

**Ministry of Defence
Defence Procurement Agency, ADRP2
Abbey Wood
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BS34 8JH**

OBSOLESCENCE NOTICE

All DTD specifications were declared obsolescent from 1st April 1999. All DTD 900 series approvals also lapsed at that time. The standards will no longer be updated but will be retained as obsolescent documents to provide for the servicing of existing equipment.

Further Guidance

The aim in declaring the specifications obsolescent is to recognise that the documents are not being updated and thus should be used with care by both purchaser and supplier. For example, a specification could contain valid technical information but may also contain type approval clauses that contradict procurement policy and/or use materials that do not comply with environmental legislation. The obsolescent specification can still be used as a basis for a purchase provided that the supplier and purchaser agree suitable changes to the specification within the purchase order/contract.

For the DTD 900 system, each specification has provided an MoD approved material and process. For these items, the declaration of obsolescence will constitute the termination of both the extant MoD approval and the continuing MoD assessment that had underpinned those approvals. Again, the technical content of the document remains valid and can be used by both purchaser and supplier as a basis for a contract but an acceptable (to the parties) approval/assessment procedure would be required.

Aerospace Material Specification
UNSATURATED POLYESTER RESINS
FOR LOW PRESSURE LAMINATING PURPOSES

NOTE 1. This specification is one of a series issued by the Procurement Executive, Ministry of Defence, either to meet a limited requirement not covered by an existing British Standard for aerospace material, or to serve as a basis for inspection of material, the properties and uses of which are not sufficiently developed to warrant submission to the British Standards Institution for standardization.

NOTE 2. It is one of a series of resin specifications in which the material is classified by the base polymer employed.

NOTE 3. The tests employed in this specification are chosen for their reproducibility and ability to control the properties of the material. They are not intended to be simulated service tests which, because of variability of test conditions, may be unsatisfactory for control purposes.

NOTE 4. The temperature limitations given in Section 1 are for guidance only, and the suitability of a particular resin for an application will depend on the time and conditions of service. Users are advised to confirm the fitness of a resin for a specific purpose by appropriate service tests.

NOTE 5. This Specification calls for the use of substances and test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves either the supplier or the user from statutory obligations related to health and safety at any stage of manufacture or use.

This Specification has been devised for the use of the Ministry of Defence and its contractors in the execution of contracts for the Ministry and, subject to the Unfair Contract Terms Act 1977, the Ministry will not be liable in any way whatever (including but without limitation negligence on the part of the Ministry its servants or agents) where the Specification is used for other purposes.

SECTION 1

Scope

1.1 Scope

The materials covered by this specification shall be synthetic resins of the unsaturated polyester type which can be cured using suitable curing systems so that they may be used in the manufacture of high strength fibre reinforced composites. The specification shall be applicable to resins as supplied by resin manufacturers. These include not only unmodified polyester resins but also resins which have been modified by the inclusion of constituents such as thixotropic agents, fire retardants, pigments, fillers etc.

1.2 Classification

The resins, to which this specification applies fall into two main types:

Type M - For use where mechanical properties only are of primary importance.

Type E - For use where both mechanical and electrical properties are of primary importance.

Both types are subdivided, according to temperature resistance, into five classes as follows:

Classes MA and EA - For use at temperatures not exceeding 70°C.

Classes MB and EB - For use at temperatures not exceeding 90°C.

Classes MC and EC - For use at temperatures not exceeding 150°C.

Classes MD and ED - For use at temperatures not exceeding 200°C.

Classes MF and EF - For use at temperatures not exceeding 260°C.

The type and class required must be stated on all drawings and contracts.

1.3 Definitions

The following definitions apply to some of the terms used herein:

1.3.1 Low pressure laminating. A process of laminating or moulding in which the applied moulding pressure is within the range 0 to 1.4 MPa.

1.3.2 Unsaturated polyester resin. A solution in styrene (or other reactive monomer or mixture of monomers) of a polymer in which the structural units are linked by ester groupings obtained by the condensation of one or more polycarboxylic acids (and if desired a minor proportion of a monocarboxylic acid) with one or more polyhydric alcohols (and if desired a minor proportion of a monohydric alcohol). At least one of the acids or alcohols is ethylenically unsaturated.

