MIL-O-82672A(OS)
6 August 1986
SUPERSEDING
MIL-O-82672(OS)
19 January 1979

MILITARY SPECIFICATION

OTTO FUEL II

This specification is approved for use within 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 Scope. This specification covers one type of liquid monopropellant designated Otto Fuel II. This specification applies at the time of formulation by the manufacturer.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this specification to the extent specified herein. Unless otherwise specified, the issue 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-N-3399	2-Nitrodiphenylamine
DOD-B-82669(OS)	Butyl Sebacate, Di-Normal
DOD-P-82671(OS)	Propylene Glycol Dinitrate

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Ordnance Station, Standardization/ Documentation Division (5241), Indian Head, MD 20640-5000, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC NO. 3966 FSC 9135

STANDARDS

FEDERAL

FED-STD-313	Material Safety Data Sheets Preparation and Submission of

MILITARY

MIL-STD-129	Marking for Shipment and Storage
MIL-STD-1322-55	Unit Load for Domestic and Overseas Shipments
MIL-STD-286	Propellants, Solid: Sampling, Examination
MIL-STD-1218	ACS Chemicals

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

DRAWING

NAVSEA #5012852	Container, Shipping (Otto Fuel)

PUBLICATIONS

NAVAL SEA SYSTEMS COMMAND

OD 43852 (Code Ident 10001)	Determination of Sodium in Propylene Glycol Dinitrate (PGDN) by Atomic Absorption Method
S6340-AA-MMA-010 (Code Ident 53711)	Otto Fuel II; Safety, Storage and Handling Instructions

Code of Federal Regulations

49 CFR 100-177	Transportation
49 CFR 178-199	Transportation

(Application for copies of the Code of Federal Regulations should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20404. Order for the above publication should cite "the latest edition and Supplements thereto".)

(Copies of specifications, standards, handbooks, drawings, and publications required by manufacturers in connections with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, 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 First article. When specified in the contract or purchase order, a sample shall be subjected to first article inspection (see 4.4 and 6.3)."

3.2 Materials.

3.2.1 2-Nitrodiphenylamine (2NDPA). The 2-nitrodiphenylamine shall conform to the requirements of MIL-N-3399.

3.2.2 Di-n-butyl sebacate. The di-n-butyl sebacate shall conform to the requirements of DOD-B-82669(OS).

3.2.3 Propylene glycol dinitrate. The propylene glycol dinitrate shall conform to the requirements of DOD-82671(OS).

3.3 Chemical requirements. The Otto Fuel II (moisture-free basis) shall conform to the requirements of TABLE I when tested as specified herein.

TABLE I. Chemical Requirements (moisture-free basis).

Component	Requirement		Test Method
	Minimum (% by wt)	Maximum (% by wt)	
Propylene glycol dinitrate	75.8	76.2	4.5.2.1
2-Nitrodiphenylamine	1.4	1.6	4.5.2.2
Di-n-butyl sebacate	22.2	22.8	4.5.2.3
Sodium	-	0.8 ppm	4.5.2.5

3.3.1 Moisture content. The maximum moisture content shall be 0.10% by weight as determined in accordance with 4.5.2.4.

3.4 Material safety data sheet. A Material Safety Data Sheet (MSDS) shall be prepared and submitted in accordance with FED-STD-313. Questions pertinent to the effect of Otto Fuel II on the health of personnel when used for its intended purpose are described in S6340-AA-MMA-010. Additional questions shall be referred by the acquiring activity to the Naval Medical Command which will act as advisor to the acquiring activity (see 4.1.2 and 6.8).

3.5 Workmanship. Otto Fuel II shall be free from dirt, sediment, and other suspended foreign matter when examined visually by transmitted light.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order (see 6.2.1), the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order (see 6.2.1), 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 this specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.1.1 Responsibility for compliance. This material 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 materials, either indicated or actual, nor does it commit the Government to acceptance of defective material.

4.1.2 Toxicological information. The contractor shall have the toxicological formulation and associated information available for review by the contracting activity to evaluate the safety of the material for the proposed use.

4.2 Inspection provisions.

4.2.1 Lot formation. A lot shall consist of the Otto Fuel II offered for acceptance at one time which has been produced by one manufacturer, at one plant, during a period of time not exceeding 20 hours, from the same lots of 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 6.2.1 and 6.6).

4.2.2 Sampling. Using a dipper made of rubber or safety department approved plastic, a sample of approximately 50 milliliters (ml) of Otto Fuel II shall be removed from each lot. The sample shall be transferred to a stoppered bottle made of rubber or an approved plastic. The bottle shall be labeled to show the lot number and date of manufacture.

4.2.2.1 Cleaning sample bottle. After disposal of the Otto Fuel II from the sample bottle (see S6340-AA-MMA-010), the bottle shall be cleaned as follows:

a. Rinse thoroughly with acetone.

b. Flush generously with hot tap water (3 minutes under a direct flow is considered adequate.)

c. Rinse with distilled water and dry.

