

Adhesive for Bonding Fixed Glass of Automobile

1. Scope

This standard specifies one part urethane adhesive (hereinafter referred to as "adhesive") to be used for bonding fixed glass directly on a painted automobile body.

Remarks: In this standard, units and numerical values are based on SI (International System Units), while units and numerical values given in { } are customary units system, and are given for reference.

2. Purpose

This standard aims to standardize the required performance and testing method of the adhesive for bonding fixed glass and to ensure the proper quality.

3. Definitions

Definitions of major terms used in this Standard are as follows:

3.1 Materials To Be Used

(1) One part urethane adhesive

Adhesive that cures into a rubber type elastic material by reacting with environmental moisture in room temperatures, and bonds and fixes the fixed glass on the body.

(2) Primer

For Glass: Primer coated for pretreatment, on the glass surface to be adhered to, in order to bond firmly the glass and the adhesive.

For coated surf: Primer coated for pretreatment, on the coating film surface to be adhered to, in order to bond firmly the body coating film surface and the adhesive.

3.2 Test Method

(1) Foreign substance

This shall be other than raw materials used for the adhesive, and be distinguished from raw materials insufficiently dispersed. However, even raw materials, coarse particles which are considered to be unusual shall be included in the foreign substance.

(2) Apparent viscosity

This shall mean the ratio of shearing rate to shearing stress, and shall be indicated in Pa.s{P} unit. This shall be calculated by the Poiseuille's Equation, and shall be used for non-Newtonian fluid.

(3) Shearing rate

This shall mean relative movement rate

Applicable Standards:	JIS D 0205	General Rules of Weatherability for Automobile Parts
	JIS G 3141	Cold Rolled Carbon Steel Sheets and Strip
	JIS K 1522	Isopropyl Alcohol (Isopropanol)
	JIS K 6301	Physical Testing Methods for Vulcanized Rubber
	JIS K 6830	Testing Methods for Sealant for Automobiles
	JIS K 8891	Methanol
	JIS R 3202	Float and Polished Plate Glasses
	JIS R 3503	Glass Apparatus for Chemical Analysis
	JIS Z 8401	Rules for Rounding off of Numerical Values
	JIS Z 8703	Standard Atmospheric Conditions for Testing
	ASTM D 1092	APPARENT VISCOSITY OF LUBRICATING GREASES
	DIN 65262	Zweikomponenten Dictmassen auf Basis Polysulfid-Polymeren
		Sealants for Automobile Bodies
Reference Standards:	JASO M323	

of a series of adjacent layers of non-Newtonian fluid. It is proportional to the value of linear flow rate divided by radius of capillary tube, and shall be indicated in reciprocation second (S^{-1}).

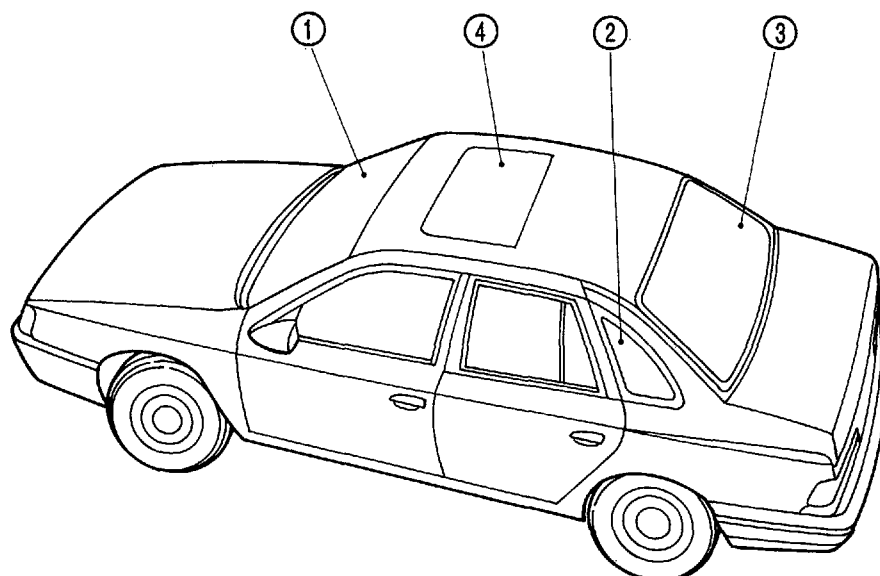
4. Parts of Application

Parts of application shall be as shown in **Table 1** and **Fig. 1**.

Table 1

Parts of Application	
①	Windshield
②	Side Glass
③	Rear Glass
④	Roof Glass

Fig. 1



5. Performance

5.1 General Performance Required

5.1.1 Composition

- (1) The adhesive shall contain terminal-isocyanate-prepolymer as principal ingredient, and shall become rubber type elastic material by reacting and curing in room temperatures.
- (2) The primer shall be of melting solution type, with principal ingredients including silane or polyisocyanate melted in organic solvent.

5.1.2 Safety of Working Environment

The adhesive and the primer shall not affect the skin, eye, respiratory organs, and so forth in ordinary working.

5.2 Detailed Performance Required

The adhesive and the primer shall hold performance as specified in **Table 2** to **5** in accordance with test procedures in **9**.

