

MICROSPHERES, CARBON
Hollow

1. SCOPE:

- 1.1 Form: This specification covers hollow carbon microspheres.
- 1.2 Application: Primarily as a filler material in syntactic foam shapes.

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 D280 - Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments
ASTM D2841 - Sampling Hollow Microspheres
ASTM D3101 - Determination of Bulk Density and Packing Factor of Hollow Glass Microspheres

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

REAFFIRMED

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2.3.1 Military Standards:

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

3. TECHNICAL REQUIREMENTS:

3.1 Material: The hollow microspheres shall be composed of thin walled carbon microballoons, filled with an inert gas.

3.2 Finish: Unless otherwise specified, microspheres shall not be finished or coated.

3.3 Properties: The product shall conform to the following requirements; tests shall be performed on the product supplied and in accordance with test methods specified in 4.5:

3.3.1 Bulk Density 0.012 - 0.020 g/cm³

3.3.2 Size Distribution, Sieve Analysis

3.3.2.1 Weight retained on No. 50 mesh 2%
(300 μ m) screen, max

3.3.2.2 Weight passing through No. 325 25%
mesh (45 μ m) screen, max

3.3.3 Volatile Content, max 2%

3.3.4 Ash Content, max 4%

3.4 Quality: The microspheres, 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 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 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 microspheres conform to the requirements of this specification.

4.2 Classification of Tests:

4.2.1 Acceptance Tests: Tests to determine conformance to requirements for bulk density (3.3.1), size distribution (3.3.2), and 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 are classified as preproduction tests and shall be performed prior to or on the initial shipment of microspheres 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 microspheres shall be sampled in accordance with ASTM D2841, using spike sampler "B" modified for small containers, 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 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 lb (365 kg) of microspheres 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 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 material or processing, or both, 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: Shall be as follows:

Bulk Density	ASTM D3101
Size Distribution (Sieve Analysis)	4.5.1
Volatiles	4.5.2
Ash Content	4.5.3

4.5.1 Size Distribution, (Sieve Analysis):

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

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

4.5.1.3 Calculate the percentage retained on each sieve as follows:

4.5.1.3.1 Weight retained on NO. 50 mesh = $\frac{\text{weight of sample on 50 mesh}}{\text{original sample weight}} \times 100$
(300 μ m) screen, % max

4.3.1.3.2 Weight passing through No. 325 mesh = $\frac{\text{weight of sample on pan}}{\text{original sample weight}} \times 100$
(45 μ m) screen, % max

4.5.2 Volatile Content: Shall be determined in accordance with ASTM D280. 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°C $\pm$ 5 (1290°F $\pm$ 9) for not less than 30 minutes.

4.5.3.3 Remove dishes from furnace, cool to below 300°C (570°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 g and record weight, (W₁). Spoon in approximately 2 g of microspheres to the dishes. Reweigh all dishes, (W₂).

4.5.3.5 Return dishes to 700°C $\pm$ 5 (1290°F $\pm$ 9) furnace for not less than 2 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 hr or until all black residue is gone. Allow the dishes to cool to below 300°C (570°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 follows:

$$\% \text{ Ash} = \frac{W_2 - W_1}{W_3 - W_1} \times 100$$

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 three copies of 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 of this specification. This report shall include the purchase order number, AMS 3753, vendor's product designation, lot number, date of manufacture, and quantity.

4.6.1 A material safety data sheet conforming to AMS 2825 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.

4.7 Resampling and Retesting: If any sample used in the above tests fails to meet the specified requirements, disposition of the microspheres may be based on the results of testing three additional samples for each original nonconforming sample. Failure of any retest sample to meet the specified requirements shall be cause for rejection of the microspheres represented and no additional testing shall be permitted. Results of all tests shall be reported.

5. PREPARATION FOR DELIVERY:

5.1 Packaging and Identification:

5.1.1 The microspheres shall be packed in sealed, moisture-resistant plastic bags.