

BS EN 4106:2013



BSI Standards Publication

Aerospace series — Non-metallic materials — Structural adhesive systems — Paste adhesive — Technical specification

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National foreword

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Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 4106:2013) has been prepared by the Aerospace and Defence Industries Association of Europe - Standardization (ASD-STAN).

After enquiries and votes carried out in accordance with the rules of this Association, this Standard has received the approval of the National Associations and the Official Services of the member countries of ASD, prior to its presentation to CEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2013, and conflicting national standards shall be withdrawn at the latest by July 2013.

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Introduction

This standard is part of the series of EN non-metallic materials standards for aerospace applications. The general organisation of this series is described in EN 4385. This document is a level 3 document as defined in EN 4385. It has been prepared in accordance with EN 4387.

1 Scope

This European Standard defines the requirements for manufacture, qualification, inspection and testing of products in structural adhesive systems where the adhesive is supplied in the form of a paste, either a one-part or two-part system, for aerospace applications. The adhesive may be used in conjunction with a primer whose requirements are also included in this European Standard. It is applicable whenever referenced on a material standard.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 542, *Adhesives — Determination of density*

EN 924, *Adhesives — Solvent-borne and solvent-free adhesives — Determination of flashpoint*

EN 2243-1, *Aerospace series — Non-metallic materials — Structural adhesives — Test methods — Part 1: Single lap shear*

EN 2243-2, *Aerospace series — Non-metallic materials — Structural adhesives — Test method — Part 2: Peel metal-metal*

EN 2243-6, *Aerospace series — Non-metallic materials — Structural adhesives — Test method — Part 6: Determination of shear stress and shear strain*

EN 2379, *Aerospace series — Fluids for assessment of non-metallic materials* ¹⁾

EN 2757, *Aerospace series — Structural adhesives system — Test method — Determination of the drying and ignition residues of primers*

EN 2781, *Aerospace series — Non-metallic materials — Structural adhesives — Test methods — Determination of the primer thickness*

EN 4385, *Aerospace series — Non-metallic materials — General organisation of standardisation — Links between types of standards* ¹⁾

EN 4387, *Aerospace series — Non-metallic materials — Rules for drafting and presentation of technical specifications* ¹⁾

EN 6040, *Aerospace series — Non-metallic materials — Test method — Analysis of thermoset systems by High Performance Liquid Chromatography (HPLC)* ¹⁾

EN 6041, *Aerospace series — Non-metallic materials — Test method — Analysis of non-metallic materials (uncured) by Differential Scanning Calorimetry (DSC)* ¹⁾

EN 6042, *Aerospace series — Organic compounds — Test method — Analysis by infrared spectroscopy* ¹⁾

EN 6064, *Aerospace series — Non-metallic materials — Analysis of non-metallic materials (cured) for the determination of the extent of cure by Differential Scanning Calorimetry (DSC)* ¹⁾

¹⁾ Published as ASD-STAN Prestandard at the date of publication of this standard (www.asd-stan.org).

EN 9100, *Quality Management Systems — Requirements for Aviation, Space and Defence Organizations*

EN 9133, *Aerospace series — Quality management systems — Qualification procedure for aerospace standard parts*

EN 12092, *Adhesives — Determination of viscosity*

ISO Guide 22, *General criteria for supplier's declaration of conformity*

ISO 3768:1976, *Metallic coatings — Neutral salt spray test (NSS test)*

ESA PSS-01-702, *A thermal vacuum test for the screening of space materials*

TR 7000-6, *Aerospace series — Non-metallic materials — Rules for the drafting and presentation of material standards — Part 6: Structural adhesive systems*²⁾

3 Terms, definitions, symbols and abbreviations

For the purposes of this document, the terms, definitions, symbols and abbreviations given in EN 9100, EN 9133, TR 7000-6 and the following, apply.

3.1 batch
quantity of adhesive material which is homogenous and has been manufactured from a single mix of each of its individual ingredients in one continuous production run

3.2 product
in this document the word product refers to all forms of paste adhesive, either a single component or comprised of two parts

3.3 adhesive system
combination of a paste adhesive and primer

3.4 adhesive material
any paste or primer of which an adhesive system may be composed

3.5 filler
inorganic material incorporated in an adhesive to improve its cure rheology and service performance

3.6 corrosion inhibitor
substance added to the primer to improve its corrosion inhibiting characteristics

2) Published as ASD-STAN Technical Report at the date of publication of this standard (www.asd-stan.org).