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

a. First article inspection (see 4.4)

b. Quality conformance inspection (see 4.5)

4.4 First article inspection. First article inspection shall consist of all of the quality conformance tests and examinations specified herein and any additional tests specified by the acquiring activity (see 6.3).

4.4.1 Rejection. Failure to meet all the requirements of section 3 shall cause rejection of the sample or lot. Reformulation or adjustment of the lot is permitted and resampling is authorized for lot acceptance.

4.5 Quality conformance inspection. The quality conformance inspection shall consist of the following tests and examinations (see 6.2.2). Unless otherwise specified, all chemicals shall be ACS grade in accordance with MIL-STD-1218 and distilled water shall be used (see 6.2.1).

4.5.1 Visual inspection. Each lot of Otto Fuel II shall be visually inspected for conformance to workmanship, packaging, packing, and marking requirements.

4.5.2 Test methods and procedures.

4.5.2.1 Propylene glycol dinitrate content. The propylene glycol dinitrate content shall be determined by one of the two following methods:

CAUTION:

Safety shields and personal protective clothing shall be used. The lower stopcock must be open until the gas has been generated, and any temperature rise in the sample during the reaction must be avoided.

4.5.2.1.1 Nitrometer method. Prepare the nitrometer as described by MIL-STD-286B, method 209.3.2. Wash the nitrometer generator using 25 ml of glacial acetic acid for each of two rinses. Expel all air from the stopcock and sidearm and cap the sidearm. Add 2 ml of glacial acetic acid to the generator cup and draw it approximately three quarters of the way into the nitrometer. Weigh accurately to 0.1 milligram (mg) a sample containing 0.90 ± 0.05 gram (g) of Otto Fuel II and transfer quantitatively to the generator cup. Draw the acid sample mixture into the generator. Wash the generator cup twice with 2 ml portions of glacial acetic acid and draw these washings into the generator. Add 25 ml of 94.5% sulfuric acid to the generator cup and draw the sulfuric acid into the generator tube being careful to exclude any air. Close the top stopcock and immediately open the bottom stopcock. React the sample by gently shaking the top of the generator bulb holding the bottom end of the bulb as the axis. Allow the generator to stand until the mercury settles. Drain the excess mercury until about 50 millimeters (mm) of mercury remain in the bottom of the generator bulb. Close the bottom stopcock, shake the generator bulb vigorously for 3 to 5 minutes and allow the generator to stand for an additional 5 minutes. Vent any air from the reading tube, the stopcock, and the sidearm of the reading tube. Connect the generator to the reading tube with an airtight connection. Transfer the gas to the reading tube and adjust the mercury level in the compensatory tube so that it is level with the calibration mark. Allow the system to come to equilibrium at ambient temperature, make a final adjustment to the mercury level, and read the gas volume.

Calculation:

$$\%\ \text{PGDN in sample} = \%\ \text{PGDN measured by nitrometer} + \%\ \text{PGDN combined with 2NDPA}$$

$$\%\ \text{PGDN in sample} = \frac{R}{.1687W} + \frac{WxBxFx.1687}{.1687W}$$

$$\%\ \text{PGDN in sample} = 5.9277x\ \frac{R}{W} + 0.98x\ \text{2NDPA}$$

Where: R = reading of the mercury level in the measuring tube, percent nitrogen
W = weight of sample (on moisture-free basis), grams
B = percent 2NDPA in sample by analysis
F = 0.98 empirical factor determined in the laboratory to correct for interference by the 2NDPA
.1687 = theorectical mole fraction of nitrogen in PGDN

4.5.2.1.2 High performance liquid chromatography method. This method for PGDN analysis is given in the appendix.

4.5.2.2 2-Nitrodiphenylamine content. The 2-nitrodiphenylamine content in Otto Fuel II shall be determined by one of the three following methods:

4.5.2.2.1 Spectrophotometric method.

NOTE

This method is applicable to the determination of 2-nitrodiphenylamine at a concentration on the order of 1 or 2 percent in freshly manufactured Otto Fuel II only. Any compound exhibiting an absorption at the analytical wavelength will interfere and must be separated prior to analysis or corrections made based on an independent determination.

Apparatus.

a. Spectrophotometer. Any instrument capable of quantitative absorbance at 422 nanometers (nm). The following instruments have been used satisfactorily: Beckman Models DB, B, and DU; Bausch and Lomb Spectronic 20; and Coleman Model 6-C.

b. Absorption cells, 1-centimeter (cm) path length, matched.

Reagents and standards.

a. Ethanol, 95 percent. Denatured formulations, which contain no interfering substances, may be used.

b. 2-Nitrodiphenylamine, MIL-N-3399.

c. 2-Nitrodiphenylamine stock solution. Weigh 0.1 g of 2-nitrodiphenylamine to the nearest 0.1 mg into a 250-ml volumetric flask. Dissolve the 2-nitrodiphenylamine in, and dilute to volume with, 95 percent ethanol and mix thoroughly. This solution will contain approximately 0.4 mg 2-nitrodiphenylamine per ml.

d. Using a pipette, measure 1-, 2-, 3-, and 4-ml aliquots of the 2-nitrodiphenylamine stock solution into 100-ml volumetric flasks. Dilute to volume with 95 percent ethanol and mix thoroughly. Measure the absorbance of each solution at 422 nm in one cm cells using 95 percent ethanol in the reference cell.

e. Plot absorbance values as ordinates against concentration of 2-nitrodiphenylamine in mg per 100 ml.