- 1.3.3 Catalyst. An additive which increases the rate of curing of unsaturated polyester resin.
- 1.3.4 Accelerator. An additive which enhances the effect of a catalyst in increasing the rate of curing of a polyester resin. The term "Promotor" is sometimes used in this sense.
- 1.3.5 Batch. A quantity of material so designated by the supplier and substantially uniform in quality.

SECTION 2

Related Documents

2. Reference is made in this document to the following:

- BS 188 Method for the determination of the viscosity of liquids in cgs units.
- BS 733 Density bottles
- BS 903 Methods of testing vulcanized rubber
- BS 1595 Isopropyl alcohol
- BS 2782 Methods of testing plastics
- BS 3396 Woven glass fibre fabrics for plastics reinforcement.

The related documents listed are those applicable at the date of publication of this specification. Their current applicability must be confirmed by all users of this specification. The Quality Assurance Authority will supply, on request, information concerning any changes that may be necessary due to cancellation, replacement, suppression or amendment of any related document.

SECTION 3

Instructions for use

3.1 Test instructions

- 3.1.1 The manufacturer shall specify the constituents of the curing system to be used in testing his resin for compliance with this specification, and the same constituents must be used throughout the tests.
The manufacturer shall state the proportions in which the constituents are used for (a) determining the gelation time and (b) preparing a catalysed resin system for manufacture of the test laminate.
The pot life of the laminating system must also be stated, ie the maximum time in an ambient air temperature of 25°C during which the mixed system will remain fit for use and provide a laminate complying with the requirements of this specification.
- 3.1.2 The resin manufacturer shall also identify fully the glass fibre reinforcement to be used in preparing the test laminate, and shall define the curing conditions, including post cure if applicable.

3.2 Instructions for general use

For general use the manufacturer shall preferably provide a list of curing systems which may also be used for his resin, and should be prepared to specify to the user, on request, recommended formulations and cure and post cure conditions for any of these alternative curing systems.

3.3 Precautions

Any necessary precautions to be taken when handling, mixing or curing the resin must be clearly stated.

SECTION 4

Storage Life

- 4.1 The manufacturer shall state the storage lives of the polyester resin when stored in the original sealed container at (a) 25°C maximum and (b) 40°C maximum.
- 4.2 It is emphasised that the resin must conform fully with all the requirements of the specification for the whole of the periods stated to be storage lives.
- 4.3 The manufacturers should state clearly on the containers the preferred conditions of storage and the storage life under these conditions (see Section 8)
- 4.4 Storage life shall be quoted in months from the date of dispatch by the manufacturer.

SECTION 5

Type Approval

- 5.1** Before any particular resin can be accepted as complying with the requirements of this specification it shall have received type approval. To obtain such approval the manufacturer of the resin shall satisfy the type approval authority that the resin will meet all the appropriate requirements of this specification.
- 5.2** The type approval authority for material to this specification is:
 Director, Materials Quality Assurance (DMQA)
 Headquarters Building
 Royal Arsenal East
 Woolwich, LONDON SE18 6TD
- 5.3** When applying for type approval the manufacturer shall submit the following:
- 5.3.1 Full details of the chemical composition of the resin. This information will be treated as confidential.
 - 5.3.2 Test instructions as stipulated in Section 3.1. These instructions will be published as part of the official approval (see Note in Section 7. 1).
 - 5.3.3 A request for consideration for a particular Type and Class in Section 1.2
 - 5.3.4 A list of nominal values for the properties in Table 1 when determined by the listed test methods.
 - 5.3.5 Sufficient resin and associated materials (see 3.1) to enable all the tests in the specification to be carried out, including storage life tests at two temperatures.
 - 5.3.6 Laminates prepared by the manufacturer using resin identical to the sample resin, ie taken from the same resin batch and manufactured according to the instructions provided under Section 3.1. The total area of the laminates must be at least 0.36 m² and one dimension of each laminate must be at least 0.3 m.
 - 5.3.7 Results (except long term weathering results) obtained by the manufacturer on tests carried out according to this specification on resin from that batch of resin and on laminates prepared from the resin according to Section 3.1.
- 5.4** After formal type approval has been given, no changes in chemical composition or test instructions may be made without the prior written consent of the type approval authority.

