

POWDER, FUMED SILICON DIOXIDE

1. SCOPE:

- 1.1 Form: This specification and its supplementary detail specifications cover silicon dioxide in the form of white, anhydrous powders.
- 1.2 Application: Primarily as a thickening (thixotropic) agent for resin systems.
- 1.3 Classification: Powders shall be classified by thickening efficiency as specified in the detail specifications.

2. APPLICABLE DOCUMENTS: The following publications form a part of this specification to the extent specified herein. The latest issue of Aerospace Material Specifications (AMS) shall apply. The applicable issue of other documents shall be as specified in AMS 2350.

- 2.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096.

2.1.1 Aerospace Material Specifications:

AMS 2350 - Standards and Test Methods
AMS 2825 - Material Safety Data Sheets

- 2.2 ASTM Publications: Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

ASTM C819 - Specific Surface Area of Carbon or Graphite
ASTM D1824 - Apparent Viscosity of Plastisols and Organosols at Low Shear Rates by Brookfield Viscometer
ASTM D2773 - Loss on Ignition of Electrical Grade Magnesium Oxide
ASTM E300 - Sampling Industrial Chemicals

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AMS 3755

2.3 U.S. Government Publications: Available from Commanding Officer, Naval Publications and Forms Center, 5801 Tabor Avenue, Philadelphia, PA 19120.

2.3.1 Military Specifications:

MIL-R-7575 - Resin, Polyester, Low Pressure Laminating

2.3.2 Military Standards:

MIL-STD-794 - Parts and Equipment, Procedures for Packaging and Packing of

3. TECHNICAL REQUIREMENTS:

3.1 Detail Specifications: The requirements for a specific material shall consist of all the requirements specified herein in addition to the requirements specified in the applicable detail specification. In case of conflict between the requirements of this basic specification and an applicable detail specification, the requirements of the detail specification shall govern.

3.2 Material: Shall be a fumed, white, anhydrous silica.

3.3 Properties: Powder shall conform to the properties specified in the applicable detail specification; tests shall be performed on the powder supplied and in accordance with test methods specified in 4.5.

3.4 Quality: The powder, as received by purchaser, shall be uniform in quality and condition, clean, smooth, and free from foreign materials and from imperfections detrimental to usage of the powder.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection: The vendor of powder shall supply all samples for vendor's tests and shall be responsible for performing all required tests. Results of such tests shall be reported to the purchaser as required by 4.6. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the powder conforms to the requirements of this specification.

4.2 Classification of Tests:

4.2.1 Acceptance Tests: Tests to determine conformance to requirements for grit content and loss on ignition as specified in the applicable detail specification, and for quality (3.4) are classified as acceptance tests and shall be performed on each lot.

4.2.2 Preproduction Tests: Tests to determine conformance to all technical requirements of this specification and the applicable detail specification are classified as preproduction tests and shall be performed prior to or on the initial shipment of powder to a purchaser, when a change in material or processing, or both, requires reapproval as in 4.4.2, and when purchaser deems confirmatory testing to be required.

4.2.2.1 For direct U.S. Military procurement, substantiating test data and, when requested, preproduction test material shall be submitted to the cognizant agency as directed by the procuring activity, the contracting officer, or the request for procurement.

4.3 Sampling: Shall be as follows:

4.3.1 For Acceptance Tests: Each lot of powder shall be sampled in accordance with ASTM E300 at random to provide sufficient material to perform all required tests. The number of determinations for each requirement shall be as specified in the applicable test procedure or, if not specified therein, not less than three.

4.3.1.1 A lot shall be all powder produced in a continuous production run from the same batch of raw material under the same fixed conditions and presented for vendor's inspection at one time. An inspection lot shall not exceed 800 lb (365 kg) of powder and may be packaged in small quantities under the basic lot approval provided the lot identification is maintained.

4.3.1.2 When a statistical sampling plan and acceptance quality level (AQL) have been agreed upon by purchaser and vendor, sampling shall be in accordance with such plan in lieu of sampling as in 4.3.1 and the report of 4.6.1 shall state that such plan was used.