Procedure.

Weigh 1.0 g of Otto Fuel II to the nearest 0.1 mg into a 250-ml volumetric flask, dilute to volume with 95 percent ethanol, and mix thoroughly. Fill the reference cell with 95 percent ethanol. Transfer 1-ml of the solution to give about 1 mg of 2-nitrodiphenylamine in the final solution to a 100-ml volumetric flask. Dilute to volume and mix thoroughly. Measure the absorbance of the resulting solution at 422 nm in a one cm cell. Determine the concentration of 2-nitrodiphenylamine in the solution by reference to the established calibration curve. Calculate the weight percent of 2-nitrodiphenylamine in the original material as follows:

$$\text{Weight percent 2-nitrodiphenylamine} = \frac{CF}{10W}$$

Where: C = concentration of the solution obtained from the calibration curve
F = aliquot factor
W = weight of sample, g

4.5.2.2.2 Volumetric bromination method.

Reagents and standards.

a. Glacial acetic acid, reagent grade.

b. Potassium bromate - bromide solution, 0.2N. Prepare in accordance with Method 605.1, MIL-STD-286B.

c. Sodium thiosulfate solution, 0.2N, standardized. Prepare in accordance with Method 602.1, MIL-STD-286B.

d. Hydrochloric acid solution, 1:1.

e. Potassium iodide solution, 10%.

f. Starch indicator solution. Prepare in accordance with Method 701.1, MIL-STD-286B.

Procedure.

Weigh 3.75 ± 0.25 g of Otto Fuel II to the nearest 0.1 mg into a 250-ml iodine flask. Add 50 ml of acetic acid measured from a graduated cylinder, 25-ml of potassium bromate-potassium bromide solution measured from a buret, and 10 ml of 1:1 hydrochloric acid from a graduated cylinder. Immediately stopper the flask and from a 25-ml portion of 10% potassium iodide solution make a ring seal around the stopper. While holding the stopper in place, agitate (as much as the ring seal will permit) the contents of the flask for approximately 15 seconds and allow bromination to proceed for one minute from the time of addition of the hydrochloric acid. Partly remove the stopper from the flask and add the remainder of the 25-ml portion of the potassium iodide solution, being careful not to lose bromine fumes from the flask. Wash the stopper and sides of the flask with a small amount of distilled water. With constant swirling, titrate the contents of the flask with 0.2N sodium thiosulfate solution until near the end point as indicated by the disappearance of the strong iodine color. Add approximately 5 ml of starch until the blue color disappears. Titrate a blank, using exactly the same volumes of the solutions, omitting only the sample.

Calculation:

$$\text{Percent 2NDPA} = \frac{(A - B) \times N \times 3.570}{W}$$

Where: A = sodium thiosulfate to titrate blank, ml
B = sodium thiosulfate to titrate sample, ml
N = normality of sodium thiosulfate solution
W = weight of sample, g

4.5.2.2.3 High performance liquid chromatography method. This method is described in the Appendix.

4.5.2.3 Di-n-butyl sebacate content. The di-n-butyl sebacate content in Otto Fuel II shall be determined by subtracting the sum of the percentages of proplylene glycol dinitrate and 2-nitrodiphenylamine fromm 100 percent.

4.5.2.4 Moisture content in Otto Fuel II. The moisture content in Otto Fuel II shall be determined as follows: To the titration flask, add 75 to 100 ml of methanol. Titrate the methanol to a potentiometric end point with a stabilized Karl Fischer reagent. Weigh a 5 to 15 g sample of Otto Fuel II accurately to 0.1 mg and quantitatively transfer to the titration flask. Titrate the sample to a potentiometric end point. Calculate the moisture content as follows:

$$\text{Percent moisture} = \frac{100\ (KF)}{W}$$

Where: K = Amount of Karl Fischer reagent used in titration, ml
F = reagent factor (g of water per ml of reagent)
W = weight of Otto Fuel II, g

4.5.2.5 Sodium content. The sodium content in Otto Fuel II shall be determined in accordance with OD 43852 or with an alternate procedure approved by the procuring activity prior to use.

4.6 Bulk shipping inspection. When bulk shipping is selected, the following evaluations shall be performed to monitor the integrity of the system. The tests and examinations of 4.5 shall be performed on all samples. Failure of a sample to meet any requirements shall be cause for rejection of the material and shall be referred to the procuring activity.

4.6.1 Bulk storage. For the storage of multiple lots in a bulk storage tank, each lot shall have met all the requirements of 4.5 prior to placing the material in the storage tank.

4.6.2 Bulk storage testing. The contents of each tank used for bulk storage of fuel prior to delivery shall be sampled and tested a minimum of once per month.

4.7 Rejection criteria. Failure of a sample to meet all the requirements of the specification shall be cause for rejection of the lot.

