

MIL-STD-650
 NOTICE 2
 3 April 1972

MILITARY STANDARD

EXPLOSIVE: SAMPLING, INSPECTION AND TESTING

TO ALL HOLDERS OF MIL-STD-650

1. The following pages of MIL-STD-650 have been revised and supersede the pages listed:

New Page	Date	Superseded Page	Date
iii	3 April 1972	iii	3 August 1962
iv	"	iv	"
v	"	v	"
vi	"	vi	"
vii	"	vii	"

2. The following methods have been revised and supersede the methods listed:

Revised Method	Page	Date	Superseded Method	Superseded Page	Date
101.301	1	3 April 1972	101.3	1	3 August 1962
101.4.1	2	"	101.4	2	"
102.3.1	1	"	102.3	1	"
108.1.1	1	"	108.1	1	"
301.6.1	1	"	201.6	1	"
413.2.1	1	"	413.2	1	"

3. The following is a cumulative list of earlier changes:

a. Superseded pages

New Page	Date	Superseded Page	Date
V	31 August 1970	V	3 August 1962
VI	31 August 1970	VI	3 August 1962

b. New Method

New Method	Title	Date
T109.6	Rate of Detonation	31 August 1970

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4. Retain this notice and insert before the table of contents.

5. Holders of MIL-STD-650 will verify that page changes and additions indicated above have been entered and will destroy the previous notice (Notice page only). The latest notice (notice page) will be retained as a check sheet. This issuance, together with appended pages, is a separate publication. Each notice is to be retained by stocking points until the Military Standard is completely revised or cancelled.

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METHOD 101.3.1

MOISTURE DISTILLATION METHOD

1. SCOPE.

1.1 This method is used to determine moisture in an explosive, when appreciable amount of moisture is present and a relatively large amount of sample is available.

2. SPECIMEN.

2.1 The specimen shall consist of approximately 50 gm. of explosive. Weighed within 10 mg.

3. APPARATUS.

3.1 Round bottom "Balloon Type Flask" with a short ring neck, 500 ml.

3.2 Condenser (Liebig Type, 250 mm Straight tube with the lower end cut obliquely.

3.3 Drying tube containing calcium chloride.

3.4 Steam, hot water or oil bath.

3.5 Moisture tube (fig. 1) cleaned with bichromate cleaning solution.

4. MATERIALS.

4.1 Triethylene Glycol

5. PROCEDURE.

5.1 Place the specimen in the 500 ml balloon flask and add 200 ml of carbon tetrachloride.

5.2 Connect the flask with the moisture tube. Connect a calcium chloride tube to the top of the condenser to keep out atmospheric moisture.

5.3 Maintain a sufficient stream of cold water through the condenser jacket to condense all the solvent.

5.4 Heat the flask by means of a suitable bath so that the distillate falls from the end of the reflux condenser in a steady stream. Continue the heating for 30 minutes, then remove the apparatus from the bath and cool to room temperature.

5.5 Disconnect the apparatus from the condenser and tilt tube if necessary so that sufficient solvent drains back into the flask to bring the water layer into the graduated tube. Drain the upper part of the tube completely so that all the water is collected in the graduated area.

5.6 Hold the tube in a vertical position and read (1) The top of the upper meniscus of the water layer (straight across) and (2) The top of the carbon tetrachloride layer, estimating to 0.01 ml on each reading.

Note. Should the water layer extend beyond the graduated portion of the tube, repeat the determination using a smaller sample.

5.7 Determine the difference in the readings and record the difference as the volume of water in the specimen.

5.8 Considering one millimeter of water as equal to one gram, calculate (by weight) the percentage of moisture in the specimen.

5.9 Calculation.

$$\text{Percent moisture} = 100 \frac{V}{W}$$

where:

V = corrected volume of water, ml.

W = Weight of sample, gm.

