

MIL-F-70676 (AR)
29 June 1988

MILITARY SPECIFICATION

FERRIC ACETYLACETONATE

This specification is approved for use within the US Army Armament, Munitions and Chemical Command and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1. Scope. This specification covers the requirements and quality assurance provisions for the manufacture and acceptance of one type of ferric acetylacetonate (see 6.1).

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.

SPECIFICATIONS

MILITARY

MIL-A-48078 - Ammunition, Standard Quality Assurance Provisions, General Specification for

STANDARDS

MILITARY

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

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 Armament, Munitions and Chemical Command, Attn. AMSMC-QA, Picatinny Arsenal, New Jersey 07806-5000 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.

FSC 6810

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MIL-STD-129 - Marking for Storage and Shipment
 MIL-STD-286 - Propellants, Solid: Sampling,
 Examination and Testing
 MIL-STD-1168 - Lot Numbering of Ammunition
 MIL-STD-1218 - ACS Chemicals

(Copies of specifications, standards, handbooks, drawings, publications, 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.)

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

CODE OF FEDERAL REGULATIONS

Title 49, Transportation, Parts 100-199

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402. Orders for the above publication should cite: 49 CFR 100-199 (latest revision.)

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 DOD adopted shall be those listed in the issue of the DODISS shall be the issue of the nongovernment documents which is current on the date of the solicitation.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM E300-73 - Recommended Practice for
 Sampling Industrial Chemicals

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

UNITED NATIONS

United Nations - Transport of Dangerous Goods
 (Recommendations of the Committee of Expert on the Transport of Dangerous Goods.)

(Application for copies should be addressed to United Nations, Sales Section, First Ave., New York, N.Y.)

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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 Material. The ferric acetylacetonate shall conform to the following requirements when tested in accordance with the applicable test method.

<u>Property</u>	<u>Min</u>	<u>Max</u>	<u>Applicable Test Method</u>
Iron Content, %	14.4	16.2	4.5.1
Methylene Chloride Insolubles, %	-	1.0	4.5.2
Moisture, %	-	0.5	4.5.3

3.2 Color. The ferric acetylacetonate shall be an orange-red crystalline solid, when tested in accordance with 4.5.4.

3.3 First article inspection. This specification contains technical provisions for first article inspection. Requirements for the submission of first article samples by the contractor shall be as specified in the contract.

3.4 Workmanship. The manufacturer shall implement procedures and controls to assure that the process and the product produced are not compromised by foreign materials and contaminants or any other conditions which may degrade the composition. Determination of foreign materials shall be in accordance with 4.5.5.

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.

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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 Classification of inspections. The inspection requirements specified herein are classified as follows:

- a. First article inspection (see 4.3).
- b. Quality conformance inspection (see 4.4).

4.3 First article inspection.

4.3.1 Submission. The contractor shall submit a first article sample as designated by the Contracting Officer for evaluation in accordance with provisions of 4.3.2. The first article sample shall be ten (10) pounds of ferric acetylacetonate. The sample shall be obtained from the first production lot which has been produced by the contractor using the same production processes, procedures and equipment as will be used in fulfilling the contract. All materials shall be obtained from the same sources of supply as will be used in regular production.

4.3.2 Inspection to be performed. The sample will be subjected by the Government to any or all of the examinations or tests specified in Table II (see MIL-A-48078).

4.3.3 Rejection. See MIL-A-48078.

TABLE II. First article inspection

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CLASSIFICATION OF DEFECTS & TESTS

PARAGRAPH	TITLE	SHEET OF		DRAWING NUMBER
		NO. OF SAMPLE UNITS	REQUIREMENT PARAGRAPH	
CATEGORY	EXAMINATION OR TEST	AQL OR 100%	PARAGRAPH REFERENCE / INSPECTION METHOD	NEXT HIGHEN ASSEMBLY
	Iron Content Methylene Chloride Insolubles Moisture Color Workmanship		3.1 3.1 3.1 3.2 3.4	4/5/1 4.5.2 4.5.3 4.5.4 4.5.4
NOTES:				

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4.4 Quality conformance inspection.

4.4.1 Lot formation. A lot shall consist of one or more batches of ferric acetylacetonate produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each lot shall consist of that quantity of ferric acetylacetonate that has been subjected to the same unit chemical or physical process intended to make the final product homogeneous. The lot shall comply with the provisions for submission of product as specified in MIL-STD-105. The criteria and procedure for the assignment of lot numbers shall be in accordance with MIL-STD-1168. Also, MIL-A-48078 applies.