5. PACKAGING

5.1 Preservation and packaging. Preservation and packaging shall be level A. In accordance with MIL-STD-129J (Maximum Protection).

5.1.1 Quantities of 55 gallons or less.

5.1.1.1 Container capacity. The inner container shall not be filled to more than 95 percent capacity.

5.1.1.2 Laboratory samples. Laboratory samples shall be packaged in polyethylene containers.

5.1.1.3 Small quantities. When shipped in a small quantity other than laboratory samples, the fuel shall be packaged in a 5-gallon polyethylene container and packed in accordance with 5.2.1.

5.1.1.4 Nonbulk quantities in excess of 5 gallons. Quantities in excess of 5 gallons, but not exceeding 54 gallons, shall be packed as follows:

5.1.1.4.1 Material. The container shall be made of polyethylene DOT-2S

having these properties (see Section 49 of the Code of Federal Regulations (CFR) 178.35):

Melt index (maximum)	2.6
Density	0.910 to 0.925 g/ml
Tensile strength (minimum)	15000 psi
Elongation (minimum)	400%

5.1.1.4.2 Capacity. The container shall have a minimum capacity of 53 gallons and a maximum capacity of 54 gallons.

5.1.1.4.3 Wall thickness. The polyethylene shall fit snuggly inside of a 55 gallon removable-head steel drum; the side clearance shall be less than 0.13 inch. The clearance between the tip bung and the underside of the lid of the steel drum shall be less than .25 inch.

5.1.1.4.5 Closure. Openings shall not exceed 2.3 inches in diameter. The closing device shall be a polyethlene screw plug, with or without a polyethylene bushing, and shall prevent leakage. An overcap shall be used to keep the threads free from dirt.

5.2 Packing. Packing shall be level A, in accordance with MIL-STD-129J (Maximum Protection).

5.2.1 Small quantities. The material packaged in accordance with 5.1.1.3 shall be overpacked in a steel drum which is epoxy-lined or coated with a material impervious to Otto Fuel II. This container shall fully comply with DOT-6J container specification (49 CFR 178.100) for a steel drum overpack for a polyethylene container. Any void between the inner and outer containers shall be filled with a fire-retardant or inert material.

5.2.2 Nonbulk quantities in excess of 5 gallons. The material packaged in accordance with 5.1.1.4 shall be packed in a container in accordance with NAVSEA DWG 5012852.

5.2.2.1 Material. For drums shipment, Otto Fuel is classified "Chemical, N.O.I.B.N.", except when shipped by air. For air shipment other than carry-on baggage, the fuel is classified as "Toxic Material, NOR".

5.2.2.2 Labeling. The outer container shall be clearly marked to indicate the upper end.

5.2.2.3 Palletizing Procedure. If required, the drums shall be palletized in accordance with MIL-STD-1322-55.

5.3 Bulk shipment, quantities in excess of 55 gallons. Bulk (tank truck) quantities in excess of 55 gallons shall be shipped as specified below:

a. For bulk shipments, Otto Fuel II shall be classified as "Chemical, N.O.I.B.N." All regulations pertaining to this type of material shall be observed. Only Department of Defense approved carriers shall be utilized for bulk shipments.

b. The originating activity (consignor) shall ensure that the tank truck is free of any contaminants prior to filling.

c. Subsequent to loading and unloading, the tank dome and all other tank openings shall be sealed with a Navy-numbered strap-type seal or equivalent serialized seal.

d. The bill of lading and other applicable shipping documents shall carry the seal numbers and admonition to consignee that the shipment or empty truck is not to be accepted unless the seal(s) is (are) intact.

e. Consignee shall unload Otto Fuel II by gravity flow or by the application of up to a maximum of 50 psi air pressure. Use of a pump is satisfactory if the pump is of a design compatible with the fuel. (See S6340-AA-MMA-010).

f. After unloading, the tank dome and other tank openings shall be sealed with no attempt to cleanup or decontaminate. The sealed tank trailer shall be delivered to the Naval Ordnance Station, Indian Head, MD, or other NAVSEA approved decontamination station for cleanup or decontamination prior to reuse. Cleanup of tank trailers is not required prior to reuse provided such trailers remain in exclusive Otto Fuel II service and are not contaminated by foreign material.

g. If the tank is to be reused for any material other than Otto Fuel II, the tank must be decontaminated by the Naval Ordnance Station, Indian Head, MD, or other NAVSEA approved decontamination station prior to use in the new service.

h. Motor carriers designated by Military Traffic Management Command (MTMC) shall submit drawings, plans, or other documentation relative to the tankage section of their tank vehicles to the Naval Ordnance Station, Indian Head, MD, 20640-5000. These documents shall be reviewed for adequacy of design, construction and materials applicable to the shipment of Otto Fuel II. Approval by the Naval Ordnance Station shall be forwarded to Naval Sea Systems Command (SEA-06T1) for official approval as an authorized carrier of subject material.

i. The metal preferred for the container is stainless steel, although virtually any noncopper-containing metal may be acceptable.

j. The outage or ullage to be observed shall be a minimum of 5 percent. (This is well in excess of that required to allow for thermal expansion.)

k. The safety settings on the container dials, or pressure relief settings, shall be such that promiscuous venting would be avoided yet the container would be fully protected from any pressure even remotely approaching its yield strength. For purposes of comparison, this pressure would probably be in the 25- to 100-psi pressure range.