3.7

primer

material used to coat the substrates prior to the application of adhesive to improve either the durability of the joint or adhesion characteristics and/or to protect a pre-treated metal surface prior to bonding, so allowing an increased period of handling

Note 1 to entry: Standard adhesive primers for adhesive systems consist of solvents and polymers.

Note 2 to entry: Corrosion inhibiting adhesive primers for adhesive systems consist of solvents, polymers and solid inhibitors. The solid inhibitors reduce corrosion of the substrate.

3.8

shelf life

period of time during which the adhesive material when stored in accordance with the specified conditions shall meet the specified property requirements

3.9

shop life

maximum cumulative period of time during which the adhesive material can be held out of refrigerated storage before commencement of cure

3.10

pot life

period of time at room temperature during which a two part paste adhesive and/or a primer may be used, after mixing

3.11

lot

consignment of adhesive material which is either whole or part of a batch

3.12

T_{min} .

minimum service temperature of the material

3.13

T_{max} .

maximum service temperature of the material

3.14

T_g

glass transition temperature of the material

4 Requirements

4.1 General requirements

4.1.1 Manufacturing schedule for paste adhesive

The product shall be manufactured to fulfil the requirements of the relevant material standard and this technical specification in accordance with EN 9133. The manufacturer shall define the raw material, processes and inspection requirements in a manufacturing schedule. Once approved any modifications to the manufacturing schedule shall be in accordance with EN 9133.

4.1.2 Traceability

All products shall be traceable to the raw material batches at all stages of manufacture and delivery. Similarly, each raw material batch shall be traceable to all products at all stages of manufacture and delivery.

4.1.3 Freedom from defects

All products shall be free from defects not complying with the requirements of the material standard or order and this technical specification, or which may be prejudicial to the subsequent manufacture and/or use of the products.

Each primer component shall:

- a) be uniform in colour;
- b) be free from deleterious contamination, foreign matter or other defects;
- c) mix readily to a homogenous solution or suspension of consistency suitable for application;
- d) be free from lumps;
- e) not settle out or separate during a normal working day or the pot life, whichever is the shorter.

The adhesive shall:

- 1) be uniform in colour;
- 2) be free from deleterious contamination, foreign matter or other defects;
- 3) be free from lumps.

In the case of two part adhesives it shall also:

- i) mix readily to a homogenous solution or suspension of consistency suitable for application;
- ii) not settle out or separate during a normal working day or the pot life, whichever is the shorter.

4.1.4 Health and safety

It is the responsibility of the supplier to establish satisfactory health and safety information to ensure conformity with any European, National or local laws/regulations.

The safety data sheet shall be available and shall be provided prior to qualification testing.

4.1.5 Dimensions

Not applicable.

4.2 Technical requirements

4.2.1 General

The product shall satisfy the requirements of the relevant material standard and/or the order.

4.2.2 Required testing

Unless otherwise specified in the material standard, the test method and the test frequency for screening, qualification testing and release testing are given in Table 3 to Table 7.

The manufacturer, having regard for the provisions of EN 9133, shall implement a test programme under the supervision of the mandated body, on a minimum of three production batches of both the primer and the adhesive.

For the purposes of qualification a fixed combination of primer and adhesive, as defined in the material standard, shall be used throughout. Each pair of adhesive and primer involves a separate qualification programme.

Alloys, surface preparation, test panel manufacturing, machining and conditioning before testing of test pieces shall conform to the requirements of the Material Standard.

4.2.3 Test samples

Samples and associated test pieces shall be marked in such manner that their identity with respect to the product and the batch is maintained.

Samples taken from fully finished products (condition of use) shall not be further processed.

Samples representing products in a condition other than the condition of use shall be processed in accordance with the material standard before testing.

4.2.4 Shelf life

The adhesive system shall maintain all of its physical, chemical and mechanical properties within the specification requirements for the period of time (shelf life) specified in the material standard, when stored under the specified conditions.

Shelf life shall start from the date of manufacture.

4.2.5 Shoplif

The product shall meet the physical, chemical and mechanical properties of the material standard for a period of time known as the shoplif during storage under normal shop conditions. These requirements will be met at all stages of the shelf life.

4.2.6 Requirements specific to paste adhesive systems

4.2.6.1 Colour

The colour of the primer and adhesive shall be established between the manufacturer and the mandated body at the time of qualification.

4.2.6.2 Composition

There shall be no restriction on the chemical type or composition of the material except as limited by the material standard.

4.2.6.3 Curing

The adhesive and/or primer shall cure satisfactorily at the temperature, pressure, time, vacuum and ventilation under breather layers as specified in the material standard.