4.4.2 Examination. See MIL-A-48078.

a. Workmanship. Examination for workmanship shall be conducted on each sample selected for testing in accordance with 4.4.3.1. If any sample fails to meet any test requirement the batch represented by the sample shall be rejected.

4.4.3 Testing. PRECAUTION: This specification covers sampling and testing of toxic and hazardous materials. Accordingly, it is emphasized that all applicable safety rules, regulations and procedures must be followed in handling and processing the triphenyl bismuth.

4.4.3.1 Sampling. Approximately 500 grams of the composition shall be selected from each batch to be sampled using ASTM Method E300-73 for solids. Samples shall be selected for inspection in accordance with table below. If any sample fails to meet any test requirement the batch represented by the sample shall be rejected. Each required test shall be performed in duplicate. No composite samples shall be used. The classification of defects shall be as given in Table III.

<u>Number of containers in batch</u>		<u>Number of containers to be sampled</u>
2	- 100	2
100	- 200	5
200	- 300	8
300	- 50	10
500	- 1000	13
1000	- 3000	20
3000	- 10,000	32

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TABLE III. Classification of defects

<u>Test Examination</u>	<u>Paragraph</u>	<u>Defects Classification</u>
Iron Content	3.1	Major
Methylene Chloride Insolubles	3.1	Major
Moisture	3.1	Major
Color	3.2	Major
Workmanship	3.4	Major

4.4.4 Inspection equipment. For the performance of all tests and examinations specified in 4.4 and 4.5, commercial inspection equipment should be employed. The contractor shall have available and utilize correctly, this equipment and is charged with the responsibility of assuring that proper calibration procedures are followed.

4.5 Methods of inspection. All tests given in this section shall be performed using prescribed procedures for replicate determinations given in standard analytical chemistry textbooks. Also, unless otherwise specified herein, all chemicals shall be Reagent Grade or ACS Grade in accordance with MIL-STD-1218. See 6.3 for use of equivalent test methods.

4.5.1 Iron content. The iron content of ferric acetylacetonate shall be determined as follows:

4.5.1.1 Method. Organic matter is first removed by treatment with sulfuric and nitric acids. The iron is then reduced with stannous chloride solution and titrated with standard potassium dichromate solution to the diphenylamine sulfonate end point.

4.5.1.2 Apparatus.

- a. Erlenmeyer flask; 300ml
- b. Buret; 50ml
- c. Graduated cylinder

4.5.1.3 Reagents.

- a. Phosphoric acid; reagent grade, 85%
- b. Sulfuric acid; reagent grade
- c. Nitric acid; reagent grade

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- d. Hydrochloric acid; reagent grade
- e. Stannous chloride solution; 60g stannous chloride dissolved in 50ml of hydrochloric acid - dilute to 1 liter with water.
- f. Mercuric chloride solution; 5g mercuric chloride/ 100ml water.
- g. Potassium dichromate solution; 0.1N prepare as primary standard from oven dried crystal (4.9032g/ liter water)
- h. Diphenylamine indicator; 0.2 sodium diphenylamine sulfonate/ 100ml water

4.5.1.4 Procedure.

- a. Accurately weigh a 1g sample into a 300ml flask.
- b. Carefully add 10ml of sulfuric acid and 5ml of nitric acid.
- c. Heat until heavy white fumes of SO_3 are evolved. Cool slightly and very carefully repeat the nitric acid addition. Again heat until heavy white fumes are evolved.
- d. Repeat the nitric acid addition and heating cycle until all the organic matter has been removed. The solution should be nearly colorless (white salts of ferric sulfate may form). No brown color should be visible. Before continuing remove any residual nitric acid by heating strongly until heavy white fumes are evolved.
- e. Cool to nearly room temperature and add 100ml of water and 5ml of hydrochloric acid.
- f. Heat to near boiling to dissolve any precipitate that may have formed. While the solution is still hot, add stannous chloride solution dropwise to discharge the yellow color. Add 1-2 drops excess. The sample solution should now be colorless.
- g. Cool to room temperature, and quickly add with stirring 10ml pf mercuric chloride solution. A small amount of white precipitate should form. (No precipitate formation indicates that an insufficient amount of stannous chloride was used. Formation of a grayish precipitate indicates too large an excess of stannous chloride was used. In either case, the sample should be discarded.)
- h. Add 15ml of phosphoric acid and 5-10 drops of diphenylamine sulfonate indicator. Titrate with standard 0.1N potassium dichromate to the violet endpoint.