5.4 Marking. Unless otherwise specified by the contract (see 6.2), the inner containers and shipping containers shall be marked in accordance with the requirements of MIL-STD-129 and shall include the following:

a. Title, number and date of this specification
b. Quantity in container
c. Date of manufacture
d. Lot number and manufacturer's name
e. Precautionary marking (see 5.4.1)

5.4.1 Precautionary marking. All steel drums shall be marked with the label shown in FIGURE 1.

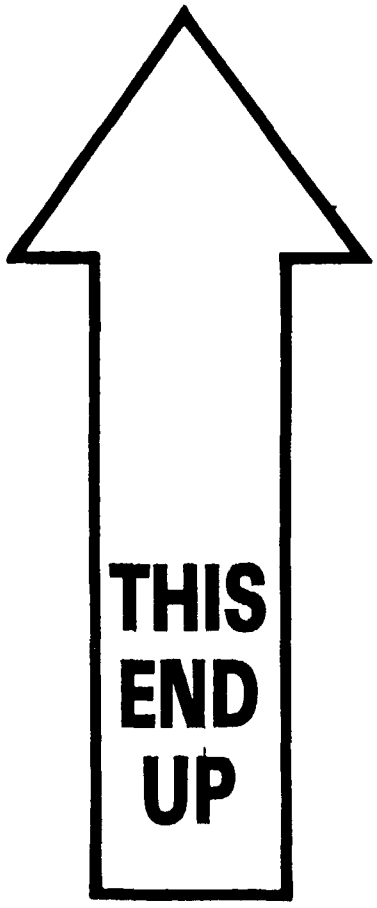
6. NOTES

6.1 Intended use. Otto Fuel II is intended for use as a liquid monopropellant.

6.2 Ordering data. Acquisition documents should specify the following:

6.2.1 Acquisition requirements.

a. Title, number and date of this specification.
b. Place of delivery.
c. Whether a preproduction sample is required (see 3.1 and 6.3)
d. Whether the contractor is responsible for acceptance testing (see 4.1).
e. Whether the contractor shall use his own laboratory or other suitable facility (see 4.1).
f. Lot size (if applicable) (see 4.2.1).
g. Whether additional first article tests are required (see 4.4).
h. Whether a special grade of chemicals is required (see 4.5).
i. Type and size of shipping container desired (see 5.1).
j. Additional marking (see 5.4).



OTTO FUEL II

HANDLE WITH CARE

ATTENTION

- This material must be stored alone upon arrival at its destination
- OTTO FUEL II has been assigned a liquid hazard classification of group I (low fire hazard, flash point 265°F), compatibility group G Storage shall be in accordance with OP 5 and S6340-AA-MMA-010
- **Physical exposure to this liquid or its vapors will cause severe headaches and/or nausea. Medical advice should be sought in such cases.**
- In the event of spillage, the provisions of S6340-AA-MMA-010 must be strictly followed
- In case of unusual problems contact

NAVAL ORDNANCE STATION
Indian Head, Maryland 20640-5000
Commercial phone: 301-743-4438
Autovon. 364-4438

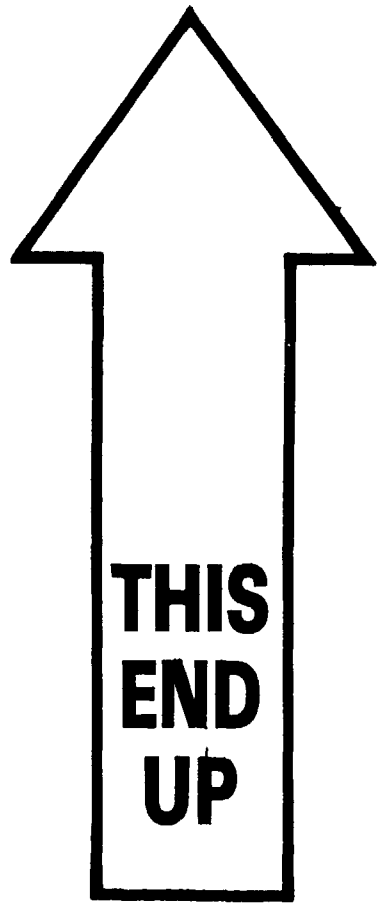


FIGURE 1 Sample of label for use on Otto Fuel II shipping containers

k. That the safety precaution requirements of the "Contractors Safety Manual for Ammunition, Explosives and Related Dangerous Material" (DOD 4145.26M) are applicable. NOTE: When this specification is used as part of the description of work to be accomplished by a Government activity, the safety precaution requirements of OP 5 and S6340-AA-MMA-010 are applicable.

