

MIL-S-24167A(SHIPS)  
6 December 1972  
SUPERSEDING  
MIL-S-24167(SHIPS)  
31 August 1965  
(See 6.7)

MILITARY SPECIFICATION

SYNTACTIC MATERIAL, RIGID, POUR-IN-PLACE, STRUCTURAL VOID FILLING

1. SCOPE

1.1 This specification covers the requirements for a pour-in-place, rigid cellular resin system.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of the specification to the extent specified herein.

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.  
MIL-STD-120 - Marking for Shipment and Storage.

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

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

UNIFORM CLASSIFICATION COMMITTEE

Uniform Freight Classification Rules.

(Application for copies should be addressed to the Uniform Classification Committee, Room 1106, 222 South Riverside Plaza, Chicago, Illinois 60606.)

NATIONAL MOTOR FREIGHT TRAFFIC ASSOCIATION, INC., AGENT

National Motor Freight Classification.

(Application for copies should be addressed to the American Trucking Association Inc., Traffic Order Section, 1616 P Street, N.W., Washington, D.C. 20036.)

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

D1621-64 - Test for Compressive Strength of Rigid Cellular Plastics.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa. 19103.)

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

3. REQUIREMENTS

3.1 Sample for first article inspection. Prior to beginning production a sample shall be tested as specified in 4.2 (see 6.5).

3.2 Material. The syntactic material shall consist of a polyester resin filled with low density hollow phenolic microspheres or hollow glass microspheres and hardened with a curing agent. When mixed in accordance with the instructions supplied by the contractor, the ingredients shall form a pourable material which after curing shall be a rigid cellular system suitable for use in buoyancy and structural void filling applications. The material shall air-cure at ambient temperatures and attain the physical properties described herein within 7 days after mixing.

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**3.3 Homogeneity.**

3.3.1 The freshly mixed material shall be homogeneous and free from lumps and coarse particles.

3.3.2 Single pours. The cured material shall be homogeneous throughout with uniform cells. There shall be no void openings, cracks, or any other occlusions, when measured as specified in 4.7.2.1.

3.3.3 Multiple pours (first article only). Each cured individual pour shall have the uniformity specified in 3.3.2. The fusion line between successive pours shall be well knit, and shall not show separation, cracks, or any significant amount of non-uniformity, when tested as specified in 4.7.1.2.

3.3.4 Density variation. The overall density shall not vary from the core density by more than 5 percent.

3.4 Density. The density of the cured syntactic material shall be as low as possible consistent with the strength requirements specified herein. The maximum allowable density shall be 44 pounds per cubic foot.

3.5 Color. The color shall be the natural color of the product.

3.6 Odor. The cured syntactic material shall be free from any objectionable odor.

3.7 Application properties. Application properties shall conform to the requirements of table I when tested in accordance with the applicable procedure of 4.7.3.

Table I - Application properties.

Property	Test paragraph	Requirements	Limits
Initial viscosity	4.7.3.1	20,000 cps	Maximum
Application life	4.7.3.2	1 hour	Minimum
Cure time	4.7.3.3	7 days	Maximum
Peak exotherm	4.7.3.4	300°F.	Maximum
Shrinkage	4.7.3.5	1.0 percent	Maximum

3.7.1 Initial viscosity. The freshly mixed syntactic material shall have a viscosity not exceeding 20,000 cycles per second (cps), when determined as specified in 4.7.3.1.

3.7.2 Application life. The application life of the freshly mixed syntactic material shall be not less than 1 hour when tested as specified in 4.7.3.2. The viscosity at the end of the 1 hour period from the time of addition of catalyst shall be not greater than 30,000 cps, when tested as specified in 4.7.3.2.

3.7.3 Cure time. The cure time for the syntactic material to attain full cure shall be not more than 7 days, when tested as specified in 4.7.3.3.

3.7.4 Peak exotherm. The peak exotherm during curing of the syntactic material shall be not greater than 300°F. when tested as specified in 4.7.3.4.