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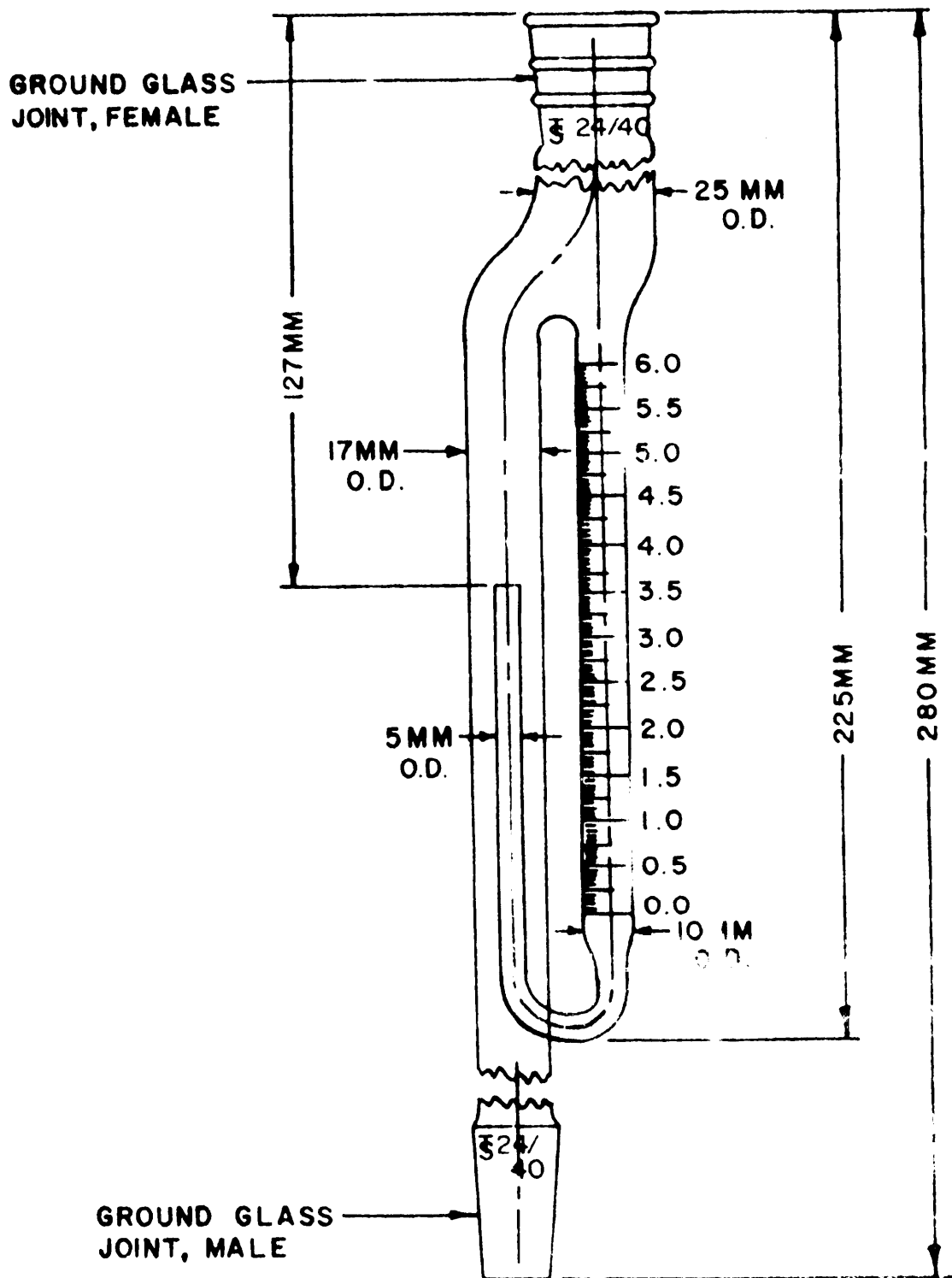


FIGURE 1. *Moisture tube.*

METHOD 101.4.1

MOISTURE (KARL FISCHER METHOD)

1. SCOPE.

1.1 This is a general method for determining moisture of explosives that do not react with the reagents used and which dissolve readily in the reagents or from which the moisture can be removed by extraction.

