

MIL-A-3850

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SUPERSEDING

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MILITARY SPECIFICATION**ACETYLENE BLACK**

This specification has been approved by the Departments of the Army, the Navy, and the Air Force.

1. SCOPE

1.1 This specification covers one grade of acetylene black.

2. APPLICABLE SPECIFICATIONS, STANDARDS, DRAWINGS, AND PUBLICATIONS

2.1 The following specifications and standard, of the issue in effect on date of invitation for bids, form a part of this specification:

SPECIFICATIONS

FEDERAL

RR-S-366 —Sieves; Standard, Testing.

MILITARY

MIL-G-2550 —General Specification for Ammunition Except Small Arms Ammunition.

NAVY DEPARTMENT

General Specifications for Inspection of Material.

STANDARDS

MILITARY

MIL-STD-129—Marking of Shipments.

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

3. REQUIREMENTS.

3.1 Material. — The acetylene black shall be manufactured by the incomplete combustion of acetylene by any suitable method which will produce material conforming to the requirements of this specification.

3.2 Chemical and physical requirements. — The acetylene black shall conform to the chemical and physical requirements shown in table I, when tested in accordance with the applicable test method specified in section 4.

TABLE I. —*Chemical and physical requirements of the acetylene black.*

Property	Requirement	Test method paragraph
Moisture content, percent, maximum -----	0.50	4.4.1
Sulfur content, percent, maximum -----	0.05	4.4.2
Ether-extractable matter, percent, maximum -----	0.50	4.4.8
Volatile matter, percent, maximum -----	2.0	4.4.4
Water-soluble matter, percent, maximum -----	0.10	4.4.5
Ash, percent, maximum - - - -	0.10	4.4.6
pH of water extract - - - - -	6.0 ± 1.0	4.4.7
Apparent density, g/cc.-----	0.08 ± 0.03	4.4.8
Coarse particles:		
Retained on No. 325 sieve ¹ , percent, maximum -----	0.10	4.4.9
Grit, percent, maximum - - - -	0.05	4.4.10

¹The sieve shall conform to Specification RR-S-366.

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3.3 Workmanship. — The acetylene black shall be processed in accordance with high grade commercial practices for the manufacture of this type of material.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES.

4.1 Lot. - A lot shall consist of 1000 pounds maximum.

4.2 Sampling. — Ten percent of the containers comprised in a lot, but in no case more than 10 or less than 3 containers shall be selected by the inspector so as to be representative of the lot. If there are less than 4 containers in the lot, all the containers shall be sampled. The material in each selected container shall be mixed to a uniform consistency throughout, and approximately 8 ounces shall be removed from each container. Each of these primary samples shall be placed in an airtight container and labeled so that the container from which it was taken can be identified. A composite sample of approximately 8 ounces shall be made from equal portions of the primary samples. The composite sample shall be thoroughly mixed and placed in an airtight container labeled to show the name of the material, manufacturer, plans, contract or order number, lot number, and lot size. All acceptance tests shall be made on the composite sample. However, if it becomes apparent during sampling that the lot is not uniform, the inspector may require that any primary sample be tested for compliance with the requirements of this specification. All primary samples shall be held for possible future examination should the composite sample fail to meet the requirements.

4.3 Inspection

4.3.1 Army. — Inspection shall be made in accordance with Specification MILG-2550, and shall be at the point of delivery unless otherwise specified.

4.3.2 Navy. — The general inspection procedures shall be in accordance with General Specifications for Inspection of Material.

4.4 Tests.

4.4.1 Moisture. — Transfer an accurately weighed portion of approximately 2 grams of the sample to a tared, wide-mouth, short weighing bottle fitted with a glass stopper. Heat, with stopper removed, for 2 hours at $105 \pm 2^\circ$ C. Insert stopper, cool in a desiccator and weigh. Repeat the heating at $105 \pm 2^\circ$ C. until constant weight is attained. Calculate the loss in weight of the sample as percent moisture.