3.7.5 Shrinkage. The shrinkage of the syntactic material during curing shall not exceed 1.0 percent when measured as specified in 4.7.3.5.

3.8 Mechanical and physical properties. The mechanical and physical properties of the fully cured syntactic material shall be in accordance with table II when tested as specified in 4.7.4.

3.9 Supplier's instruction sheet. Suppliers of syntactic material ingredients under this specification shall provide the following information to procuring activities:

- (a) Supplier's code numbers and commercial designation.
- (b) Recommended storage procedure.
- (c) Usable storage life.
- (d) Handling precautions.

In addition to the above information, the polyester resin supplier shall provide a recommended syntactic formulation together with mixing instructions that will meet the physical property requirements of this specification.

Table II - Mechanical and physical properties.

Property	Test paragraph	Requirement	Limits	Specimens	
				Number	Sizes (inches) <sup>1/</sup>
Compressive yield strength:					
0.2 percent offset, minimum					
Dry	4.7.4.1.1	2,000 p.s.i.	Minimum	3	4 by 4 by 4
Wet	4.7.4.1.2	1,700 p.s.i.	Minimum	3	4 by 4 by 4
After humidity aging					
0.2 percent offset	4.7.4.1.3	1,700 p.s.i.	Minimum	3	4 by 4 by 4
Modulus of elasticity in compression (wet)	4.7.4.2	100,000 p.s.i.	Minimum	3	4 by 4 by 4
Compression set:					
After static loading	4.7.4.3	0.001 in./in.	Maximum	3	4 by 4 by 4
Impact resistance	4.7.4.4	8.5 ft. lbs.	Minimum	3	4 by 4 by 1
Water absorption:	4.7.4.5				
After 24-hour immersion		1.0 percent	Maximum	3	4 by 4 by 4
After 7 days immersion		1.5 percent	Maximum	3	4 by 4 by 4
After 30 days immersion		2.0 percent	Maximum	3	4 by 4 by 4

<sup>1/</sup> The allowable tolerance on the size of the test specimens shall be -1/16 inch on a side.

#### 4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 First article inspection. First article inspection shall consist of the examination and tests specified in 4.7.

#### 4.3 Sampling.

4.3.1 Inspection lot. For purposes of sampling, a lot of syntactic material shall be that quantity which can be compounded by mixing (a) hollow phenolic microspheres or hollow glass microspheres which have been produced in one batch with (b) polyester resin which has been produced in a single batch, (c) curing agent which has been produced in a single batch and (d) silicone surfactant which has also been produced in a single batch. The suppliers of each of these materials shall have applied or affixed identification on each container so that the foregoing lot requirement can be met.

4.3.2 Sampling for quality conformance tests. Samples for quality conformance tests specified in 4.5 shall be selected in accordance with table III.

Table III - Sampling for quality conformance tests.

Lot size, pounds	Sample size <sup>1/</sup> test blocks
Up to 400	2
401 to 800	3
801 to 1,600	4
1,601 to 3,200	5
For each additional 800 pounds	Add 1

<sup>1/</sup> The materials selected and mixed to make each of these test blocks shall have been taken from a different container.

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4.4 Examination of filled containers. A random sample of filled containers containing syntactic foam ingredients shall be taken from each lot in accordance with MIL-STD-105 at inspection level I and acceptable quality level = 2.5 percent defective, to verify compliance with this specification in regard to fill, closure, marking, and other requirements not involving tests. Containers shall be examined for defects of construction of the container and the closure, for evidence of leakage, and for unsatisfactory markings. Each sample filled container shall be weighed to determine the amount of contents. Any containers in the sample having one or more defects, or under required fill, shall not be offered for delivery. If the number of defective containers in any sample exceeds the acceptance number of the appropriate sampling plan of MIL-STD-105, this shall be cause for rejection of the lot represented by the sample.