4.3.2 For Preproduction Tests: As agreed upon by purchaser and vendor.

4.4 Approval:

4.4.1 Sample powder shall be approved by purchaser before powder for production use is supplied, unless such approval be waived by purchaser. Results of tests on production powder shall be essentially equivalent to those on the approved sample.

4.4.2 Vendor shall use ingredients, manufacturing procedures, processes, and methods of inspection on production powder which are essentially the same as those used on the approved sample powder. If necessary to make any change in ingredients, in type of equipment for processing, or in manufacturing procedures, vendor shall submit for reapproval a statement of the proposed changes in material or processing, or both, and, when requested, sample powder. Production powder made by the revised procedures shall not be shipped prior to receipt of reapproval.

4.5 Test Methods: Shall be as follows:

Surface Area	ASTM C819
Grit Content	4.5.1
pH, 4% Aqueous Dispersion	4.5.2
Loss on Ignition	ASTM D2773
Thickening Efficiency	4.5.3

4.5.1 Grit Content:

- 4.5.1.1 Manually disperse a 10.0-g $\pm$ 0.1 sample of powder into 900 mL of filtered or screened water in a 1-L beaker.
- 4.5.1.2 Pour the dispersion into the funnel of the grit determination apparatus shown in Fig. 1 with the spray water on at 16.0 psi $\pm$ 0.2 (110.0 kPa $\pm$ 1.5) gage pressure. Continue the spray for 45 sec $\pm$ 5 after the funnel is empty, washing down the sides of the funnel with the hose.
- 4.5.1.3 After washing is completed, remove the screen containing the residue from the funnel, dry for not less than 20 min. at 105°C $\pm$ 5 (220°F $\pm$ 9) and weigh.
- 4.5.1.4 Remove the material or grit from the screen and reweigh the screen. The difference in weight indicates the amount of grit.
- 4.5.1.5 Calculate as follows:

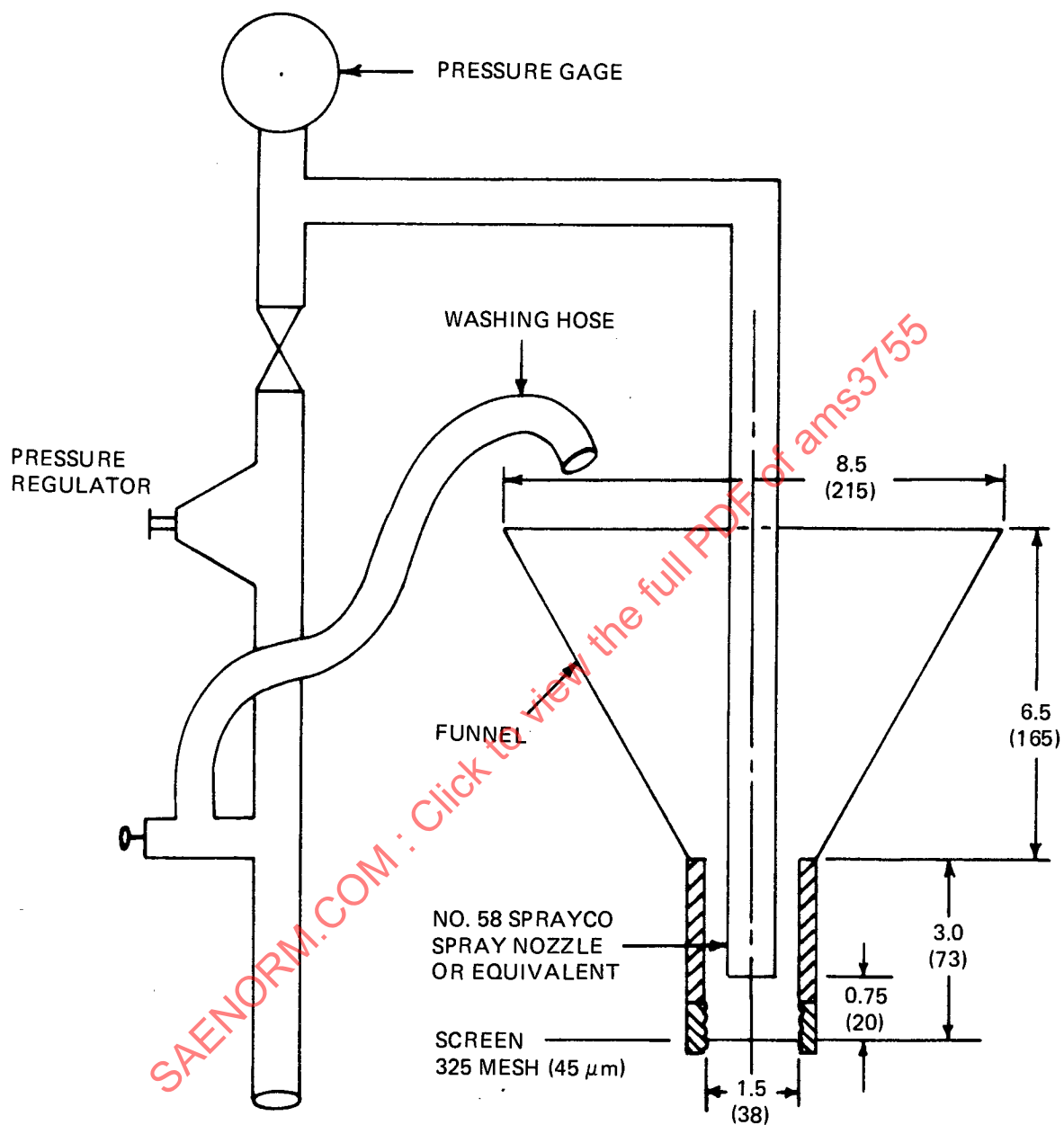
$$\% \text{ of grit} = \frac{\text{weight of grit}}{\text{weight of sample}} \times 100$$