Note. Although the application of the Karl Fischer Reagent for determining the moisture content of explosives is general in scope, its chemical reactivity with materials other than moisture imposes certain limitations that must be taken into account by modifications of the method for the purpose of adaptation to specific applications. Detail specifications should be consulted for adaptations to materials that require modifications of the procedure listed herein.

2. SPECIMEN.

2.1 The specimen shall consist of approximately 8 to 10 gm. of explosive, weighed within 0.2 mg.

Note. The specimen should contain not more than 0.01 gm. of water. The specimen weight should be adjusted after a preliminary determination, so as not to exceed the 0.01 gm. maximum.

The sample should be placed in a glass stoppered weighing bottle. The weighing bottle and contents are weighed, before and after the introduction of the specimen into the beaker through the sample porthole of the rubber stopper (fig. 3) obtaining the specimen weight by the difference in the weighing taken.

Loss of 0.05 gm. of the specimen has no appreciable effect on the accuracy of the determination.

3. APPARATUS.

3.1 Titration assembly The apparatus shown on figures 1, 2, and 3 shall be used. The rubber stopper shall be fitted with the electrodes, buret tips, and an air vent to a drying tube, and placed in the iron ring. The sample porthole shall be closed with a glass

stopper and the iron ring shall be fastened to a ring stand. The ring with the stopper shall remain fixed throughout the determination. The titration vessel shall remain fitted onto the stopper, and the magnetic stirrer placed beneath the beaker, to serve as a support as well as a stirring mechanism.

3.2 Titration vessel. A 200 ml tall form beaker, without pouring lip, as shown on figure 2, has been found satisfactory. The dried beaker shall be taken from the drying oven and immediately connected to the stopper in the titration assembly. As the beaker cools, dry air will be drawn in through the drying tube at the vent. A minimum of four dry beakers will be required.

3.3 Buret assembly. Two all glass buret assemblies (one for Karl Fischer reagent and one for water-in-methanol solution), to permit introduction of the reagent into the titration vessel (200 ml beaker, Fig. 2) by gravity from a reservoir, without exposure to atmospheric moisture, shall be used. The Machlett automatic burets have been found satisfactory. The reservoirs connected to the burets with all glass tubing shall be protected against atmospheric moisture by drying tubes and protected from light with black paint or dark paper on the outside of the reservoir bottles.

3.4 Electrodes. Two platinum electrodes, 0.016 inch diameter, sealed-in-glass, with mercury connections, shall be used.

3.5 Timer. Any interval timer which can measure time to the nearest second and can record minutes, shall be used.

4. MATERIALS.

4.1 Karl Fischer reagent. To make 1000 ml

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of the reagent, 264 gm. of anhydrous pyridine of highest purity shall be placed in a 1000 ml Florence flask, and 61 gm. of liquid sulfur dioxide (SO₂) shall be added by inserting the cylinder and allowing the SO₂ to in through a glass tube into the pyridine in the flask on a balance pan. A 664 ml. portion of anhydrous methanol (not more than 0.02 percent moisture) shall be added. A calcium chloride drying tube shall be attached and the flask chilled in an iced bath. When thoroughly chilled 65 gm. of chemically pure (c.p.) re-sublimed iodine shall be added at one time. The solution shall be kept chilled and agitated by swirling until the iodine has dissolved completely. Due to its rapid initial deterioration, the reagent should not be used for 24 hours. One ml. is equivalent to approximately 0.0025 gm. water. Alternatively commercially available Karl Fischer reagent may be used.

4.2 Standard water-in-methanol solution. To a previously dried 1000-ml. volumetric task, 2 ml. of water shall be pipetted, immediately diluted to the mark with anhydrous methanol, stoppered, and mixed thoroughly.

4.3 Special solvent. Equal volumes of anhydrous methanol and benzene shall be mixed. If necessary, the solvents may be dried by distillation.