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4.5.1.5 Calculations.

$$\frac{\text{ml K}_2\text{Cr}_2\text{O}_7 \times \text{N K}_2\text{Cr}_2\text{O}_7 \times 5.585}{\text{sample weight (g)}} = \% \text{ Fe}$$

4.5.2 Methylene chloride insolubles. The methylene chloride insolubles shall be determined as follows:

4.5.2.1 Apparatus.

- a. Beaker; 150ml
- b. Gooch crucible; medium frit
- c. Filter Flask and vacuum system
- d. Oven capable of maintaining 100° to 105°C

4.5.2.2 Reagents.

Methylene chloride

4.5.2.3 Procedure.

- a. Accurately weigh two grams of sample in a 150ml beaker.
- b. Add 100ml of methylene chloride and mix without heating.
- c. Filter thru a tared medium porosity pyrex crucible (dried at 105°C for 1 hour, cooled in desiccator and weighed), using suction.
- d. Rinse beaker with four 20ml aliquets of methylene chloride, pour washes into crucible. Aspirate for 10 minutes to air dry the residue.
- e. Place the crucible and contents in a 105°C oven for one hour.
- f. Cool in a desiccator and weigh.
- g. Calculate the percent insoluble material as follows:

$$\frac{(\text{weight of residue, g}) (100)}{\text{weight of sample, g}} = \text{insoluble}$$

4.5.3 Moisture. The moisture content shall be determined in accordance with MIL-STD-286, Method 102.1.3.

4.5.4 Color. The sample shall be examined for conformance with proper color.

4.5.5 Workmanship. Visually examine the sample for contamination or foreign matter.

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5. PACKAGING.

5.1 Packaging and Packing.

5.1.1 Level A. - When applicable packaging shall be as specified in the contract.

5.1.2 Level B. - Unless otherwise specified in the contract, packaging and packing shall be in accordance with the standard commercial practice applicable to the type of material. The packaging and packing shall be of such construction and materials that the contents will be adequately protected against damage, loss, or contamination when shipped from the contractor or supply source to the receiving activity, or stored for one year from the date of manufacture. Container size/capacity shall be as specified in the contract. The type of containers and packing materials selected shall comply with applicable carrier rules and regulations. In addition, any hazardous material must be packed and shipped in accordance with the Code of Federal Regulations (CFR), Title 49, Parts 100-199, and also when offered for overseas shipment, the United Nations-Transport of Dangerous Goods (Recommendations of the Committee of Experts on the Transport of Dangerous Goods). It shall be the responsibility of the supplier to obtain all data necessary from DOT, and if applicable, from the Committee of Experts on the Transport of Dangerous Goods (UN-Transport of Dangerous Goods) to ship hazardous materials legally.

5.1.3 Level C. - Same as Level B, except not authorized for overseas shipment and that the United Nations-Transport of Dangerous Goods does not apply.

5.2 Marking. In addition to any special marking required by the contract, each container shall be marked in accordance with MIL-STD-129, the Code of Federal Regulations, (CFR) Title 49, Parts 100-199, and the United Nations-Transport of Dangerous Goods (Recommendations of the Committee of Experts on the Transport of Dangerous Goods). Marking shall include but not be limited to, the following information:

- a. Title, Number and Date of this Specification
- b. Manufacturer's Name and Location
- c. Material Trade Name
- d. Net Weight and Volume

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- e. Lot Number, Batch Number(s), and Date of Manufacture
- f. Storage Conditions/Requirements
- g. Toxicity Precautions, if applicable
- h. Contract Number
- i. Warrantly Provisions

6. Notes.

6.1 Intended use. The ferric acetylacetonate is intended for use in the M864 propellant.

6.2 Ordering data. See MIL-A-48078 (AR).

6.3 Equivalent test methods. The test methods given in this specification are the official methods to be used. The contractor may request using other methods providing that the proposed method is equivalent (accuracy and precision) to the method given in this specification. Prior approval of the Contracting Officer is required for use of equivalent test methods. A description of the proposed method should be submitted through the Contracting Officer to: Commander, AMCCOM, ATTN: AMSMC-QAR-R(D), Picatinny Arsenal, NJ 07806-5000. This description should include, but not be limited to, the procedures used, the accuracy and precision of the method, test data to demonstrate the accuracy and precision and drawings of any special equipment required (see MIL-I-45208).

6.4 Subject term (key word) listing.

Propellant
Antioxidant
Projectile

Custodian:
Army-AR

Preparing Activity
Army-AR

(Project 6810-A019)

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions - Reverse Side)

1. DOCUMENT NUMBER

MTI-E-70676

2. DOCUMENT TITLE

FERRIC ACETYLACETONATE

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 Area Code) - Optional

c. MAILING ADDRESS (Street, City, State, ZIP Code) - Optional

8. DATE OF SUBMISSION (YYMMDD)