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New Page	Date	Superseded Page	Date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Revised Method	Page	Date	Superseded Method	Superseded Page	Date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a. Superseded pages

New Page	Date	Superseded Page	Date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

FSC 1376

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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CONTENTS

Page
ALPHABETICAL INDEX OF TEST METHODS iii
NUMERICAL INDEX OF TEST METHODS vi
INTRODUCTION 1
SAMPLING AND INSPECTION 3
TESTING 5
TEST METHODS vi
Group 100 — General Test Methods
200 — Physical Test Methods
300 — Sample Preparation
400 — Chemical Test Methods
500 — Stability, Sensitivity and Surveillance Test
600 — Standard Solution
700 — Standard Indicators

ALPHABETICAL INDEX OF TEST METHODS

Title — Method No.
Acidity or Alkalinity (Hot Aqueous Extraction Method) 102.1
Acidity or Alkalinity (Cold Extraction Method) 102.2
Acidity or Alkalinity (Complete Solution Method) 102.3.1
Ammoniacal Nitrogen 403.2
Ammoniacal Nitrogen (Kjeldahl Method) 403.1
Ammonium Thiocyanate Indicator Solution (20 percent) 707.1
Antimony Sulfide (Potassium Bromate Method) 401.1
Ash 107.1
Average Particle Diameter (Fischer Sub-Sieve Sizer) 206.1
Barium Diphenylanine Sulfonate Indicator Solution 706.1
Barium Salts (Chromate Method) 404.1
Barium Salts (Sulfate Method) 405.1
Brisance 508.1
Bulk or Apparent Density (Dry Method) 201.3
Bulk or Apparent Density (Standard Vessel Method) 201.1
Bulk or Apparent Density (Wet Method) 201.2
Density (in Vacuo) 202.1

SUPERSEDES PAGE iii OF 3 AUGUST 1962

ALPHABETICAL INDEX OF TEST METHODS - Continued

Title	Method No.
Dissolution	301.1
Dissolution-Extinction	301.2
Explosion Temperature Test	606.1
Extraction	301.3
Ferric Ammonium Sulfate (0.15N Standard Solution).	603.1
Granulation (Dry)	204.1
Granulation (Wet)	204.2
100°C Heat Test Method	502.1
Hydrochloric Acid (0.1N Standard Solution)	601.1
Hygroscopicity (Equilibrium Method)	208.1
Ignition	301.8
Impact Sensitivity Test (U.S. Bureau of Mines Apparatus)	606.1
Initiating Efficency (Sand Test Method)	309.1
Inorganic Chlorides	406.1
Insoluble Material (Solvent Insoluble Method)	105.1
Insoluble Particles or Grit (Solvent Extraction Method)	106.1
75°C. International Test Method	501.1
Leaching	301.4
Lead Azide (Eudiometer Method)	407.1
Lead Styphnate, Normal (Spectrophotometric Method)	408.1
Melting Point (Capillary)	209.1
Mercury Fulminate	410.1
Methyl Orange Indicator Solution	704.1
Methyl Red Indicator Solution	702.1
Moisture (Karl Fischer Method)	101.4.1
Moisture (Dessication Method)	101.2
Moisture (Dish and Cone Method)	101.1
Moisture (Distillation Method)	101.3.1
Moisture and Volatiles (Oven Method)	101.5
Moisture and Volatiles (Vacuum Oven Method)	101.6
Multiple Solvent Extraction	301.7
Nitrogen Content	414.1
Nitro-Nitrogen Content (Titunous Chloride Method)	409.1
Organic Destruction and Sample Dissolution	201.5
Particle Size (Microscopic)	206.1