4.4.2 Sulfur. — Transfer an accurately weighed portion of approximately 0.5 gram of the sample to the cup of a Parr sulfur bomb. Add approximately 1 gram of potassium perchlorate, and mix thoroughly with a glass rod. Add approximately 15 grams of sodium peroxide, cover the cup, and mix by shaking. Assemble and tighten the bomb, and place it in the holder within a shield. Ignite the contents of the bomb either electrically or by directing the flame of an oxy-gas burner against the bottom of the cup. After the initial ignition continue the heating until the lower half of the cup attains an even cherry-red color. Remove the bomb from the shield, allow it to stand at room temperature for approximately 5 minutes, and then cool it in water. Remove the outer fastenings of the bomb without disturbing the cap, wash the exterior of the cap and the cup with water, and discard the washings. Remove the bomb cap, place the cup on its side in a 250-ml. beaker, and wash the material adhering to the underside of the bomb cap into the beaker with a stream of water from a wash bottle. Cover the beaker with a watch glass, and cautiously add 100 ml. of hot distilled water. When the material is dissolved remove the cup, rinsing any adhering fused material into the beaker. Make the solution neutral to litmus paper with approximately 12N hydrochloric acid, allowing sufficient time between additions of the acid for completion of the reaction. Add 1 ml. of acid in excess. Filter the solution, and wash the filter paper with hot distilled water. Make up the filtrate to 200 ml. with distilled water. Heat the solu-

tion to boiling, and slowly add 10 ml. of a 10-percent solution of barium chloride to the boiling solution. Continue boiling for 15 minutes, and then allow to stand for 2 hours at a temperature just below the boiling point. Filter the barium sulfate precipitate through a tared filtering crucible and wash thoroughly with hot distilled water. Dry the crucible and its contents in an oven, and then ignite at a temperature of $850 \pm 50^\circ\text{C}$. Cool in a desiccator and weigh. Calculate the increase in weight of the crucible to percent sulfur content of the sample, as follows:

$$\text{Percent sulfur} = \frac{13.74A}{W}$$

where: A = weight of barium sulfate precipitate, grams.

W = weight of sample, grams.

4.4.2.1 Alternative method. —The sulfur content may also be determined with a Parr-type oxygen bomb, using oxygen from an external source.

4.4.2.1.1 Apparatus.

4.4.2.1.1.1 Bomb. — Self-sealing oxygen combustion bomb equipped with a double valve (see 6.3),

4.4.2.1.1.2 Cup. —Stainless steel cup of a capacity of 6.5 ml.

4.4.2.1.1.3 Firing wire. —Iron-nickel-chromium alloy wire of 0.0063-inch nominal diameter.

4.4.2.1.2 Ignition —The firing wire shall be clean and so arranged that it will touch the sample when the cup is placed in position in the bomb. Place 5 ml. of distilled water in the bomb to saturate the oxygen used for combustion with water" vapor. Place an accurately weighed portion of approximately 0.4 gm. of the sample in the cup. Place the cup in the bomb, and close the bomb. Tighten the bomb cover firmly to avoid blowing the sample from the cup, and allow the oxygen to flow into the bomb. Cease the addition of oxygen after a pressure of 40 atmospheres is

reached. Make the electrical connections, place the bomb in a bucket of cold water, and ignite the sample. After 10 minutes remove the bomb from the water and release the pressure at a uniform rate, so that the operation requires at least 1 minute. Open the bomb, and examine the contents for traces of unburned sample. If any traces are found the determination shall be discarded and another test performed.

4.4.2.1.3 Dissolution, precipitation, and calcination. —The dissolution and precipitation of the material shall be carried out as specified in 4.4.2. The calculation of percent sulfur content shall be performed as specified in 4.4.2.

4.4.9 Ether-extractable matter. —Transfer an accurately weighed sample of approximately 4 grams of the lump-free material to an extraction thimble, and extract with ether using a tared extraction flask and a Soxhlet extractor, or equivalent. Use a hot-water bath or a steam plate as the source of heat, adjusting the applied heat so that ether drips from the reflux condenser at the rate of 2 to 3 drops per second. Continue the extraction for at least 8 hours. Remove the extraction flask and evaporate the ether by heating on a steam bath until approximately 26 ml. of the ether solution remains. Complete the evaporation to dryness by blowing a slow current of dry air over the solution. Dry the flask to constant weight in a vacuum desiccator containing sulfuric acid. Calculate the gain in weight of the flask to percent ether-extractable matter.

4.4.4 Volatile matter.

4.4.4.1 Apparatus.

4.4.4.1.1 Crucible. — A platinum crucible with a capacity of approximately 20 ml. (approximately 25 mm. in diameter and 35 mm. deep), and equipped with a closely fitting platinum cover.