4.5 Quality conformance tests. Quality conformance tests shall consist of the following tests:

- (a) Examination of product for homogeneity (single pour only) (see 4.7.1).
- (b) Density variation (see 4.7.2.1).
- (c) Density (see 4.7.2.2).
- (d) Application life (see 4.7.3.2).
- (e) Cure time (see 4.7.3.3).
- (f) Compressive yield strength 0.2 percent offset dry and wet (see 4.7.4.1).
- (g) Modulus of elasticity in compression (see 4.7.4.2).
- (h) Compression set (see 4.7.4.3).
- (i) Water absorption (24-hour immersion only) (see 4.7.4.5).
- (j) Impact resistance (see 4.7.4.4).

The test blocks, selected as specified in 4.3.2, shall be subjected separately to the above tests as specified under 4.7. If any test block fails one or more of these tests, the lot shall not be offered for delivery.

4.5.1 In addition, the product shall be subject to any of the other tests specified herein which the procuring activity considers necessary to determine conformance with the requirements of this specification.

#### 4.6 Test conditions.

4.6.1 Atmospheric test conditions. Unless otherwise specified (see 6.2), the atmospheric conditions surrounding the specimen prior to and during test shall be  $73.5^{\circ} \pm 1.8^{\circ}\text{F}$ . ( $23^{\circ} \pm 1^{\circ}\text{C}$ .) and  $50 \pm 2$  percent relative humidity. The conditioning period prior to test shall be a minimum of 24 hours. The temperature, the relative humidity, and the period of time for conditioning shall be recorded for each test.

#### 4.6.2 Test block preparation.

##### 4.6.2.1 Single pour test blocks.

4.6.2.1.1 Mold. The mold shall be 1/4-inch thick steel, Teflon coated with inside dimensions of 13 inches by 13 inches by 13 inches. The mold shall consist of four solid sides and a solid bottom with the top left open to allow pouring of the syntactic material into the mold. The sides and bottom shall be so constructed that the mold can be disassembled to allow removal of the specimen from the mold.

4.6.2.1.2 Molding of the test block. The base ingredients shall be mixed in the proportions specified in the polyester resin supplier's instruction sheet. A total amount of base material which will fill the mold specified in 4.6.1 shall be mixed in accordance with the supplier's instructions and poured into the mold. The amount of material mixed shall include a 10 percent excess to cover material remaining on the mixer blades and in the mixing vessel. The syntactic material shall be allowed to cure at room temperature in the mold for 48 hours before testing. The mixing and curing characteristics of the syntactic material shall be determined during the preparation of the test block.

4.6.2.1.3 Test specimen preparation. The test block of syntactic material shall be examined, weighed and measured. The block shall then be cut into test specimens in accordance with 4.7. The test specimens shall be machined to assure accuracy of dimensions.

4.6.2.2 Preparation of the multiple pour specimen. Multiple pour is applicable for first article inspection only.

4.6.2.2.1 Mold. The mold for the multiple pour specimen shall be an open head 55-gallon container having the approximate dimensions of 1-7/8 feet in diameter by 2-3/4 feet in height.

4.6.2.2.2 Molding of the multiple pour specimen. Three separate pourings of syntactic material shall be made to fill the mold specified in 4.6.2.2.1. The syntactic material for each pouring shall be mixed in accordance with polyester resin supplier's instructions.

4.6.2.2.3 Preparation of the multiple pour specimen. The multiple pour specimen prepared in accordance with 4.6.2.2.2 shall be vertically cut in half. If the specimen cannot be removed from the mold, it shall be permissible to cut through the mold during the cutting operation. The multiple pour specimen shall be examined as specified in 4.7.1.2.

4.6.3 Health precautions. During the molding of the single and multiple pour specimens adequate ventilation and safety precautions shall be provided as specified in the polyester resin supplier's instructions.

#### 4.7 Test methods.

##### 4.7.1 Homogeneity.

4.7.1.1 Uniformity of single pours. The test block molded in accordance with 4.6.2.1.2 shall be visually examined on all surfaces for any void openings, cracks, or any other occlusions. The test block shall be cut horizontally into three equal sections and the inside surfaces examined in a similar manner. Any void openings or cracks shall be cause for rejection.