ALPHABETICAL INDEX OF TEST METHODS—Continued

Title	Method No.
Phenophthalein Indicator Solution	703.1
pH Value (Electrometric Method)	103.1
Plasticity (Elongation)	211.1
Plasticity (Modulus of Compressability)	211.1
Potassium Bromate (0.06N Standard Solution)	610.1
Reactivity Test	504.1
Setting	207.1
Sensitivity to Initiation (Sand Test Method)	507.1
Sodium Diphenyl Benzidine Sulfonate Indicator Solution	706.1
Sodium Hydroxide (0.lN Standard Solution)	602.1
Sodium (Magnesium Uranyl Acetate Method)	417.1
Sodium Thiosulfate (O.1N Standard Solution)	613.1
Solidification Point	210.1
Volubility (Leaching Method)	104.1
Solvent Extraction (Selective Solvent Method)	108.1
Soxhlet Extraction	301.6
Specific Gravity (Bottle Method)	201.2
Specific Gravity (Mercury Displacement)	203.1
Starch Indicator Solution	701.1
Sulfate	411.1
Sulfuric Acid (O.lN Standard Solution)	612.1
Tetracene (Polarograph Method)	416.1
Titanous Chloride (0.2N Standard Solution)	606.1
Total Lead (Chromate Method)	402.2
Total Lead (Sulfate Method)	402.1
Trinitrotoluene	413.1.1
Trinitrotoluene (in Tetrytol)	413.2
100° C and 120°C. Vacuum Stability Test	503.1
Viscosity (Efflux)	212.1

SUPERSEDES PAGE V OF 3 AUGUST 1962

NUMERICAL INDEX OF TEST METHODS

Group 100 — General Test Methods

Method No.	
101.1	Moisture (Dish and Cone Method)
101.2	Moisture (Desiccation Method)
101.3.1	Moisture (Distillation Method)
101.4.1	Moisture (Karl Fischer Method)
101.5	Moisture and Volatiles (Oven Method)
101.6	Moisture and Volatiles (Vacuum Oven Method)
102.1	Acidity or Alkalinity (Hot Aqueous Extraction Method)
102.2	Acidity or Alkalinity (Cold Aqueous Extraction Method)
102.3.1	Acidity or Alkalinity (Complete Solution Method)
103.1	pH Value (Electrometric Method)
104.1	Solubility (Leaching Method)
105.1	Insoluble Material (Solvent Insoluble Method)
106.1	Insoluble Purchases or Grit (Solvent Extaction Method)
107.1	Ash
108.1.1	Solvent Extraction (Selective Solvent Method)

Group 200 — Physical Test Methods

Method No.	
201.1	Bulk or Apparent Density (Standard Vessel Method)
201.2	Bulk or Apparent Density (Wet Method)
201.3	Bulk or Apparent Density (Dry Method)
202.1	Density (In Vacuo)
203.1	Specific Gravity (Mercury Displacement)
203.2	Specific Gravity (Bottle Method)
204.1	Granulation (Dry)
204.2	Granulation (Wet)
205.1	Average Particle Diameter (Fisher Sub-Sieve Sizer)
206.1	Particle Size (Microscopic)
207.1	Settling No.
208.1	Hygroscopicity (Equilibrium Method)
209.1	Melting Point (Capillary)
210.1	Solidification Point
211.1	Plasticity (Elongation)
211.2	Plasticity (Modulus of Compressibility)
212.1	Viscosity (Efflux)

NUMERICAL INDEX OF TEST METHODS—Continued

Group 300—Sample Preparation

Method No.

301.1 Dissolution
301.2 Dissolution—Extraction
301.3 Extraction
301.4 Leaching
301.5 Organic Destruction and Sample Dissolution
301.6.1 Soxhlet Extraction
301.7 Multiple. Solvent Extraction
301.8 Ignition

Group 400—Chemical Tee Methods

Method No.

401.1 Antimony Sulfide (Potassium Bromate Method)
402.1 Total Lead (Sulfate Method)
402.2 Total Lead (Chomate Method)
403.1 Ammoniacal Nitrogen (Kjeldahl Method)
403.2 Ammoniacal Nitrogen
404.1 Barium Salts (Chromate Method)
405.1 Barium Salts (Sulfate Method)
406.1 Inorganic Chlorides
407.1 Lead Azide (Audiometer Method)
4081 Lead Styphnate Normal (Spectrophotometric Method)
409.1 Nitro-Nitrogen Content (Titanous Chloride Method)
410.1 Mercury Fulminate
411.1 Sulfates
413.1 Trinitrotoluene
413.2.1 Trinitrotoluene (in Tetrytol)
414.1 Nitrogen Content
416.1 Tetracene (Polarograph Method)
417.1 Sodium (Magnesium Uranyl Acetate Method)

Group 500 — Stability, Sensitivity and Surveillance Tests

Method No.