4.5.2 pH, 4% Aqueous Dispersion:

- 4.5.2.1 Weigh 4.000 g $\pm$ 0.010 of powder into a 250-mL beaker and add 100 mL $\pm$ 10 of distilled water with pH of not lower than 5.5. Boiling may be necessary to remove carbon dioxide to give a minimum pH of 5.5. (Do not adjust the pH of the water with caustic.)
- 4.5.2.2 Stir the mixture vigorously for not less than 5 minutes. When liquid agitation ceases, measure pH.

4.5.3 Thickening Efficiency:

- 4.5.3.1 Weigh 394.0 g $\pm$ 0.5 of standardized polyester resin (See 4.5.3.6) into a 1-qt or 1-L wide mouth container. Weigh 6.00 g $\pm$ 0.01 of fumed silicon dioxide powder, sample or standard, onto the resin.
- 4.5.3.2 Carefully incorporate all of the powder into the resin.



DIMENSIONS ARE IN INCHES (MILLIMETRES), APPROXIMATE

FIGURE 1. GRIT DETERMINATION APPARATUS

4.5.3.3 Place the container into a nitrogen atmosphere and start the nitrogen flow. While under nitrogen, agitate the mixture with a dispersion head (See 8.1) for 5.0 min. ± 0.1 at 3000 rpm ± 100 . Deaerate the sample at 9 in. ± 1 (230 mm ± 25) of mercury for not less than 5 minutes.

4.5.3.4 Measure the viscosity after not less than 15 min. rotation at 3.0 rpm ± 0.5 at 28°C ± 0.2 (82°F ± 0.5) in accordance with ASTM D1824 using a Brookfield LVT viscometer with No. 3 spindle.