Note. The special solvent or solvents employed for each adaptation will vary depending on the nature of the material being tested. Some of the solvents commonly employed here: Methanol, Ethanol, Dioxane, Diethyl Ether, Acetic Acid and Dimethyl-formamide.

4.4 Sodium tartrate dihydrate. The water content of this material shall be established by determining the loss in weight on drying at 105°C. for 3 hours. The water content of this material shall be 15.66 ± 0.05 percent. If it is not, a fresh bottle of the material shall be used.

5. PROCEDURE.

5.1 Standardize the solutions as follows:

5.1.1 Karl Fischer reagents

a. Transfer a 100 ml. portion the special solvent to one of the dry 200 ml. beakers (see fig. 2) through the sample porthole of the rubber stopper.

b. Add a slight excess of Karl Fischer reagent and titrate with standard water-in-methanol solution to the preliminary end point exactly as specified in 5.3.

c. Immediately upon reaching the preliminary end point release the timer and take buret readings.

d. Add an accurately measured portion of approximately 10 ml. of the Karl Fischer reagent and titrate the solution immediately with standard water-in-methanol solution.

e. Control these operations so as to reach the second end point within 3.5 minutes plus or minus 15 seconds of the preliminary end point.

Note. This second end point may be used as the preliminary end point for a check standardization. In this case, the timer shall be returned to zero and released as soon as the second end point has been reached, and the titration for the third and point shall be completed within an additional 3.5 minutes.

f. Calculate the ml. of standard water-in-methanol solution per ml. of Karl Fischer reagent. This is the ratio R referred to in the calculation of F. Check determinations should agree within 0.010.

Note. Because the ratio is constantly changing due to deterioration of the reagent, the standardization shall be made at least once a day, more often for more accurate work.

1.2 Standard water-in-methanol solution.

a. Transfer a 100 ml portion of special solvent to another one of the dry 200 ml. beakers through the sample porthole of the rubber stopper.

b. Titrate with Karl Fischer reagent and standard water-in-methanol solution into the preliminary end point, exactly as specified in 6.2.

c. Release the timer and take buret readings immediately.

Method 101.4.1

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METHOD 102.3.1

ACIDITY OR ALKALINITY

(COMPLETE SOLUTION METHOD)

1. SCOPE.

1.1 This method is used to determine acidity or alkalinity of explosives that readily dissolve in a solvent or combination of solvents.

Note. This method is applicable to a variety of acidity or alkalinity determinations, and is not intended to restrict or limit such factors as the solvent or solvents used, the indicator, or the acid or base being determined. For specific instructions refer to the applicable explosive specification.

2. SPECIMEN.

2.1 The specimen shall consist of approximately 5 to 10 gm. of the explosive weighed to within 10 mg.

3. APPARATUS.

3.1 Steam bath.

3.2 Beaker, 1000 ml.

3.3 Buret, 25 ml.

4. MATERIALS.

4.1 Suitable solvent or solvents such as acetone, aliphatic naphtha, benzene, toluene as specified in the applicable method or explosive specification.

4.2 Indicator see *Note* in Method No. 102.1 following Paragraph 4.1.

4.3 Standard solution.

a. Sodium Hydroxide, 0.1N standard solution as specified in Method No. 602.1.

b. Hydrochloric acid 0.1N standard solution as specified in Method No. 601.1.

5. PROCEDURE.

5.1 Transfer the specimen to a beaker.

5.2 Add a sufficient amount of solvent to dissolve the sample.

5.3 Heat the beaker and contents on a steam bath until the material is dissolved.

Note. In cases where more than one component is present in a composition and the components are not mutually soluble in a common solvent, more than one solvent must be employed.

5.4 Add a volume of distilled water to the mixture, equal to the volume of solution(s) in the beaker. stir and add an additional 250-300 ml. of water.

5.5 Allow the mixture to cool to room temperature.

5.6 Add twelve drops of indicator to the water layer in the beaker.

5.7 Note the color of the solution after the addition of the indicator. If the solution is acid, titrate with standard 0.1N sodium hydroxide; if it is alkaline, titrate with standard 0.1N hydrochloric acid.