4.7.1.2 Uniformity of multiple pours. The two vertical surfaces of the multiple pour specimen prepared in accordance with 4.6.2.2 shall be visually examined for any void openings, cracks, or any other occlusions. The fusion line between successive pours shall be examined for adhesion, separation, cracks, or any significant amount of non-uniformity. A significant lack of adhesion between successive pours, or the existence of void openings or cracks shall be cause for rejection.

##### 4.7.2 Density variation.

4.7.2.1 Core density. A 4 by 4 by 4 inch block shall be cut near the center of the test block molded in accordance with 4.6.2.1.2. The 4 by 4 by 4 inch block shall then be machined to assure accuracy of dimensions and weighed to the nearest tenth of a gram. The core density shall be calculated in pounds per cubic foot.

4.7.2.2 Overall density. The test block molded in accordance with 4.6.2.1.2 shall be accurately measured, weighed, and its overall density calculated in pounds per cubic foot. It might be necessary to cut 1/4 inch from the top of the cubic foot sample to assure accurate measurement. A 4 by 4 by 4 inch block shall be cut from diagonally opposite corners of both the top and bottom section of the test block and their densities accurately calculated in pounds per cubic foot. The densities of the top and bottom blocks shall not vary by more than plus or minus 5 percent from the core density. This variation allowance is subject to the maximum density specified in 3.4.

##### 4.7.3 Application properties.

4.7.3.1 Initial viscosity. The initial viscosity determination on the freshly mixed syntactic material shall be made with the Brookfield Viscosimeter, Model RVF (or equal) with a no. 7 spindle operated at 10 revolutions per minute (r.p.m.). Readings shall be taken when the pointer assumes a reasonably steady position. Because of the nature of the material, exact readings are difficult to obtain. The components of the syntactic material shall be kept at  $73.5^\circ \pm 1.8^\circ\text{F.}$  ( $23^\circ \pm 1^\circ\text{C.}$ ) for 4 hours prior to mixing.

4.7.3.2 Application life. The application life of the syntactic material shall be measured at the end of a 1-hour period, from the time of addition of catalyst. The viscosity of the mixed syntactic material shall be determined using a Brookfield Viscosimeter, Model RVF (or equal) with a no. 7 spindle operated at 10 r.p.m. Readings shall be taken when the pointer first assumes a reasonably steady position after release of the clutch. Readings shall be taken at  $73.5^\circ \pm 1.8^\circ\text{F.}$  ( $23^\circ \pm 1^\circ\text{C.}$ ). The material must still be pourable 1 hour from the time of addition of catalyst.

4.7.3.3 Cure time. The test specimens cut from the test block of syntactic material prepared in accordance with 4.6.2.1.2 shall be capable of passing the test requirements specified herein within 7 days from the time of addition of catalyst.

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4.7.3.4 Peak exotherm. The peak exotherm of the syntactic material during curing shall be measured by inserting a thermocouple into the center of the test block specified in 4.6.2.1.2 prior to the start of curing. The temperature readings shall be taken every 15 minutes until the maximum temperature of 300°F. is obtained using a potentiometer connected to the thermocouple leads.

4.7.3.5 Shrinkage. The test block of syntactic material molded in accordance with 4.6.2.1.2 shall be measured for shrinkage from the inside of the mold. The shrinkage of the block is the difference between the width of the mold and the width of the block. The width of the block shall be measured 6 inches down from the top of the block. The percent shrinkage shall be calculated as follows:

$$\text{Percent shrinkage} = \frac{W_1 - W_2}{W_1} \times 100$$

Where:

$W_1$  = Inside width of the mold.

$W_2$  = Width of the cured test block of syntactic material.