5 Qualification

5.1 Introduction

To use a certain product in an aerospace application product qualification alone may not be sufficient; additional pre-and/or post-qualification testing may be required by the user:

- Pre-qualification: manufacturer's data or screening test to verify basic properties according to the requirements of the relevant materials standard and processes routes. Results from this testing may be used as part of the qualification.
- Post-qualification: specific components tests, when required, separately specified as part of the project certification process. These components tests are the responsibility of the user and are not covered by this document.

5.2 Approval of quality system

See EN 9100.

5.3 Product qualification

See EN 9133.

5.4 Test requirements

5.4.1 Material Qualification

If the manufacturer cannot perform the required testing, a material test laboratory acceptable to the mandated body shall be appointed.

For new products or new manufacturers the full qualification testing shall be required (see Table 3 to Table 7).

5.4.2 Reduced qualification

If the product is already qualified by an aerospace company or was submitted to a screening programme against a recognised materials standard (e.g. national or company specification) the mandated body in conjunction with (potential) user(s), may, if sufficient data (a qualification test report) is available, waive all or part of the qualification requirements tests of the material standard.

Reduction in qualification testing may also be considered for other cases, such as discontinued and restarted supply or the production of related product forms. (For example different grades of a product based on common formulation).

5.4.3 Re-qualification

See EN 9133.

5.5 Qualification re-tests

A re-test may be required when one or more test results do not meet the requirements of the product qualification test. A re-test shall be performed on products from the same batch or batches agreed with the mandated body. All tests and re-test results shall be reported.

5.6 Qualification test report

The product qualification test report shall contain the following information:

- manufacturer's name and address;
- tested products;
- manufacturing schedule;
- material standard number;
- batch number(s);
- test piece manufacture;
- test piece conditioning;
- test and re-test results.

5.7 Interpretation of test results

See EN 9133.

6 Release testing

6.1 General

Series products shall be manufactured in accordance with the manufacturing schedule agreed during qualification and any agreed revisions. Batch release testing to the requirements of the material standard shall be the responsibility of the manufacturer. The test samples extracted shall be representative of the whole batch delivered in accordance with the relevant material standard. The purchaser reserves the right to perform any of the inspections and/or tests required by the material standard.

When required, the manufacturer shall inform the purchaser or his chosen agent of the planned dates for the extraction of samples and release testing, in order that these operations may be witnessed.

6.2 Sampling

6.2.1 Primer

For inspection and test purposes each batch of adhesive primer shall be divided into elementary units of 40 l.

The number of elementary units to be taken from a specific batch size for acceptance testing is shown in Table 1.

Table 1 — Sampling plan for acceptance testing of primer

Batch size in litres	Quantity of elementary units
$v < 40$	1
$40 \leq v < 240$	2
$240 \leq v < 1\,200$	3
$1\,200 \leq v < 2\,000$	4
$v \geq 2\,000$	5

6.2.2 Adhesive

For inspection and test purposes each batch of paste adhesive shall be divided into elementary units of 250 l.

The number of elementary units to be taken from a specific batch size for acceptance testing is shown in Table 2.

Table 2 — Sampling plan for acceptance testing of paste adhesives

Batch size in litres	Quantity of elementary units
$v < 250$	1
$250 \leq v < 500$	2
$500 \leq v < 750$	3
$750 \leq v < 1\,000$	4
$1\,000 \leq v < 1\,250$	5
$v \geq 1\,250$	6

6.3 Required tests

The tests required for batch release are listed in Table 3 to Table 7.

6.4 Conformance

6.4.1 General

Release testing shall conform to the requirements specified in the relevant material standard. If the procedure or the test piece preparation is faulty, testing shall be repeated after rectification of the original cause of failure.

6.4.2 Rejection

Any failure of the product to meet the requirements of the relevant technical specification shall be cause for rejection of the product, or re-test, as appropriate.

6.4.3 Re-tests

When failure cannot be attributed to faulty testing, or test piece preparation, further test samples shall be selected.

The original elementary unit shall be re-tested unless already withdrawn by the manufacturer after suitable identification of the cause of failure. Additionally, a second set of tests shall be undertaken on a separate elementary unit from the same batch.

In the case of an elementary unit which has been withdrawn, testing shall be undertaken on two additional separate elementary units.

If all re-test results conform the batch shall be accepted.

If one or more re-tests do not conform the batch shall be rejected.

6.5 Special tests

Special tests may be required by the purchaser. In such cases, the nature of the test, method, frequency and technical requirements shall be specified on the order or inspection schedule and shall be mutually agreed by the manufacturer and purchaser.