5.8 Run a blank determination following the same procedure without the sample to correct for the acidity or alkalinity of the solvents.

5.9 Calculate the percent acidity or alkalinity from the volume of standard solution used.

5.10 Calculation.

$$\text{Percent acidity or alkalinity} = \frac{(V-v) NF}{W}$$

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Where:

V = ml. of standard solution used in
titrating sample

v = ml. of standard solution used in
titrating blank

N = normality of standard solution.

W = weight of specimen.

F = factor for acid or base, Method
No. 102.1, Paragraph 5.9.

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METHOD 108.1.1

SOLVENT EXTRACTION (SELECTIVE SOLVENT METHOD)

1. SCOPE.

1.1 This method is used for the quantitative determination of components in explosive composite as by the selective extraction technique.

1.2 This method is used for extracting soluble ingredients from explosive compositions for use in subsequent determinations.

1.3 This method is used for extracting soluble explosive components which would interfere with subsequent determinations.

Note. This method is applicable to a variety of extraction conditions, and is not intended to restrict or limit such factors as the solvent or apparatus used, or the time of extraction. For specific instructions refer to the applicable explosive specification.

2. SPECIMEN.

2.1 The specimen shall consist of an accurately weighed portion of the explosive as specified in the applicable method or specification.

3. APPARATUS.

3.1 Vacuum suction apparatus.

3.2 Filtering crucible of suitable porosity according to the particle size of the material being tested.

3.3 Beaker.

3.4 Stirring rod.

3.5 Oven.

3.6 Steam bath.

3.7 Dessicator with indicating desiccant.

4. MATERIALS.

4.1 Extracting solvent, such as acetone, aliphatic naphtha, toluene, or benzene as specified in the applicable method or explosive specification.

5. PROCEDURE.

5.1 Transfer a specimen to a beaker or directly to a tared filtering crucible.

a. When a beaker is employed, add a sufficient amount of the appropriate solvent and allow a sufficient contact period for the soluble ingredient of the composition to dissolve (to facilitate solution heat the beaker on a steam bath and stir with a glass rod). Filter the mixture through a tared filtering crucible with the aid of a stream of the suitable solvent and a stirring rod.

b. If the specimen is weighed directly in a crucible, add a sufficient quantity of solvent, (solvent may be added hot to facilitate solution) in small portions, to the crucible and allow the solvent to drain through before applying suction.

5.2 Wash the residue in the crucible with portions of the suitable solvent until extraction is complete (which may be indicated by any suitable test).

5.3 When extraction is complete proceed according to one or more of the applicable steps that follow:

a. Evaporate the solvent using a stream of dry air, and retain the dry residue for further determinations.

b. Aspirate the crucible and residue to remove all volatile solvents. Retain the residue for further determinations.

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c. Dry the crucible and residue by aspirating to remove all volatile solvents, then dry in an oven at an appropriate temperature, cool in a desiccator and weigh. Calculate the loss in weight an percent constituent extracted by one of the suitable solvents as follows:

$$\text{Percent constituent} = \frac{(A - B) 100}{W}$$

where:

A = weight of crucible and residue,
gm.

B = weight of crucible, gm.

W = weight of sample, gm.

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METHOD 301.6.1

SAMPLE PREPARATION (SOXHLET EXTRACTION)

1. SCOPE.

1.1 This method is used for extracting soluble ingredients from an explosive for use in subsequent determinations.

2. SPECIMEN.

2.1 The specimen shall consist of an accurately weighed portion of the explosive, suitable for the subsequent determinations.

3. APPARATUS.

3.1 Soxhlet or equivalent extractor.

3.2 Extraction thimble.

3.3 Steam or hot water bath.

4. MATERIALS.

4.1 Extraction solvent, such as ether, aliphatic naphtha or methylene chloride as specified in the applicable method or explosive specification.