SECTION 6

Routine Quality Control

6.1 Frequency of testing

A representative sample shall be drawn from each batch of resin and shall be tested for compliance with the requirements of Table 1.

6.2 Further testing

The Quality Assurance Authority named in the contract may at any time require the manufacturer to test for compliance with any of the provisions of this specification.

SECTION 7

Requirements

- 7.1** The requirements for the resin and resin system are given in Table 1.

Table 1

Material	Test	Test Requirement	Test Method
Resin	Density at 25°C Viscosity at 25°C Acid value, mg KOH/g	Nominal value \pm 0.01% Nominal value \pm 20% Nominal value \pm 4	Appendix 1 BS 188 BS 2782: Part 4: Method 432B
Resin System	Gelation time at 25°C Gelation time at 82°C	Nominal value \pm 20% Nominal value \pm 20%	BS 2782: Part 1: Method 111D BS 2782: Part 1: Method 111C
Fully cured resin	Barcol hardness, minimum	35	Appendix 2

NOTE. The gelation time test at 82°C is obviously inapplicable for a resin used primarily in 'room temperature' laminating systems, and for which the manufacturer has stipulated such a system as the cure system for test purposes.

Users are advised that the Approved Products List of materials complying with this specification identifies the test curing systems and conditions specified by the manufacturers. The absence of a gelation time test at 82°C for a particular resin can therefore be easily realized.

7.2 The physical and mechanical requirements for laminates prepared according to Appendix 3 are given in Table 2.

They are applicable for both Type M and Type E resins.

Table 2

	Test	Test Requirement	Test Method
(a)	Flexural strength, MPa (min)	380	BS 2782: Part 3: Method 335A
(b)	Flexural strength at high temperature after heat ageing MPa (min) (i) after 30 minutes (ii) after 192 ± 4 hours	140 170	Appendix 4
(c)	Flexural strength after weathering, MPa (min)	310	Appendix 5
(d)	Resistance to water (i) flexural strength after immersion, MPa (min) (ii) absorption of water mg (max) (iii) leaching in water mg (max)	310 80 5	Appendix 6
(e)	Resistance to liquids (i) retention of flexural strength, % (min) (ii) change in weight, %	80 ± 0.5	Appendix 7
(f)	Rate of burning, cm/min (max)	2.5	Appendix 8
(g)	Glass content, %	62.5 ± 2.5	Appendix 9
(h)	Physical appearance (i) as prepared (ii) after any of the exposure or immersion tests	No cracking, crazing, delamination or other signs of physical deterioration	Visual inspection

7.3 The electrical requirements for laminates are given in Table 3. They are applicable only to laminates prepared from Type B resins.

Table 3

Test	Test requirement		Test method
	Before storage	After storage for 21 days at $20 \pm 2^\circ\text{C}$ and $90 \pm 2\text{RH}$	
Permittivity	4.4 (max)	5.5 (max) and not more than 25% higher than before storage	Appendix 10
Loss tangent	0.020 (max)	0.025 (max) and not more than 25% higher than before storage	Appendix 10

SECTION 8

Packaging and Identification

The resin shall be packaged in secure sealed containers. Each container shall be clearly marked with

- (i) This specification number and the resin type and class.
- (ii) The name or trade symbol of the manufacturer.
- (iii) The manufacturer's grade reference and batch number.
- (iv) The maximum and minimum temperatures at which the contents should be stored.
- (v) Storage lives from the date of dispatch (see Section 4).

APPENDIX 1

Method for the determination of density of 25°C

The method shall be as described in BS 733, Appendix B. Other methods eg Westphal balance or simple hydrometer may be used if they can be shown to give the same result.

In case of dispute the method described in BS 733 shall be used.

APPENDIX 2

Method for the determination of the Barcol Hardness.

A test plate approximately 100 mm square and 4 mm thick shall be cast using a catalysed resin system having the composition stipulated for the preparation of test laminates. The cure and post cure conditions shall be as stated by the manufacturer.

The cast resin plate shall be conditioned for not less than 40 hours at $23 \pm 2^\circ\text{C}$ and 50 ± 5 RH, and its Barcol Hardness determined according to BS 2782 Part 10 method 1001.

APPENDIX 3

Method of preparation of test laminate

The reinforcing material shall be glass cloth to BS 3396 Part 3. The cloth shall be desized to level H and finished to Grade S.