4.4.4.1.2 Burner. —A Mekker-type burner having a top approximately 42mm. in diameter, and giving a flame approximately 16 cm. high (see 6.4). The temperature of the flame

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shall be such that when the crucible is placed 1 inch above the burner, a temperature of $950 \pm 20^\circ\text{C}$. shall be maintained inside the crucible. Check the temperature inside the crucible with a pyrometer before starting the determination, by inserting the thermocouple through a hole in an asbestos lid fitted to the platinum crucible and placing the junction of the thermocouple in contact with the bottom of the crucible (see 6.5).

4.4.4.2 Procedure. —Transfer an accurately weighed portion of approximately 1.00 gram of the sample to the tared platinum crucible. Cover the crucible, making certain that the lid fits tightly all around the edge, and place the crucible upright in a nichrome or vitreosil triangle adjusted so that the bottom of the crucible is approximately 1 inch above the top of the burner. Light the burner, and using the full flame allow the crucible to heat for $7 \pm \frac{1}{4}$ minutes. Extinguish the flame at the end of this period, and when the crucible and contents have cooled to below a red heat place them in a desiccator, and weigh when cooled. Calculate the percent volatile matter as follows:

$$\text{Percent volatile matter} = \frac{100 A}{W} - B$$

where: A = Less in weight of the sample, grams.

B = percent moisture in the sample (determined as specified in 4.4.1).

W = weight of sample, grams.

4.4.5 Water-soluble matter. —Transfer an accurately weighed portion of approximately 5 gm. of the sample to a beaker, and add 150 ml. of distilled water. Boil for 15 minutes, cool, and transfer to a 200-ml. volumetric flask. Dilute to the mark with distilled water, and mix. Allow the solution to settle, remove approximately two-thirds of the supernatant liquid, and filter through a dry filter paper. Discard the first 10 ml. of the filtrate. Transfer 100 ml. of the clear filtrate to a tared evaporating dish. Evaporate to dryness, cool in a desiccator, and weigh. Calculate the per-

centage of water-soluble matter in the sample, as follows:

$$\text{Percent water-soluble matter} = \frac{200 A}{W}$$

where: A = gain in weight of the dish, grams.

W = weight of sample, grams.

4.4.6 Ash. —Transfer an accurately weighed portion of 1 to 2 grams of the sample to a tared platinum or porcelain crucible, and ignite with a gas flame, or a muffle furnace. Continue heating until all combustible matter is consumed, cool in a desiccator, and weigh. Calculate the weight of the residue in the crucible to percent ash in the material.

4.4.7 pH value of water extract. —Transfer an accurately weighed portion of 2.0 grams of the sample to a flask fitted with a ground-glass stopper. Add 100 ml. of hot freshly boiled distilled water having a pH of 6.0 ± 0.5 , and stopper the flask. Shake the flask to obtain complete dispersion of the sample, and then allow it to stand for 2 hours with occasional shaking. Cool the mixture if necessary and determine the pH of the solution at $25^\circ \pm 2^\circ\text{C}$. by means of a pH meter which reads directly in pH units and is capable of measuring accurately to within 0.1 pH unit. The pH meter shall be equipped with a glass electrode and a saturated calomel electrode (see 6.6) and shall be calibrated with standard buffer solutions (see 6.7). In cases of dispute the standard buffer solutions prescribed by the U. S. Bureau of Standards shall be used to calibrate the pH meter.

4.4.8 Apparent density. —Assemble a Scott Volumeter as shown in figure 1, placing a No. 40 U. S. Standard sieve, conforming to Specification RR-S-366, on the top hopper. The tared receiving cube, having a volume of 1 cubic inch, shall be placed directly under the lower funnel, and resting on the base of the apparatus. Use a sufficient quantity of the sample to fill the receiving cube to overflowing. Pour the material slowly into the hopper, brushing it through the sieve if necessary.

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Carefully strike off the excess material collected in the cube using a spatula, taking care not to jar the cube during this operation. Weigh the filled cube and from the weight and volume of the material in the cube, calculate its apparent density, in grams per cubic centimeter. Make 3 such determinations, and report the average of the 3 values obtained as the apparent density of the material.