6.6 Capability clause

Reference to “capability clause” signifies that sufficient evidence of a statistical nature with respect to the properties under consideration can be submitted to show that the requirements of the relevant standard will be met on the basis of a reduced amount of testing. Such action in no way reduces the obligations of the manufacturer to fulfil all requirements. If subsequent testing indicates that a product does not comply with the requirements, the batch shall be rejected.

6.7 Release test report

The manufacturer shall furnish, with each batch, a report stating the following:

- manufacturer’s name and address;
- material standard number;
- delivery condition of product;
- manufacturer’s designation;
- batch number;
- date of manufacture;
- any deviation from the material standard;
- requirements and results of the tests, re-tests and characterisation;
- validation by an identified, responsible person and date of report.

7 Preparation for delivery

7.1 Packaging

The product shall be packaged in such a manner as to prevent damage and contamination during handling, transportation and storage.

The primer shall be filled into airtight containers.

The adhesive shall be filled into airtight containers.

7.2 Marking

7.2.1 Product containers

Each product container shall be legibly and durably marked in such a manner and location that the marking remains in place until the product is completely used. The method of marking used shall not cause damage, deterioration or contamination. The marking shall remain permanently visible after handling, storage and contact with protective products.

Markings shall include, but not be limited to, the following information:

- manufacturer's name and address;
- manufacturer's designation;
- material standard number;
- batch number;
- date of manufacture, if appropriate, and expiry date;
- cautionary marking (if relevant);
- quantity;
- storage conditions.

This requirement applies equally to all components of the adhesive system.

7.2.2 Packages

The following information shall be accessible on the exterior of the packaging:

- name and address of recipient;
- name and address of supplier;
- quantity of product;
- order number(s);
- storage conditions;
- sufficient information to enable the contents to be related to the relevant documentation, if required.

7.3 Shipping conditions

Materials shall be transported under conditions which do not cause deterioration in their properties and storage life.

7.4 Storage prior to shipping

Materials requiring cold storage shall have this requirement clearly marked on the shipping containers. Materials shall be stored under conditions which do not cause deterioration in their properties and shelf life.

8 Declaration of conformity

With each batch of material released the manufacturer shall issue a declaration of conformity stating that the material meets the requirements of the order (see ISO Guide 22).

The following information shall also be provided:

- a) material standard number;
- b) manufacturer's product code;
- c) batch numbers;
- d) copy of manufacturer's quality control test report related to defects (if relevant);
- e) copy of manufacturer's release test report (see 6.7).

Table 3 — Testing requirements of the primer in the supplied condition

Characteristic number	Characteristic	Test method	Number of test pieces to be tested	
			Qualification testing (number per batch)	Acceptance testing (number per elementary unit)
007	Visual appearance of cured primer	a	3	1
102	Resin composition (by IR spectroscopy)	EN 6042	3	1
103	Resin composition (by chromatography)	EN 6040	3	1
	Solvent phase (by gas chromatography)	a	1	—
104	Corrosion inhibitor content	EN 2757	3	1
	Solid content	EN 2757	3	1
107	Viscosity	EN 12092	3	1
109	Flashpoint	EN 924	3	—
110	Thickness of cured primer	EN 2781	3	1
301	Density	EN 542	3	1
999	Notes	a To be agreed between purchaser and supplier at the time of qualification.		

Table 4 — Testing requirements of the paste adhesive in the supplied condition

Characteristic number	Characteristic	Test method	Number of test pieces to be tested	
			Qualification testing (number per batch)	Acceptance testing (number per elementary unit)
102	Resin composition (by IR spectroscopy)	EN 6042	3	1
103	Resin composition (by chromatography)	EN 6040	3	1
106	Curing characteristics by DSC	EN 6041	3	1
107	Flow	EN 12092	3	1
301	Density	EN 542	3	1

Table 5 — Testing requirements of the adhesive in the cured condition

Characteristic number	Characteristic	Test method	Number of test pieces to be tested					
			Qualification testing (number per batch)			Acceptance testing (number per elementary unit)		
			<i>T</i> min.	(23 ± 2) °C	<i>T</i> max.	<i>T</i> min.	(23 ± 2) °C	<i>T</i> max.
105	Glass transition temperature ^a	EN 6064	–	6	–	–	–	–
307	Lap shear strength	EN 2243-1 Type 1 specimen	6	6	6	–	6	6
	Shear stress-strain behaviour	EN 2243-6	6	6	6	–	–	–
316	Peel strength – metal to metal	EN 2243-2	6	6	6	–	6	–
999	Notes	^a Temperature range – 55 °C to > <i>T_g</i> .						