The resin manufacturer shall specify (i) the glass cloth to be used, (ii) the curing system and its proportions and (iii) the conditions of cure and post cure.

The glass fabric shall be dried in a ventilated oven for 1 hour at $100 \pm 5^\circ\text{C}$ before use. The fabric shall be cooled in a desiccator and used immediately on removal. The fabric shall be impregnated with the resin to give a final glass content of 60-65% of the total weight, the criterion for thorough impregnation being that the wet impregnated fabric is uniformly translucent. Both impregnation and building up of the laminate shall be done in a room temperature of 17° to 25°C . The warp direction of the fabric in any ply shall be parallel to the weft direction in the adjacent ply of the laminate.

A suitable method of impregnating the glass cloth and preparing the laminate is as follows:

The weight of resin necessary to give the required resin content is calculated from the weight of glass cloth to be used. An excess (eg $1\frac{1}{2}$ times of this quantity of resin) necessary to produce void free laminates is then catalysed according to the manufacturer's instructions and is used immediately in the following way:

Twelve squares of glass cloth 30 cm square are dried in the manner detailed above and then kept in a desiccator. A suitable flat plate about 40 cm square is covered with a sheet of regenerated cellulose film.

Approximately one twelfth of the catalysed resin is poured on to the cellulose film and the first layer of glass is then laid on it and pressed by using a knife blade, squeegee roller, or other device until the fabric is wetted with the resin and becomes evenly coated and uniformly translucent. This part of the process should occupy one or two minutes only.

The procedure is repeated with successive superimposed layers of glass fabric, each rotated through 90° in relation to the preceding layer. All layers shall be the same way up.

The top layer is immediately covered with a sheet of film followed by a second flat plate. "stops" 3 mm thick are placed between the upper and lower plates. Within 5 minutes of the completion of these operations the whole "build-up" is placed between the platens of a press which is closed and adjusted so that the plates are closed to the "stops" and held under a positive pressure of 0.05 to 0.10 MPa during the curing schedule.

It is permissible to adjust the height of the "stops" to accommodate for variation in the weight of glass fabric actually used. By this method laminates may be prepared which have final glass contents within the permitted 60-65% range.

Any post-stoving treatment to the laminate shall be carried out according to the resin manufacturer's instructions.

APPENDIX 4

Method for the determination of flexural strength after heat ageing

The flexural strength shall be determined at the maximum temperature for the particular grade of resin under test. A tolerance of $\pm 2^\circ\text{C}$ shall be permitted on test temperatures up to and including 150°C , and $\pm 5^\circ\text{C}$ for higher temperatures.

The test will be in accordance with BS 2782: Part 3: Method 335A

APPENDIX 5

Method for the determination of flexural strength after weathering

Pieces of laminate shall be exposed at 45° to the horizontal, facing south. At intervals of 15 days the pieces of laminates shall be turned over and at intervals of one month they shall be exposed to the maximum temperature for the appropriate grade for 30 minutes. Where this temperature is higher than 100°C the laminates shall be pre-heated for 1 hour at 100°C before exposure to the higher temperature. One piece shall be so exposed for 3 months and another piece for 1 year. The samples shall then be conditioned for not less than 4 days at $23 \pm 2^\circ\text{C}$ and 55 ± 5 RH and their flexural strengths determined in accordance with BS 2782: Part 3: Method 335A

APPENDIX 6

Method for the determination of resistance to water

(i) At least three test pieces shall be immersed in boiling distilled water for two hours after which they shall be cooled in distilled water at room temperature for at least 15 minutes and a maximum time of 1 hour and dried with a clean cloth. Their flexural strengths shall be determined within 5 minutes of removal from the cold water in accordance with BS 2782: Part 3: Method 335A

(ii) and (iii) Three specimens shall be prepared and tested according to BS 2782 Method 502G, with the additional requirement that if W_3 is less than W , the difference shall be recorded and reported as water-soluble matter.

APPENDIX 7

Method for the determination of resistance to chemicals representative of aircraft liquids.