5. PROCEDURE.

5.1 Transfer the specimen to an extraction thimble.

5.2 Place the thimble in a Soxhlet extraction apparatus.

5.3 Place 200 ml. of the extraction solvent in the flask of the extraction apparatus.

5.4 Place the extraction apparatus on a steam or hot water bath and adjust the temperature of the bath so that the solvent drips from the end of the condenser at the rate of 2 to 3 drops per second.

5.5 continue the extraction for at least 16 hours.

5.6 Test for completeness of extraction in the following manner:

a. Allow the extraction to become almost full of the solvent.

b. Separate the extractor from the flask.

c. Draw one or two drops of the solvent from the extractor.

d. Transfer the drops to a clean spot plate.

e. Qualitatively test the drops to determine if they contain any of the solvent soluble material.

5.7 Continue the extraction until all solvent soluble material has been removed.

5.8 When extraction is complete dismantle the extractor and retain the extract and the residue for subsequent determinations.

METHOD 413.2.1

TRINITROLUENE (IN TETRYTOL)

1. SCOPE.

1.1 This method is used to determine the trinitrotoluene content in tetrytol.

2. SPECIMEN.

2.1 The specimen shall consist of approximately 0.35 gm. of the explosive, weighed to within 0.2 mg.

3. APPARATUS.

3.1 Volumetric flask, 100 ml.

3.2 Ice bath.

3.3 Pipette, 100 ml.

3.4 Rubber tubing.

3.5 Cotton filter plug.

3.6 Erlenmeyer flask.

3.7 Oven.

4. MATERIALS.

4.1 Saturated Solution of Benzene

5. PROCEDURE.

5.1 Transfer the specimen to a previously weighed 100 ml. volumetric flask having a short neck and ground glass stopper.

5.2 Add approximately 100 ml. of carbon tetrachloride to the flask and heat the mixture to boiling in order to insure the solution of all TNT present.

5.3 Stopper the flask, allow the flask and

contents to cool to room temperature and weigh.

5.4 Immerse the bulb of the flask in an ice bath for not less than 4 hours, agitating the contents every 15 minutes or half hour.

5.5 At the end of this time take a sample of the supernatant liquid, which should be between 0.0° and 0.5°C. in the following manner:

a. Attach a 100 ml. pipette to a short piece of rubber tubing containing a cotton filter plug.

b. Insert the end of the tubing into the supernatant liquid, quickly draw up as large a portion of the liquid as possible into the assembly and immediately transfer this to a dried and weighed Erlenmeyer flask having a tight fitting stopper.

5.6 Allow the flask and contents to warm up to room temperature, weigh, evaporate the carbon tetrachloride by means of a current of dry air and dry the residue at 60°C. for two hours and weigh.

5.7 Calculate the TNT content of the sample as follows:

$$\text{Percentage of TNT} = \frac{W_a (100W_n - 0.0073W_c)}{W_c \times W_t}$$

where:

W_a = total weight of carbon tetrachloride used.

W_c = weight of carbon tetrachloride in portion of supernatant liquid taken.

W_t = weight of the tetrytol sample taken for analysis.

W_n = weight of residue from portion of supernatant liquid taken.

SPECIFICATION ANALYSIS SHEET		Form Approved Budget Bureau No. 22-R255
<p>INSTRUCTIONS: This sheet is to be filled out by personnel, either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity. Comments and suggestions submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or serve to amend contractual requirements.</p>		
SPECIFICATION		
ORGANIZATION		
CITY AND STATE		CONTRACT NUMBER
MATERIAL PROCURED UNDER A <input type="checkbox"/> DIRECT GOVERNMENT CONTRACT <input type="checkbox"/> SUBCONTRACT		
1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE? A. GIVE PARAGRAPH NUMBER AND WORDING.		
B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES		
2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID		
3. IS THE SPECIFICATION RESTRICTIVE? YES NO (If "yes", in what way?)		
4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)		
SUBMITTED BY (Print or typed name and activity - Optional)		DATE

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