#### 4.7.4 Mechanical and physical properties.

##### 4.7.4.1 Compressive yield strength (0.2 percent offset).

4.7.4.1.1 Dry (initial). The compressive yield strength of the syntactic material shall be determined by ASTM D1621-64 except that the number and size of specimen shall be as indicated in table II. The specimen shall be cut from the test block molded in accordance with 4.6.2.1.2. A load-deformation curve shall be obtained either by automatic recordings or by plotting dial gage deformation readings against load readings. A stress strain curve shall be plotted from the load deformation curves. The compressive strength shall be the stress at 0.2 percent offset yield determined from the stress-strain curve.

4.7.4.1.2 Wet (after hydrostatic testing). The test specimens for the syntactic material shall be cut to the dimensions specified in table II from the test block molded in accordance with 4.6.2.1.2. The specimen shall be placed within a perforated metal cage in a suitable pressure chamber filled with tap water (temperature 65° - 90°F. (19° - 32°C.)) and exposed to 1000 p.s.i.g. hydrostatic pressure for 24 hours. The specimen shall be removed and tested for compressive yield strength as specified in 4.7.4.1.1 within 2 hours after exposure.

4.7.4.1.3 Compressive yield strength after humidity aging. The specimens for the humidity aging shall be cut to the dimensions specified in table II from the test block molded in accordance with 4.6.2.1.2. The specimen shall be placed on a perforated rack in a closed container having approximately 1 inch of water in the bottom, the rack being positioned at least 1 inch above the water. The top of the container shall be equipped with a 1-inch diameter hole for the insertion of a stirring rod. The container shall be placed in an air-circulating oven maintained at 158 + 1.8°F. (70° + 1°C) for 7 days. A suitable air-stirring device shall be inserted through the top of the container and operated for the first 4 to 6 hours of the exposure to obtain a water vapor saturated atmosphere within the container. The water level should be checked periodically throughout the exposure. If addition of water is required, it should be preheated to 160°F. After 7 days, the specimen shall be removed from the container, dried for 30 minutes at 158° + 1.8°F. (70° + 1°C.) and allowed to recover at 77° + 1.8°F. and 50 percent R.H. The specimen shall be tested for compressive yield strength as specified in 4.7.4.1.1 (first article only).

4.7.4.2 Modulus of elasticity in compression. The modulus of elasticity in compression is the ratio of stress to strain in the elastic portion of the curve and shall be calculated from the load deformation curve of 4.7.4.1.1 after hydrostatic exposure to 4.7.4.1.2.

4.7.4.3 Compression set after static loading. The test specimen shall be cut to the dimensions specified in table II from the test block molded in accordance with 4.6.2.1.2. The specimen shall be tested in a compression testing machine as specified in ASTM D1621-64. The load shall be applied to the test specimen at the rate of 0.1 inch/minute and held for 4 hours. The specimens shall be placed under a load of 1000 p.s.i. The specimen shall then be removed from the testing machine and allowed to recover for 30 minutes, before measuring final thickness. Set, expressed in inch/inch, is the difference between the initial and final thickness divided by the initial thickness (all measurements to the nearest 0.01 inch).

4.7.4.4 Impact test. The test specimen for the impact test shall be cut to the dimensions specified in table II from the test block molded in accordance with 4.6.2.1.2.

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4.7.4.4.1 Apparatus. The impact testing apparatus shall be constructed in accordance with figure 1.

4.7.4.4.2 Procedure. A 5-pound steel ball held by an electromagnet shall be dropped onto the center of the test specimen starting from a height of 1 foot. The height of drop shall be increased by 6-inch increments and the test shall be repeated until the test specimen fails. The impact strength is the number of foot pounds required to break the test specimen.

