

# AEROSPACE MATERIAL SPECIFICATION

Submitted for recognition as an American National Standard



**AMS 3753A**

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Superseding AMS 3753

## Microspheres, Carbon Hollow

### 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 following publications form a part of this specification to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order.

#### 2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 2825 Material Safety Data Sheets

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## 2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

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

## 2.3 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

MIL-STD-2073-1   DOD Materiel, Procedures for Development and Application of Packaging Requirements

## 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 <sup>3</sup> | 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 performing all required tests. 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 for 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.

4.2.2.1 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, contracting officer, or request for procurement.

#### 4.3 Sampling and Testing:

Shall be as follows:

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:

Shall be as follows:

##### 4.5.1 Size Distribution, (Sieve Analysis):

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

$$\begin{aligned} \text{Amount retained on No. 50 mesh (300 } \mu\text{m) screen, \% , maximum} &= & (\text{Eq. 1}) \\ \frac{\text{amount of sample on No. 50 mesh (300 } \mu\text{m)screen}}{\text{original sample amount}} \times 100 \end{aligned}$$

$$\begin{aligned} \text{Amount passing through No. 325 mesh (45 } \mu\text{m) screen, \% , maximum} &= & (\text{Eq. 2}) \\ \frac{\text{amount of sample on pan}}{\text{original sample amount}} \times 100 \end{aligned}$$

- 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, AMS 3753A, vendor's product designation, 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. 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.

5.1.1.1 Microspheres may be packaged in small quantities and delivered under the basic lot approval provided lot identification is maintained.

5.1.2 Individual bags of microspheres shall be packed in an exterior container capable of protecting the microspheres, during shipment and storage, against damage from exposure to weather or any other normal hazard.