6.2.2 Data requirements. When this specification is used in an acquisition and data are required to be delivered, the data requirements identified below shall be developed as specified by an approved Data Item Description (DD Form 1664) and delivered in accordance with the approved Contract Data Requirements List (CDRL), incorporated into the contract. When the provisions of DOD FAR Supplement, Part 27, Sub-Part 27.410-6 (DD Form 1423) are invoked and the DD Form 1423 is not used, the data specified below shall be delivered by the contractor in accordance with the contract or purchase order requirements. Deliverable data required by this specification are cited in the following paragraphs.

Paragraph No.	Data Requirements Title	Applicable DID No.	Option
4.5	Inspection Test Reports	DI-T-5329	

(Data item descriptions related to this specification and identified in section 6, will be approved and listed as such in DOD 5010.12-L, AMSDL. Copies of data item descriptions required by the contractors in connection with specific acquisition functions should be obtained from the Naval Publications and Forms Center or as directed by the contracting officer.)

6.3 First article sample. When a first article sample inspection is required, the material should be a first article sample from the contractor's current inventory. The first article sample should consist of enough material to perform all the tests of 4.4. The contracting officer should include specific instructions in acquisition documents regarding arrangements for additional tests, regular examinations, approval of first article sample test results and disposition of the first article sample. invitations for bids should provide that the Government reserves the right to waive the requirement for samples for first article sample inspection to those bidders offering a product which has been previously acquired or tested by the Government, and that bidders offering such products, who wish to reply on such production or test, must furnish evidence with the bid that prior Government approval is presently appropriate for the pending contract.

6.4 Additional information. Additional information pertinent to Otto Fuel II may be obtained from the following publications:

OD 30980 (Code Ident 10001)	Manufacture of Otto Fuel II by the Biazzi Process
OD 30981 (Code Ident 10001)	Manufacture of Otto Fuel II by the Batch Process
Naval Ordnance Station, Indian Head, MD, Document Number 533-174 TD-0001 (Code Ident 14083)	Laboratory Nitration of Glycerine, Propylene Glycol and 1, 2, 4 - Butanetriol

(Copies of these publications may be obtained from the procuring activity.)

6.5 Subject term listing.

Batch nitration
Bromination
Chromatography, high performance liquid
Continuous process nitration
Data Item Descriptions
Di-Butyl sebacate
Material safety data sheet
Moisture
2-Nitrodiphenylamine
Nitrometer
Propylene glycol dinitrate
Safety
Shipment in drums
Shipment in tanks
Sodium content
Spectrophotometer
Toxicity

6.6 Batch. A batch is defined as that quantity of Otto Fuel II that has been subjected to the same unit chemical or physical process intended to make the final product homogeneous.

6.7 Toxic effect. Toxic effects may occur from the inhalation of vapors from Otto Fuel II or the absorption from direct contact with the skin.

6.8 Material safety data sheets. Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313. The pertinent government mailing addresses for submission of data are listed in appendix B of FEB-STD-313.

6.9 Changes from previous issue. Asterisks (or vertical lines) are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

Preparing Activity
Navy - OS
(Project No. 9135-N116)

APPENDIX

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) ANALYSIS METHOD FOR PROPYLENE GLYCOL DINITRATE (PGDN) AND 2-NITRODIPHENYLAMINE (2NDPA) IN OTTO FUEL II

10. SCOPE

10. This method is optional and may be used for determination of propylene glycol dinitrate (PGDN) and 2-nitrodiphenylamine (2NDPA) stabilizer in Class A and Class B Otto Fuel II, which is composed of the above two materials and the diluent di-butyl sebacate (DBS). This method is based on reference (20.1). References (20.2) and (20.3) are related test methods from MIL-STD-286B.

20. APPLICABLE DOCUMENTS

20.1 Analysis Procedure No. 2-83, "Analysis procedure for propylene glycol dinitrate (PGDN) and 2-nitrodiphenlyamine (2NDPA) Stabilizer in Otto Fuel II by high performance liquid chromatography (HPLC)", Naval Undersea Warfare Engineering Station (NUWES) Chemistry Lab, 1983.

20.2 Standard Method 208.3.1, "Nitroglycerin, plasticizers, and stabilizers (liquid chromatography method)", MIL-STD-286B, 1 Sept 1980.

20.3 Standard Method 208.5.1, "Nitroglycerin, 2,4-dinitrotoluene, diphenylamine, and dibutylpthalate (liquid chromatography method)", MIL-STD-286B, 1 Sept 1980.

30. TEST PROCEDURE

30.1 Specimen. The specimen shall consist of approximately 250 grams of Otto Fuel II, which shall be divided into multiple samples, each weighing 2.0000 $\pm$ 0.00005 grams.

30.2 Equipment. The equipment shall consist of the following:

30.2.1 A high pressure liquid chromatograph equipped with the following components:

30.2.1.1 Constant mobile phase delivery system, such as Waters model 6000A solvent delivery system or the eqivalent.

30.2.1.2 Differential refractive index (RI) detector maintained at constant temperature by means of a thermostatted water bath, such as Waters model R401 or Perkin-Elmer model LC-25 or equivalent.