4.4.9 Coarse particles. —Prepare a stock solution of dispersing agent (see 6.8) in water, using as high a concentration of dispersing agent as possible without losing fluidity, and filter the solution through coarse filter paper. Place a 5-gram portion of the sample in a 600-ml. beaker. Add enough dispersing agent solution to form a heavy paste, and mix well to thoroughly incorporate the acetylene black. Dilute the mixture with water to a volume of approximately 300 ml. and then pour through a U. S. Standard No. 325 sieve. Brush the material through the sieve using a small camel's hair brush, while running water through the material. Once or twice during the screening remove the sieve from under the running water, add 1 or 2 drops of the stock dispersing agent solution, thoroughly spread it over the sieve with the brush, and continue the washing and brushing. Continue this operation until no more acetylene black passes through the sieve. Dry the sieve in an oven at 100 to 105° C. for 1 hour, cool, and weigh the residue. Calculate the weight of residue as percent coarse particles in the sample. Retain the residue for the determination of grit (see 4.4.10).

4.4.10 Grit. —Transfer the residue, if any, obtained in the determination of coarse particles (see 4.4.9) to a piece of smooth white paper. Gently rub the residue with the finger until the paper is no longer blackened by the remaining material. Transfer the remaining particles to a tared weighing dish, weigh, and calculate the weight of the particles to percent grit in the material.

4.5 Resubmission and retests. —If the composite sample, or any primary sample sub-

jected to test, fails to pass the specified tests, the lot shall be rejected. The contractor shall have the option of having a partial or complete analysis made on samples taken from all or any of the containers in the lot, at no expense to the Government. The contractor may then remove the defective portions of the lot and resubmit the lot for acceptance, provided complete replacement of the defective portions can be made to the satisfaction of the inspector. The resubmitted lot shall be accepted, provided that new samples, selected in accordance with 4.2, pass all the tests required by this specification.

5. PREPARATION FOR DELIVERY.

5.1 Packaging. —Unless otherwise specified, acetylene black shall be furnished in suitable moisture-proof, commercial containers.

5.2 Packing. —Unless otherwise specified, the containers of acetylene black shall be packed in commercial shipping containers so constructed as to insure acceptance by common or other carriers, for safe transposition, at the lowest rate of the carrier, to the point of delivery.

5.3 Marking. —In addition to any special markings required by the contract or order, shipments shall be marked in accordance with Standard MIL-STD-129.

6. NOTES

6.1 Intended use. —The acetylene black is intended for use in the manufacture of primer compositions.

6.2 Ordering data. — Procurement documents should specify the title, number, and date of this specification.

6.3 The Parr Instrument Co. No. 1101 Model BB oxygen combustion bomb has been found satisfactory for use in making the sulfur content determination by the alternative method specified in 4.4.2.1.

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6.4 The Fischer Scientific Co., No. 3-90, Mekker-type burner has been found satisfactory for use in making the volatile matter determination (see 4.4.4.1.2).

6.5 If a pyrometer is not available the temperature of the flame may be checked by noting if complete fusion of pure potassium chromate (melting point 9680 C.) takes place in the bottom of the crucible. In case of dispute a pyrometer should be used to check the temperature (see 4.4.4.1.2).

6.6 A Beckman pH meter industrial Model M, has been found satisfactory for the determination of the pH value (see 4.4.7).

6.7 Buffer solutions for calibrating the pH meter.

6.7.1 NBS standard solutions. — Prepare solutions of National Bureau of Standards' pH standards numbers 185, 1861, 18611, and 187 according to the directions given on their respective certificates.

6.7.2 Stock solutions. — Prepare a 0.05 molar in stock solutions for use in the preparation of buffer solutions (see 6.7.3).

6.7.2.1 Solution A.—Prepare a 0.1 molar solution of potassium dihydrogen phosphate by dissolving 13.61 grams of the material, ACS grade, in distilled water, and diluting to 1 liter.

6.7.2.2 Solution B.—Prepare a 0.05 molar solution of sodium borate (borax) by dissolving 19.10 grams of the material, ACS grade, in distilled water, and diluting to 1 liter.

6.7.2.3 Solution C. —Prepare a 0.05 molar succinic acid solution by dissolving 6.9 grams

of succinic acid (which has been purified by recrystallizing twice from distilled water and dried to constant weight in a desiccator over calcium chloride), in distilled water, and diluting to 1 liter.

6.7.3 Preparation of the buffer solutions. — Prepare buffer solutions of the indicated pH at 250 C. by mixing the stock solutions in the following proportions:

pH of buffer solution	Stock solution ml.		
	A	B	C
4.0	---	1.78	8.22
5.0	---	3.68	6.32
6.0	8.77	1.23	---
7.0	6.23	3.77	---
8.0	4.65	5.35	---
9.0	1.75	8.26	---

6.8 Triton 720, Darven No, 1, and Nacconol have been found satisfactory dispersing agents for making the coarse particles determination (see 4.4.9).