The liquids used shall be:

- (i) iso-propyl alcohol to BS 1595
- (ii) a mixture of 30 parts by volume of pure toluene and 70 parts by volume of pure iso-octane (2, 2, 5-trimethyl pentane)
- (iii) liquid 101 (BS 903 Part A16)
- (iv) tri-n-butyl phosphate (density 0.975-0.976 g/cc; refractive index 1.424-1.425; acidity (as H_3PO_4) 0.15% maximum).

15 test pieces, ie 5 sets of three, shall be measured and weighed. They shall be immersed in the liquids listed above, one set per liquid for 24 hours at $25 \pm 2^\circ\text{C}$. Each test piece shall be totally immersed separately in 50 ml of liquid by being placed on edge in a container and supported at an angle by the bottom and sidewall of the container.

After removal from the liquid the samples shall be wiped free of surface liquid and immediately weighed and the flexural strength determined according to BS 2782: Part 3: Method 335A.

The mean of the three determinations shall be recorded as the result and the retention of flexural strength expressed as a percentage of the original flexural strength.

The mean of the three determinations of percentage change in weight will be taken as the change in weight.

The results for each liquid will be reported separately.

APPENDIX 8

Method for the determination of rate of burning.

Three test specimens each approximately 15 cm long by 1.25 cm wide shall be cut in any direction from the test laminate. Each specimen shall be marked by scribing two lines 2 cm and 12 cm from one end. The other end shall be clamped in a support so that the longitudinal axis of the specimen is horizontal and the transverse axis inclined at 45° to the horizontal. Under the test specimen, there shall be clamped a piece of 18 mesh clean wire gauze about 12 cm square in a horizontal position 0.5 cm below the edge of the specimen, and with about 1 cm of the specimen extending beyond the edge of the gauze.

A bunsen burner (1 cm minimum outside diameter) with a luminous flame 1.25 cm to 2.0 cm height shall be placed under the free end of the test specimen and adjusted so that the flame tip is just in contact with the specimen. At the end of 30 seconds, the bunsen flame shall be removed and the specimen allowed to burn. A stop watch shall be started when the flame reaches the first mark from the end and the time observed when the flame reaches the second mark 10 cm away. The mean rate of burning determined from the three tests shall be calculated. If any specimen fails to continue to burn after the first ignition, the burner shall again be placed under the free end for a second period of 30 seconds immediately following the extinction of the first flame. If the specimen again fails to continue burning to the second mark, the sample shall be reported as "fails to burn to the second mark".

The tests shall be performed in a room or chamber protected from air currents but provided with means for venting the fumes from the burning specimens.

The mean of the 3 results shall be recorded as the rate of burning.

APPENDIX 9

Method for the determination of glass content

Three specimens each not larger than 2.5 cm square shall be cut from each test laminate. The sides shall be square to the faces and the edges shall not be frayed. Each specimen shall be weighed in a previously ignited, cooled and weighed crucible and then placed in a furnace at a temperature not higher than 340°C. The temperature of the furnace shall be raised to 550°-660°C at a rate that will not cause blowing or loss of inorganic filler. Ignition shall be to constant weight of the crucible and contents, cooling being carried out in a desiccator. The glass content shall be calculated on the basis of the percentage of residue after ignition corrected for residues from the additives (if any) incorporated in the resin.

The mean of three determinations shall be recorded as the glass content.

The glass content of all test laminates must be determined.

APPENDIX 10

Methods for the determination of permittivity and loss tangent.

3 test pieces cut from a laminate shall be exposed for 3 hours to the maximum temperature appropriate for the class of resin under test, then allowed to cool to room temperature in an atmosphere with a relative humidity of 55 ± 5 per cent and a temperature of $23^\circ \pm 2^\circ\text{C}$, under which conditions they shall be stored for at least 4 days.

The permittivity and loss tangent of test pieces shall be measured at a radio frequency of 9,375 megacycles per second. A wave-guide of internal cross section of 0.900 x 0.400 in shall be used to hold the test piece. For dielectric constant measurements a short circuit method shall be used and for loss tangent measurements an insertion method.

For the tests after storage at $90 \pm 2\%$ relative humidity and $20^\circ \pm 2^\circ\text{C}$ test pieces shall be kept for 3 weeks in an enclosed chamber. All samples shall be tested within 15 minutes of removal from the chamber.

The mean of three determinations shall be recorded as the result.

Approved for issue,

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First published 1981

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ISBN 0 11 771678 2