30.2.1.3 Fixed volume sample injector system with sample injection volume of 100-200 microliters. For maximum precision an automatic injector must be used, such as the Waters model 710B "WISP" or equivalent.

30.2.1.4 Constant temperature heated column oven maintained at or slightly above ambient temperature. If this is not available, as an alternate the following conditions must be met: (1) insulation of columns and solvent reservoirs; (2) thermostatic control of RI detector; (3) constant temperature maintained in the room which houses the analytical equipment. Temperature control is especially important when an RI detector is used, and should be maintained to ± 0.1 degree C.

30.2.1.5 Electronic integrator which receives signal data from the detector, and which has the ability to detect chromatographic peak onset, peak maximum, baseline return and peak area, and which can display chromatograms and report peak areas, response factors, sample and operator ID numbers, and date and time of analysis, such as Waters model 730 data module, the Hewlett-Packard model 3390A integrator or equivalent.

30.2.1.6 Two high-efficiency 3.9 by 15-cm chromatography columns in tandem, packed with spherical 5-micron reverse-phase packing, such as Waters REVSOLVE C-18 column. Any alternative column set must be demonstrated to have resolving power equal to or greater than this column set, for Otto Fuel II samples.

30.2.2 Analytical balance accurate to ± 0.0005 grams.

30.2.3 Constant temperature circulating water bath for RI detector capable of maintaining a temperature constant to ± 0.1 degree C, such as HAAKE model K-2/RD or equivalent.

30.2.4 Pressure filtration apparatus containing disposable 0.45-micron filter pad.

30.2.5 100-ml Class A volumetric flasks.

30.2.6 HPLC solvent degasser, such as ultrasonication device.

30.3 Reagents and solvents shall be as follows:

30.3.1 Acetonitrile, HPLC grade.

30.3.2 Water, HPLC grade.

30.3.3 Two Class A Otto Fuel II standard solutions. These should be actual Class A Otto Fuel II samples prepared by the Otto Fuel II supplier, with known and reliable values for PGDN and 2NDPA determined by nitrometer in accordance with this specification. The percent PGDN in the two standards should to the greatest extent possible bracket the acceptable limits of PGDN in Otto Fuel II (75.8% to 76.2%), and should be known to ± 0.1%/

30.3.4 Reagent grade butyrophenone.

30.4 Procedure. The procedure shall be as follows:

30.4.1 Preparation of mobile phase, standards,and sample solutions

30.4.1.1 Mobile phase. Prepare a mixture of 75 parts by weight acetonirile and 25 parts by weight water. Filter the solvent mixture through a 0.45-micron filter pad. Degas the solvent using ultrosonic apparatus. This mixture will be HPLC mobile phase and also the source of solvent for the standard and sample solutions. NOTE: depending on ana;lytical instrumentation, setup and conditions, better resolution of the peaks in the chromatogram may be obtained if the relative amounts of acetonitrile and water in the mobile phase are varied from 75 to 25. The exact composition of the mobile phase should be known accurately, be known accurately, be kept constant, and should reflect optimum peak resolution. In no case should the concentration of water in the mobile phase exceed 30 percent, because of the insolubility of Otto Fuel II in such solutions.

30.4.1.2 Internal standard solution preparation. Weigh out 0.900 g of butyrophenone in a 2-liter volumetric flask and dilute to mark with the mobile phase solution. Allow the butyrophenone to dissolve and mix thoroughly to attain a homegeneous solution. NOTE: Depending on analytical instrumentation, setup and conditions, more precise results may be obtained using an internal standard technique which gives the more precise results should be used. If the external standard technique is used, an internal standard solution is not necessary, and the mobile phase solution is used to prepare standards and samples.

30.4.1.3 Standard solution preparation.

a. Weigh out 2.0000 $\pm$ 0.0005 g of each of the two Otto Fuel II standards into separate clean 100 ml volumetric flasks. Prepare triplicates of each standard.

b. Dilute to 100 ml with internal standard or mobile phase solution. Mix thoroughly by shaking to attain a homgeneous solution. Stopper the flasks to avoid changes in PGDN and 2NDPA concentration.

c. Filter the preparation through a 0.45-micron filter and collect it in a clean, capped vial or stoppered flask.

d. Label all standards. Store standard solutions in a cool, ventilated area.

30.4.1.4 Unknown preparation.

a. Weigh out 2.0000 ± 0.0005 g of each of the Otto Fuel II unknowns into separate clean 100 ml volumetric flasks. Prepare triplicates of each unknown.

b. Dilute to 100 ml with internal standard or mobile phase solution. Mix thoroughly by shaking to attain a homogenous solution. Stopper the flasks to avoid changes in PGDN and 2NDPA concentration.

c. Filter the preparation through a 0.45-micron filter and collect it in a clean, capped vial or stoppered flask.

d. Label all preparations. Store sample solutions in a cool, ventilated area.