# 4.7.4.5 Water absorption. The test specimen for the water absorption test shall be cut to the dimensions as specified in table II from the test block molded in accordance with 4.6.2.1.2. The specimen shall be completely immersed in tap water (temperature 65° - 95°F.) for 5 seconds, removed, and allowed to drain for 10 seconds in still air by holding the specimen by one corner with the opposite corner vertically downward. The specimen shall be weighed to the nearest 0.1 gram and then subjected to the hydrostatic test of 4.7.4.1.2. After 24 hours remove the specimen from the test apparatus and drain and weigh it in the same manner in which the initial weight was obtained. The difference between the final and initial weight shall be the water pick-up of the specimen. Calculate the water absorption as percentage of the initial weight. Continue procedure using same specimen for a 7-day immersion period (7-day immersion test for first article only). Continue procedure for a 30-day immersion period (30-day immersion test only for microspheres and resin not previously tested).

4.8 Inspection of preparation for delivery. The packaging, packing, and marking shall be examined for compliance with section 5 of this specification.

#### 5. PREPARATION FOR DELIVERY

# (The preparation for delivery requirements specified herein apply only for direct Government procurements.)

#### # 5.1 Packaging and packing.

# 5.1.1 Level C. Material components, in the quantity specified (see 6.2 and 6.4), or a mock up section where specified (see 6.2 and 6.3) shall be packaged or packed in containers that will assure protection against deterioration, loss of contents, and physical damage during shipment from the supply source to the first receiving activity for immediate use. Packing (shipping containers) shall be acceptable to the common carrier which will insure safe delivery at destination in a satisfactory condition at the lowest applicable rate. Containers, packing, or method of shipment shall comply with Uniform Freight or National Motor Freight Classification Rules or Regulations or other carrier rules as applicable to the mode of transportation.

# 5.2 Marking. In addition to any special marking required by the contract or order, interior packages and exterior shipping containers shall be marked in accordance with MIL-STD-129 and shall include shelf life and hazardous material markings as applicable.

#### 6. NOTES

6.1 Intended use. The syntactic material covered by this specification is intended for use in structural void filling applications.

6.2 Ordering data. Procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) When atmospheric test conditions are other than specified (see 4.6.1).
- (c) Mock-up requirements (see 5.1.1 and 6.3).
- (d) Quantity of material required (see 5.1.1).
- (e) Handling characteristics of material.
- (f) Date of manufacture and batch number.

6.3 Mock-up section. The procuring activity may specify a mock-up model which would duplicate all or part of a section in the actual application giving such details as construction, material, thickness, cross-section, configuration, volume, and any other requirement necessary for satisfactory installation of the material (see 6.2).

6.4 Shelf life. Since the shelf life of some of the syntactic material components may be limited, the material should not be procured for storage but only for immediate application.

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6.5 First article inspection.

6.5.1 Invitations for bids should provide that the Government reserves the right to waive the requirement for samples for first article inspection as to those bidders offering a product which has been previously procured or tested by the Government, and that bidders offering such products, who wish to rely on such production or test, must furnish evidence with the bid that prior Government approval is presently appropriate for the pending procurement.

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6.6 Microsphere procurement. Procurement activities should consider the specific gravity as well as the pound cost in response to invitations for bids since the lowest per pound cost for microspheres may not necessarily yield the lowest cost per cubic foot of syntactic material. The price per pound for microspheres multiplied by the number of pounds of microspheres required to make 1 cubic foot of syntactic material should provide the basis for procurement within the maximum density constraint for the syntactic material.

6.7 THE MARGINS OF THIS SPECIFICATION ARE MARKED "#" TO INDICATE WHERE CHANGES (ADDITIONS, MODIFICATIONS, CORRECTIONS, DELETIONS) FROM THE PREVIOUS ISSUE HAVE BEEN MADE. THIS WAS DONE AS A CONVENIENCE ONLY AND THE GOVERNMENT ASSUMES NO LIABILITY WHATSOEVER FOR ANY INACCURACIES IN THESE NOTATIONS. BIDDERS AND CONTRACTORS ARE CAUTIONED TO EVALUATE THE REQUIREMENTS OF THIS DOCUMENT BASED ON THE ENTIRE CONTENT IRRESPECTIVE OF THE MARGINAL NOTATIONS AND RELATIONSHIP TO THE LAST PREVIOUS ISSUE.

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