4.5.3.5 Compute thickening efficiency as follows:

$$\text{Thickening Efficiency, \%} = \frac{\text{Sample viscosity, CPS, 6 rpm}}{\text{Standard viscosity, CPS, 6 rpm}} \times 100$$

4.5.3.6 Resin Standardization:

4.5.3.6.1 Determine the solids content of a MIL-R-7575 (See 8.2) polyester resin as follows:

4.5.3.6.1.1 Into a suitable container, preweighed to the nearest 0.1 mg, weigh in 1 - 2 g polyester.

4.5.3.6.1.2 Weigh container and contents to nearest 0.1 milligram.

4.5.3.6.1.3 Add 10 mL reagent grade acetone and mix well.

4.5.3.6.1.4 Dry container and contents at 110°C ± 5 (230°F ± 9) for 2.0 hr ± 0.1 .

4.5.3.6.1.5 Cool container and contents in desiccator and reweigh. Calculation:

$$\% \text{ Solids} = \frac{100 \times (\text{weight of dried polyester})}{\text{weight of undried polyester}}$$

4.5.3.6.2 Determine the amount of styrene to be added to the polyester resin by the following equation:

$$\text{Weight styrene (to be added)} = \frac{\text{Wt of Resin} \times (100 - \text{initial styrene content})}{100 - \text{desired styrene level}} - \text{wt. Resin}$$

where: initial styrene content = 100 - % solids
desired styrene level = 40, 36, 44%

4.5.3.6.2.1 Add styrene to bring styrene content in the resin to 40%, 36%, and 44% in two samples representing each level.

4.5.3.6.3 Standardization of Resin Viscosity:

- 4.5.3.6.3.1 Determine the viscosity resulting from incorporation of a known amount of standard (100%) silica (See 8.3) into duplicate samples of the resin at each of the three styrene levels, using the methods shown in 4.5.3.1 through 4.5.3.4.
- 4.5.3.6.3.2 Determine the optimum level of styrene by a graphical means as shown in Fig. 2.

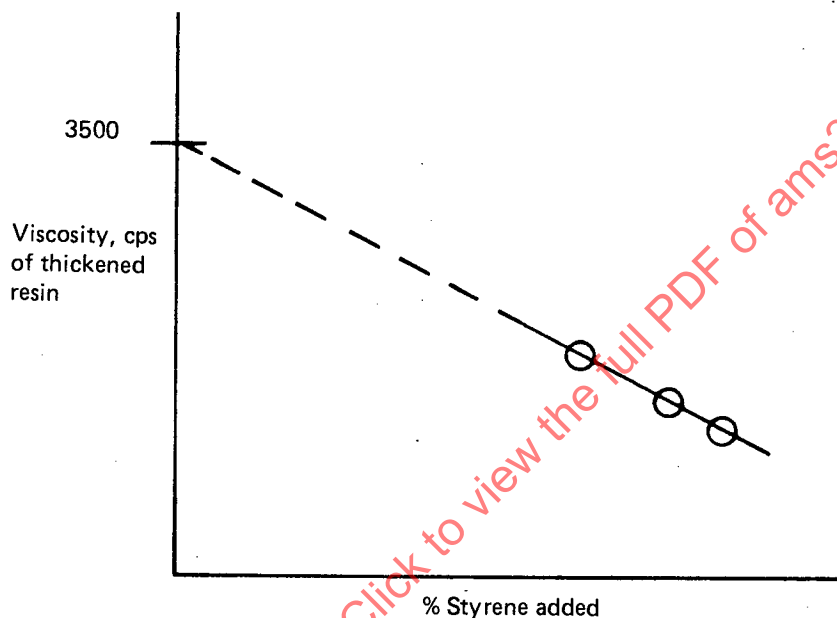


FIGURE 2

- 4.5.3.6.3.3 Adjust the resin with styrene, using the resin raw data and the graphical solution, to produce a thickened viscosity of $3500 \text{ cps} \pm 200$.
- 4.6 Reports: The vendor of powder shall furnish with each shipment three copies of a report showing the results of tests to determine conformance to the acceptance test requirements and stating that the powder conforms to the other technical requirements of this specification. This report shall include the purchase order number, AMS 3755 and the applicable detail specification number, vendor's product designation, lot number, date of manufacture, and quantity.