30.4.2 Instrumental operation.

30.4.2.1 Instrumental initialization.

a. Place the filtered, degassed mobile phase in its temperature-controlled environment and connect to the solvent delivery system.

b. Connect the precolumn and analytical columns series according to the manufacturer's specifications.

c. Flush the reference side of the differential refractive index detector with the mobile phase.

d. Allow the system to come to temperature aquilibrium, at the pre-selected temperature (ambient or slightly above).

e. Pump the mobile phase through the system at a rate of 1.0 ml per min. for a minimum of 30 minutes.

f. Zero the detector in accordance with the manufacturer's instructions. (When a Waters 730 data module is used, the zero point input reading is 5000 ± 20).

g. Set the detector to the sensitivity to be used for the chromatographic analysis. (When a Waters R401 RI detector is used, this is an attenuation factor of 32). Establish a stable baseline according to manufacturer's instructions. Re-zero the detector.

h. Flush the injector with clean mobile phase.

i. Depending on analytical instrumentation, setup and conditions, better peak resolution of chromatograms may be achieved if a programmed mobile phase flow control is used than if constant 1.0 ml per min. flow is used. The table below illustrates typical flow control conditions:

TIME, min	FLOW, ml per min	CURVE
Initial	1.00	-
10.00	1.50	linear
11.00	1.50	linear
30.00	1.00	linear

Flow control may also be useful in reducing analysis time. Set up the flow control parameters, if u_ed, or set the solvent delivery system to deliver mobile phase at a constant 1.0 ml per min. Constant flow at other than 1.0 ml per min. may be employed if this improves chromatogram quality.

j. Enter the response factors and elution time for PGDN and 2NDPA into the integrator. These are starting volues only with will be modified as the unknowns and standards are analyzed.

30.4.2.2 Calibration and unknown analysis.

a. One or two different unknowns can be run in a single series. The sequence of injections is as follows:

1) 200 microliters of Otto Fuel standard (to prepare column)
2) 200 microliters of Otto Fuel standard (to prepare column)

3) Standard A, first solution of the three prepared
4) Standard B, first solution
5) Unknown A, first solution
6) Unknown B (if there is one), first solution

7) Standard A, second solution
8) Standard B, second solution
9) Unknown A, second solution
10) Unknown B, second solution

11) Standard A, third solution
12) Standard B, third solution
13) Unknown A, third solution
14) Unknown B, third solution

15) Standard A, first solution
16) Standard B, first solution

The first two runs are for column preparation only and analysis data is not collected. Each unknown is analyzed three times, and is bracketed by the standards, which are run four times. The chromatographic trace for each sample must have returned to baseline before the next sample is analyzed. NOTE: Otto Fuel II contains the diluent DBS, which takes a long time to be eluted from thecolumn (typically over 12 minutes, where the PGDN, 2NDPA, and internal standard peaks all elute before 4 minutes). The DBS must have eluted from the column before the next sample is injected. For maximum precision,

the detector should be rezeroed before each injection, and the time allowed between each injected must be held constant.

30.4.3.1 The experimentally determined calibration factors for the Otto Fuel II standards are examined to assure that they do not change over the course of the analytical run by more than ± 0.2% for PGDN and ± 2% for 2NDPA (relative). If they do, the cause of the drift in values is determined and corrected, and the samples are re-analyzed.

30.4.3.2 If the electronic integrator has the ability to continually up-date its values for response factors as each Otto Fuel II standard is run, this may be used to obtain the response factors for each set of unknown solutions. In this case, the composition of the unknown solutions will be determined using response factors based only on the standard solutions run immediately before or after or both before and after that unknown.

30.4.3.3 If the integrator does not have continual update capability, or if continual updating of response factors proves not to yield the more precise composition values for the unknowns, the response factors for the unknowns, the response factors for PGDN and 2NDPA may be calculated from the averages from all four analyses of each of the two standards. These average response factors will then be applied to each of the unknown analyses run.

30.4.3.4 Either internal or external standard calculations with be performed using the appropriate response factors for each unknown, depending on which yields more precise results. Examples of internal and external standard calculations are given in reference (20.3) and (20.3) most electronic integrators will perform these calculations automatically.

30.4.3.5 If the range of calculated composition values for the Otto Fuel II unknown is more than ± 0.2% for PGDN and ± 2% for 2NDPA (relative), the cause of the variability is determined and corrected, and the samples are re-analyzed.

30.4.3.6 The average (mean) of the calculated composition values for PGDN and 2NDPA in Otto Fuel II are reported as the analytically determined percent compositions.

40. NOTES

40.1 The temperature of choice for the analysis is 18 ± .1 degree C.

40.2 Freshly prepared standard and unknown solutions should always be prepared for each analytical period. Do not use old standard or unknown solutions.

40.3 Do not exceed 4500 psi system pressure.

40.4 After analyses are completed, clean and flush all systems with clean mobile phase.

40.5 Because of the need for maximum reproductibility in sample handling, injection volume, and the time interval between injections for all fourteen samples of an analytical sequence, and automatic injector system must be used.

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3a. NAME OF SUBMITTING ORGANIZATION	4 TYPE OF ORGANIZATION *(Mark one)*
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	☐ MANUFACTURER
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