Notice.—When Government drawings, specification, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto

Custodians:

Army—Ordnance Corps
Navy—Bureau of Ordnance

Other interest:

Navy—S.

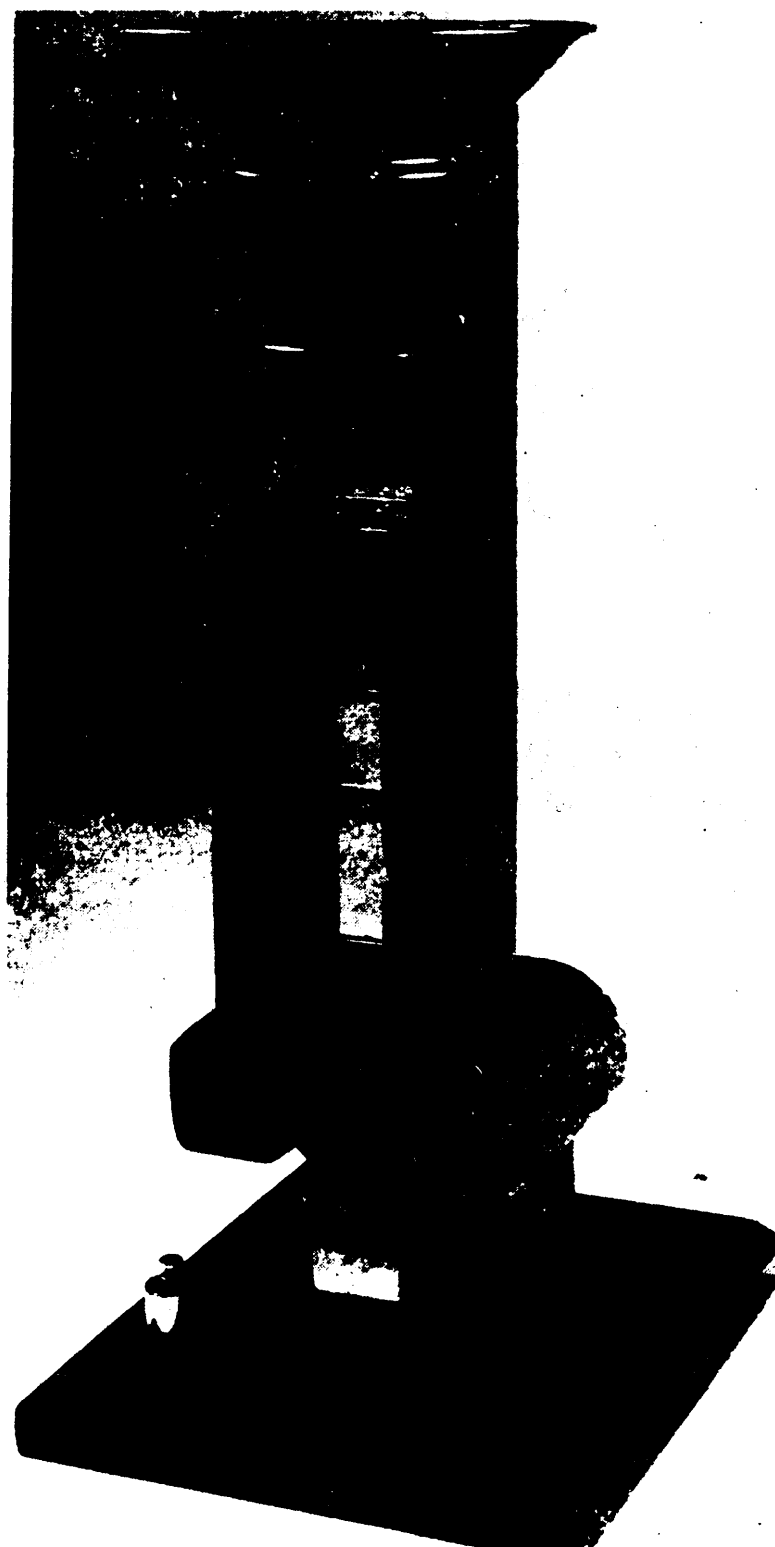


FIGURE 1. —*Apparatus for determination of apparent density.*

