited precipitate

W = gm. of sample.

M = moisture percentage expressed as a decimal fraction

4.3.5.2 *Total stearic acid content.* Transfer the ether layer obtained as specified in 4.3.5.1 to a tared 150 ml. beaker, washing the separatory funnel three times with 15 ml. portions of ether. Evaporate the ether on a steam bath, and place the beaker and contents in an oven at 100° to 105°C. until constant weight is attained, cool in a desiccator and weigh. Calculate the gain in weight as percent total stearic acid as follows:

$$\text{Percent stearic acid} = \frac{(A - B)}{C} \times 100$$

where:

A = weight of beaker and stearic acid content, gm.

B = weight of empty beaker, gms.

C = weight of sample, gms.

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4.3.5.2.1 *Melting point of fatty acid.* Use the fatty acids obtained as described in 4.3.5.2. Determine the melting point using the method described in 4.3.4.2 except that the sample shall not be dried at 70° plus or minus 2°C. or passed through a number 200 mesh sieve. Record as the melting point the temperature at which the material appears to become completely liquified.

4.3.5.2.2 *Iodine number of fatty acid.*

4.3.5.2.2.2 *Iodine number of fatty acid.* Determine the iodine number of fatty acid in accordance with Federal Standard No. 141, Method 5061, except a weighed portion of 0.12 gm. to 0.16 gms. of fatty acid shall be used.

4.3.6 *Granulation.* Transfer a weighed portion of 10 gm of the sample to a 600 ml. beaker and wet thoroughly with 95 percent ethyl alcohol, breaking up all lumps with a stirring rod. Add to the beaker about 500 ml. of a 1 : 1 water-alcohol mixture and stir vigorously to suspend the solid material in the liquid. Pour the suspension on a tared 3 inch, No. 100 U.S. Standard sieve, catching the material going through the sieve in a large dish. Wash any of the sample remaining in the beaker on the sieve by means of a stream of 1 : 1 water-alcohol mixture. Wash the residue on the sieve by brushing and washing with 1 : 1 alcohol-water mixture until the washings come through with only a slightly turbidity. Combine all the washings, stir them vigorously and pour them on a tared No. 100 U.S. Standard sieve. Repeat the procedure of brushing and washing. Pour the washings from the No. 200 sieve on a tared No. 325 U.S. Standard sieve following the same procedure indicated above, but this time discard the washings. Rinse each of the sieves once with 95 percent ethyl alcohol and then air-dry the sieves. Place the sieves in an oven at 95° to 100°C. for one-half hour, cool in a desiccator and weigh. Calculate the gain in weight of each sieve to percent lead stearate retained on that sieve.

5. PREPARATION FOR DELIVERY

5.1 Packing.

5.1.1 Level A.

5.1.1.1 *Drums.* Unless otherwise specified in the contract or purchase order, lead stearate shall be packed in a fiber, plywood or metal drum in accordance with Specification PPP-D-723 (Type III, Grade A), JAN-P-112, and PPP-D-729 (Type III, Full Removable Head Type), respectively. Fiber and plywood drums shall be furnished with a nominal 0.004 inch thick polyethylene liner, properly heat sealed or otherwise closed to afford protection to the contents equivalent to that given by a heat seal closure. (Alternately, an equivalent degree of protection may be furnished by suitable laminations or coating of the inner surface of the drum.) The drums shall be closed in accordance with Specification PPP-D-723, PPP-D-729 and JAN-P-112 as applicable.

5.1.1.2 *Alternate method.* Lead stearate may be packed in paper shipping sacks in accordance with Specification UU-S-48. Closure of sacks shall be in accordance with above Specification.

5.1.2 *Level C.* Lead stearate shall be packed to afford protection against damage during direct shipment from supply source to the first receiving activity for immediate use. Containers shall comply with Uniform Freight Classification Rules and Container Specifications for rail shipments or National Motor Freight Rules and Container Specifications for truck shipments, as applicable.

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

6. NOTES

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

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- (a) Title, number and date of the specification.
- (b) Applicable level of packing.

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 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.

Custodian:

Army—Ord

Navy—Wep.

Preparing activity:

Army—Ord