501.1 75°C. International Test Method
502.1 100°C. Heat Test Method
503.1 100°C. and 120°C. Vacuum Stability Test

NUMERICAL INDEX OF TEST METER—Continued

Method No.	
504.1	Reactivity Test
505.1	Impact Sensitivity Test (U.S. Bureau of Mines Apparatus)
506.1	Explosion Temperature Test
507.1	Sensitivity to Initiation (Sand Test Method)
508.1	Brisance
509.1	Initiating Efficeincy (Sand Test Method)

Group 600—Standard Solutions

Method No.	
601.1	Hydrochloric Acid (0.lN Standard Solution)
6021	Sodium Hydroxide (0.1N Standard Solution)
603.1	Ferric Ammonium Sulfate (0.15N Standard Solution)
605.1	Titanous Chloride (0.2N Standard Solution)
610.1	Potassium Bromate (0.05N Standard Solution)
6121	Sulfate Acid (0.lN Standard Solution)
613.1	Sodium Thiosulfate (0.lN Standard Solution)

NUMERICAL INDEX OF INDICATOR SOLUTIONS

Group 700—Standard Indicators

Method No.	
701.1	Starch Indicator Solution
702.1	Methyl Red Indicator Solution
703.1	Phenolphthalein Indicator Solution
704.1	Tethyl 0range Indicator Solution
706.1	Barium Diphenylamine Sulfonate Indicator Solution
706.1	Sodium Diphenylbenzidine Sulfonate Indicator Solution
707.1	Ammonium Thiocyanate Indicator Solutical

METHOD 101.3.1

MOISTURE DISTILLATION METHOD

1. SCOPE.

1.1 This method is used to detemine 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 nnd 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 bcyond the graduated portion of the tube, repeat the deterrmination 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.

