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Superseding AMS3753A

**Microspheres, Carbon
Hollow**

RATIONALE

This document has been revised to remove specific military requirements described in former sections 4.2.2.1, 5.1.5, and 8.4. MIL-STD-2073-1 was also deleted from section 2.3 since it was referenced in section 5.1.5.

1. SCOPE

1.1 Form

This specification covers carbon in the form of hollow microspheres.

1.2 Application

This product has been used typically as a filler material in syntactic foam shapes, but usage is not limited to such applications.

1.3 Safety - Hazardous Materials

While the materials, methods, applications, and processes described or referenced in this specification may involve the use of hazardous materials, this specification does not address the hazards which may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

2. APPLICABLE DOCUMENTS

The issue of the following documents in effect on the date of the purchase order forms a part of this specification to the extent specified herein. The supplier may work to a subsequent revision of a document unless a specific document issue is specified. When the referenced document has been cancelled and no superseding document has been specified, the last published issue of that document shall apply.

2.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or 724-776-4970 (outside USA), www.sae.org.

AMS2825 Material Safety Data Sheets

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2.2 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org.

ASTM D 280 Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments
ASTM D 1895 Apparent Density, Bulk Factor, and Pourability of Plastic Materials
ASTM D 2841 Sampling Hollow Microspheres

3. TECHNICAL REQUIREMENTS

3.1 Material

Thin walled carbon microspheres filled with an inert gas.

3.2 Finish

Microspheres shall not be finished or coated.

3.3 Properties

The product shall conform to the requirements shown in Table 1; tests shall be performed on the product supplied and in accordance with specified test methods.

TABLE 1 - PROPERTIES

Paragraph	Property	Requirement	Test Method
3.3.1	Bulk Density	0.012 to 0.020 g/cm ³	ASTM D 1895
3.3.2	Size Distribution, Sieve Analysis		4.5.1
3.3.2.1	Weight retained on No. 50 mesh (300 µm) screen, maximum	2%	
3.3.2.2	Weight passing through No. 325 mesh (45 µm) screen, maximum	25%	
3.3.3	Volatile Content, maximum	2%	4.5.2
3.3.4	Ash Content, maximum	4%	4.5.3

3.4 Quality

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

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection

The vendor of microspheres shall supply all samples for vendor's tests and shall be responsible for the performance of all required tests. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the microspheres conform to specified requirements.

4.2 Classification of Tests

4.2.1 Acceptance Tests

Bulk density (3.3.1), size distribution (3.3.2), and quality (3.4) are acceptance tests and shall be performed on each lot.

4.2.2 Preproduction Tests

Tests for all technical requirements are preproduction tests and shall be performed prior to or on the initial shipment of microspheres to a purchaser, when a change in ingredients and/or processing requires reapproval as in 4.4.2, and when purchaser deems confirmatory testing to be required.

4.3 Sampling and Testing

4.3.1 For Acceptance Tests

Each lot of microspheres shall be sampled in accordance with ASTM D 2841, using spike sampler "B" modified for small containers, at random to provide sufficient product 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 microspheres produced in a continuous production run from the same batch of raw materials under the same fixed conditions and presented for vendor's inspection at one time. An inspection lot shall not exceed 800 pounds (363 kg) of microspheres.

4.3.1.2 When a statistical sampling plan has 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 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 microspheres shall be approved by purchaser before microspheres for production use are supplied, unless such approval be waived by purchaser. Results of tests on production microspheres 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 microspheres which are essentially the same as those used on the approved sample microspheres. 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 ingredients and/or processing and, when requested, sample microspheres. Production microspheres made by the revised procedure shall not be shipped prior to receipt of reapproval.

4.5 Test Methods

4.5.1 Size Distribution, (Sieve Analysis)

4.5.1.1 Place 50 mesh (300 μm) sieve on top of a 325 mesh (45 μm) sieve and pan. Weigh a 25 gram $\pm$ 0.5 sample of microspheres and pour into the 50 mesh (300 μm) sieve. Cover.

4.5.1.2 Install sieve assembly on a Thomas shaker, operating at 190 RPM, for 30 minutes $\pm$ 1. Weigh microspheres remaining on the 50 mesh (300 μm) sieve. Weigh microspheres on pan.

4.5.1.3 Calculate the percentage retained and passing the sieves as shown in Equation 1 and Equation 2.

Amount retained on No. 50 mesh (300 μm) screen, %, maximum =

$$\frac{\text{amount of sample on No. 50 mesh (300 } \mu\text{m) screen}}{\text{original sample amount}} \times 100 \quad (\text{Eq. 1})$$

Amount passing through No. 325 mesh (45 µm) screen, %, maximum =

$$\frac{\text{amount of sample on pan}}{\text{original sample amount}} \times 100 \quad (\text{Eq. 2})$$

4.5.2 Volatile Content

Shall be determined in accordance with ASTM D 280. Sample size shall be the capacity of the weighing bottle. Report the average of three determinations.

4.5.3 Ash Content

- 4.5.3.1 Use only microspheres which have been previously tested for volatile content and have been stored in a desiccator for this test.
- 4.5.3.2 Dry five shallow form, 50 mL capacity, 80 mm rim height mullite combustion dishes in a muffle furnace at $700\text{ }^{\circ}\text{C} \pm 5$ ($1292\text{ }^{\circ}\text{F} \pm 9$) for not less than 30 minutes.
- 4.5.3.3 Remove dishes from furnace, cool to below $300\text{ }^{\circ}\text{C}$ ($572\text{ }^{\circ}\text{F}$), and place in desiccator to cool to room temperature.
- 4.5.3.4 Weigh the dishes at room temperature to the nearest 0.0001 gram and record weight, (W_1). Spoon in approximately 2 grams of microspheres to the dishes. Reweigh all dishes, (W_3).
- 4.5.3.5 Return dishes to $700\text{ }^{\circ}\text{C} \pm 5$ ($1292\text{ }^{\circ}\text{F} \pm 9$) furnace for not less than two hours.
- 4.5.3.6 Remove the dishes from the furnace. If there is no black residue, proceed. If there is a black residue, return the dishes to the furnace for not less than 0.5 hour or until all black residue is gone. Allow the dishes to cool to below $300\text{ }^{\circ}\text{C}$ ($572\text{ }^{\circ}\text{F}$) and place in a desiccator to cool to room temperature.
- 4.5.3.7 Weigh the filled dishes to the nearest 0.0001 gram, (W_2).
- 4.5.3.8 Calculate the % ash as shown in Equation 3.

$$\% \text{ Ash} = \frac{W_2 - W_1}{W_3 - W_1} \times 100 \quad (\text{Eq. 3})$$

Where:

- W_1 = weight of empty dish
- W_2 = weight of dish and contents after heating
- W_3 = weight of dish and contents before heating

4.6 Reports

The vendor of microspheres shall furnish with each shipment a report showing the results of tests to determine conformance to the acceptance test requirements and stating that the microspheres conform to the other technical requirements. This report shall include the purchase order number, lot number, AMS3753B, vendor's product designation, date of manufacture, and quantity.

- 4.6.1 A material safety data sheet conforming to AMS2825, or equivalent, shall be supplied to each purchaser prior to, or concurrent with, the report of preproduction test results or, if preproduction testing be waived by purchaser, concurrent with the first shipment of microspheres for production use. Each request for modification of formulation shall be accompanied by a revised data sheet for the proposed formulation.