Table 6 — Ageing test – Qualification only

Environment		Environmental conditioning			Test type		Test method	Number of test pieces per batch		
Characteristic No.	Characteristic	Environment	EN 2379 ref.	Ageing condition	Characteristic No.	Characteristic		T min.	(23 ± 2) °C	T max.
501	Aeronautical fluids	Hydraulic fluid	2.1	(70 ± 2) °C / 2 000 h	307	Tensile shear	EN 2243-1	–	6	6
501	Aeronautical fluids	Jet fuel	7.1	(40 ± 2) °C / 2 000 h	307	Tensile shear	EN 2243-1	–	6	6
501	Aeronautical fluids	Phosphate ester hydraulic fluid	2.3	(70 ± 2) °C / 2 000 h	307	Tensile shear	EN 2243-1	–	6	–
501	Aeronautical fluids	50/50 mixture of phosphate hydraulic fluid/ demin. Water	2.4	(50 ± 2) °C / 2 000 h	307	Tensile shear	EN 2243-1	–	6	6
501	Aeronautical fluids	Deicing fluid	3.5	(0 ± 2) °C / 2 000 h	307	Tensile shear	EN 2243-1	–	6	–
502	Water	Demineralised water (electrical conductivity < 5 µ S / cm)	–	(70 ± 2) °C / 2 000 h	307	Tensile shear	EN 2243-1	–	6	6
503	Dry heat	Dry heat	–	T max. / 2 000 h	307	Tensile shear	EN 2243-1	6	6	6
503	Dry heat	Dry heat	–	–	505	Outgassing	ESA PSS-01-702	–	6	–
503	Dry heat	Dry heat	–	T max. / 2 000 h	316	Peel properties	EN 2243-2	–	6	–
504	Wet heat	95 % relative humidity	–	(70 ± 2) °C / 1 000 h	305	Shear stress/ strain	EN 2243-6	–	6	–
504	Wet heat	95 % relative humidity	–	(70 ± 2) °C / 2 000 h	307	Tensile shear	EN 2243-1	–	6	–
504	Wet heat	95 % relative humidity	–	(70 ± 2) °C / 14 d	105	Glass transition temperature	EN 6064	–	6	–
509	Salt spray	Salt spray to ISO 3768	–	(35 ± 2) °C / 45 d	316	Metal to metal peel	EN 2243-2	–	6	–
509	Salt spray	Salt spray to ISO 3768	–	(35 ± 2) °C / 90 d	316	Metal to metal peel	EN 2243-2	–	6	–
509	Salt spray	Salt spray to ISO 3768	–	(35 ± 2) °C / 180 d	316	Metal to metal peel	EN 2243-2	–	6	–
509	Salt spray	Salt spray to ISO 3768	–	(35 ± 2) °C / 300 d	316	Metal to metal peel	EN 2243-2	–	6	–

Table 7 — Storage and work life – Qualification (one batch only)

Environment		Environmental conditioning		Test type		Test method	No. of specimens
Characteristic No.	Characteristic	Environment	Ageing condition	Characteristic No.	Characteristic		
508	Ageing	Low temperature Storage – Shelf life of 1 part Adhesives only	≤ 18 °C / 0 d	105	Tg	EN 6041	2
			≤ 18 °C / 180 d				
			≤ 18 °C / 360 d ^a				
			≤ 18 °C / 720 d ^a				
508	Ageing	Low temperature Storage – Shelf life of 2 part adhesives only	(5 ± 2) °C / 25 d	105	Tg	EN 6041	2
			(5 ± 2) °C / 50 d				
			(5 ± 2) °C / 100 d				
508	Ageing	Shop life	30 °C, 75 % RH / 0 d	105	Tg	EN 6041	2
			30 °C, 75 % RH / 10 d				
			30 °C, 75 % RH / 20 d				
			30 °C, 75 % RH / 30 d				
			30 °C, 75 % RH / 35 d				
508	Ageing	Pot life – For 2 part Adhesives only	30 °C, 75 % RH / ½ h	105	Tg	EN 6041	2
			30 °C, 75 % RH / 1 h				
			30 °C, 75 % RH / 2 h				
			30 °C, 75 % RH / 5 h				
			30 °C, 75 % RH / 12 h				
999	Notes	^a For reference only, not to impede qualification.					

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Tel: +44 20 8996 7070

Email: copyright@bsigroup.com



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