SUPERSEDES PAGE I OF 3 AUGUST 1962

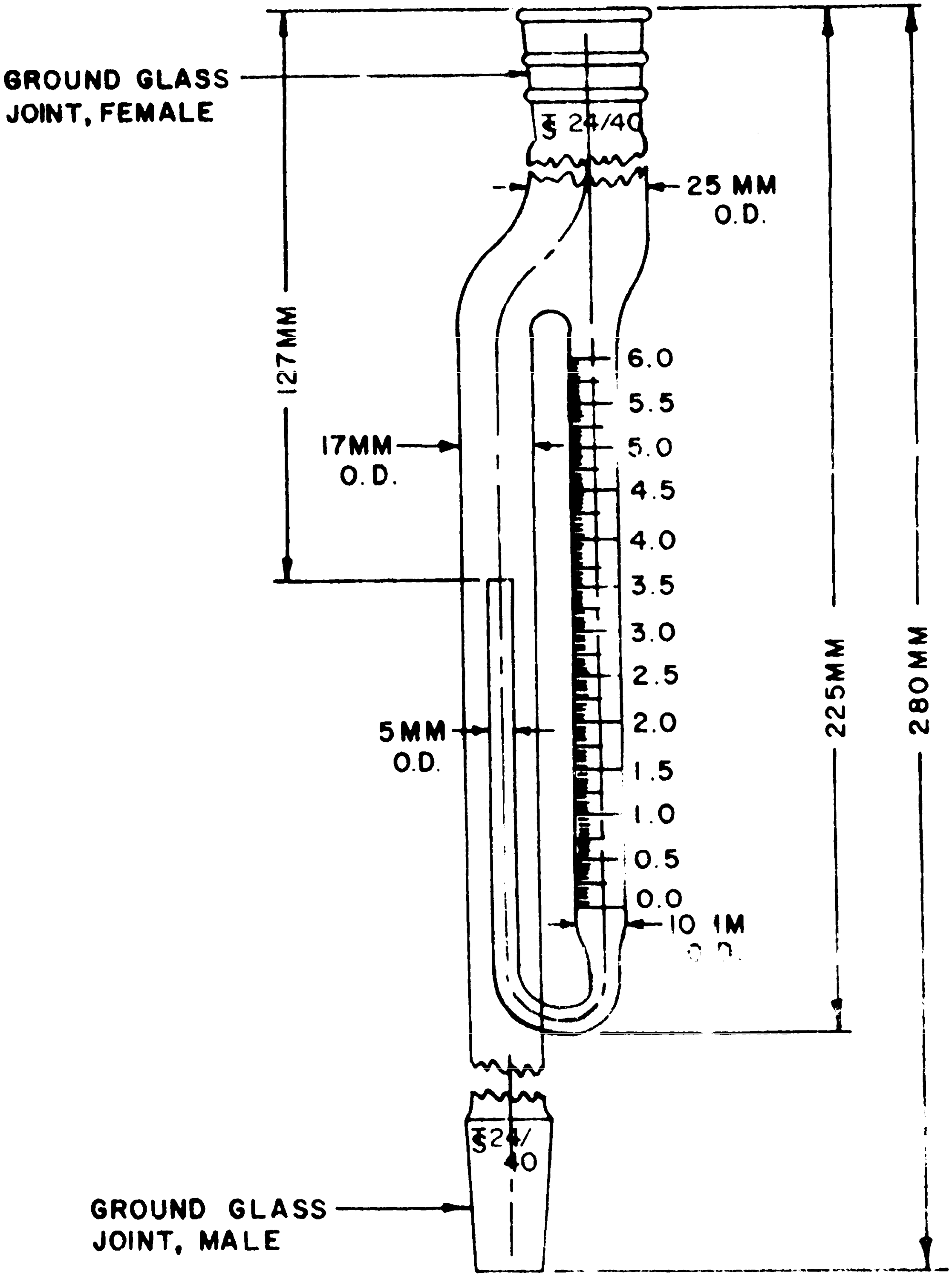

FIGURE 1. *Moisture tube.*

METHOD 101.4.1
MOISTURE (KARL FISCHER METHOD)

1. SCOPE.

1.1 This is a general method ‘or 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 rng.

Note. The specimen should contain not more than 0.01 gm. of water. The specimen weight should be adjusted after a preiminary determination, so as not to exeeed 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 weighinge 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 aseembly. Two all glass buret assemblies (one for Karl Fischer reagent and one for water-in-methanol solution), to permit introduction of the reagent i to 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 conected 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 of the rengent, 264 gm. of anhydrous pyridine of highest purity shall be placed in a 1000 ml Florence flask, and 61 gm. of liquid sulfur dioxide (SO_2) shall be added by in inserting the cylinder and allowing the SO_2 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.) resublimed 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 he used as the preliminary end point for a check standardization. In this case, the timer shall he 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 rcagent. 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.

SUPERSEDES PAGE 2 OF 3 AUGUST 1962.

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 he 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, allphatic 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 souble 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.

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

SUPERSEDES PAGE 1OF 3 AUGUST 1962

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.

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, alphatic 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 extracion 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.

SUPERSEDES PAGE 1 OF 3 AUGUST 1962

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.

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 extration 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.

SUPERSEDES PAGE 1 OF 3 AUGUST 1962

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:

$$\text{Percentage of TNT} = \frac{Wa\,(100Wn - 0.0073Wc)}{Wc \times Wt.}$$

where:

Wa = total weight of carbon tetrachloride used.

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

Wt = weight of the tetrytol sample taken for analysis.

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

SUPERSEDES PAGE 1 OF 3 AUGUST 1962

☆ U. S. GOVERNMENT PRINTING OFFICE: 1972-714-165/1400 1

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 22-R255

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.

SPECIFICATION

ORGANIZATION

CITY AND STATE	CONTRACT NUMBER

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ 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 *(Printed or typed name and activity - Optional)*	DATE

DD FORM 1 JAN 66 **1426** REPLACES EDITION OF 1 OCT 64 WHICH MAY BE USED.

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