Table 2 Performance of Adhesive

Properties/characteristics			Item of evaluation	Test conditions	Test method Item No.	Performance	
Characteristics before curing	Appearance		Existence of abnormality such as foreign substance, etc.	Visual check	9.1	No abnormality	
	Specific gravity		Specific gravity	Aluminum plate for specific gravity measurement, Method A	9.2	1.50 or less	
			Specific gravity	Hemisphere cup, Method B	9.3	1.50 or less	
	Nonvolatile matter		Nonvolatile matter %	105°C, 3 hr. heating	9.4	96.0 or over	
	Viscosity		Apparent viscosity Pa.s{P}	Pressure-viscometer	9.5	30 to 100 {300 to 1000}	
	Sagging characteristics		Apex movement distance, mm	Triangular bead cross-section, base 8 mm, height 10 mm, Method A	9.6	3 or less	
			Movement distance of lower end, mm	38 mmφ, 9.5 mm thick pillar-shaped, angle 90°, Method B	9.7	3 or less	
	Curing rate	Tack-free time	Time for no material adhesion to polyethylene film, min.	Peeling after polyethylene press-bonding, 20°C, Method A	9.8	80 or shorter	
		Bonding strength	Tensile strength N(kgf)	Bead shape 10 x 5 x 75 mm 20°C, 65% RH; 3 hr., Method B	9.9	78.5 or over {8.0 or over}	
			Shear strength MPa(kgf/cm²)	Bead shape 10 x 5 x 25 mm 20°C, 65% RH; 3 hr., Method C	9.10	0.1 or over {1.0 or over}	
				Bead shape 10 x 5 x 25 mm 20°C, 65% RH; 24 hr., Method C		1.5 or over {15.0 or over}	
	Pot life		Failure condition of with in specified time	35°C, 90% RH, 5 min.	9.11	CF-100 (?)	
Coating film contamination properties		Existence of discoloration of coating film	Weathermeter 48 hr. after wiping off, visual observation, coating colour of white group (1)	9.12	No discoloration		
Characteristics after curing	Hardness		JIS Hardness	JIS hardness tester press pin type A (instantaneous value) Thickness, 6 mm or over	9.13	Normal 20°C, 168 hr.	35 to 75
				Heat resistance 90°C, 336 hr.		35 to 75	
				Water resistance 40°C water, 336 hr.		35 to 75	
				Cold resistance -40°C, 1 hr.		35 to 75	
	Tensile strength		Breaking strength MPa(kgf/cm²)	JIS dumbbell shape No. 3, thickness 2.0 to 3.0 mm		Normal condition	3.9 or over {40.0 or over}
						Heat resistance	3.9 or over {40.0 or over}
						Water resistance	3.9 or over {40.0 or over}
						Cold resistance	3.9 or over {40.0 or over}
	Elongation		Breaking elongation %	JIS dumbbell shape No. 3, thickness 2.0 to 3.0 mm		Normal condition	300 or over
						Heat resistance	300 or over
						Water resistance	300 or over
						Cold resistance	300 or over
	Ozone resistance		Existence of crack	JIS dumbbell shape No. 1, 20% elongation, 50 pphm, 40°C, 72 hr., visual observation	9.14	No crack	
	Corrosion resistance		Existence of detrimental rust	50°C, 90% RH, 168 hr., visual observation	9.15	No rust	

Notes (1) Details of coating colour shall be in accordance with agreements between the parties concerned to delivery.

(2) Cohesive failure of the adhesive is 100%.

Table 3 Performance of Primer

Properties/ characteristics	Item of evaluation	Test conditions	Test method Item No.	Performance	
				For coated surf.	For glass
Appearance	Existence of abnormality such as foreign substance, etc.	Visual check	9.16	No abnormality	
Specific gravity	Specific gravity	Specific gravity cup 20°C	9.17	0.80 to 1.20	0.80 to 1.20
Viscosity	Flow time, s	Ford cup for paint 20°C	9.18	8.0 to 18.0	8.0 to 18.0
Nonvolatile matter	Nonvolatile matter, %	105°C, 3 hr. heating	9.19	15.0 to 40.0	15.0 to 50.0
Coating uniformity	Existence of uniform film formation	Coated surface and coating on glass plate, visual check (clairvoyance)	9.20	Formation of uniform hiding film	
Coating film contamination properties	Existence of discoloration of coating film	Weather-o-meter 48 hr. after wiping off, visual check	9.21	No discolor action	—
Pot life	Failure condition with in specified time	5°C, 40% RH and 35°C, 90% RH, 3 min., 360 min.	9.22	CF-100	CF-100

Table 4 Adhesion Performance (Adhesive and Primer)

Properties/ characteristics	Item of evaluation	Test conditions		Test method Item No.	Performance	
					For coated surf.	For glass
Shear strength	Shear strength Failure condition MPa(kgf/cm ²)	Bead shape 10×5×25 mm	Normal condition @20°C, 65% RH, 168 hr.	9.23	1.5 or over {15.0 or over} CF-100	
			Heat resistance 90°C, 336 hr.		1.5 or over {15.0 or over} CF-100	
			Water resistance 40°C water, 336 hr.		1.5 or over {15.0 or over} CF-100	
			Cold resistance -40°C, 1 hr.		1.5 or over {15.0 or over} CF-100	
		Failure condition, visual check	Hot resistance 80°C, 1 hr.		1.5 or over {15.0 or over} CF-100	
			Accelerated weathering test 2,000 hr.		1.5 or over {15.0 or over} CF-100	
			Outdoor exposure test		1.5 or over {15.0 or over} CF-100	
			Windwasher (WW) fluid resistance 20°C fluid, 168 hr.		1.5 or over {15.0 or over} CF-100	
Peel strength	Failure condition	Adhesive thickness 3 mm	Normal condition	9.24	CF-100	CF-100
			Heat resistance		CF-100	CF-100
		Knife-cutting, hand-peeling	Water resistance		CF-100	CF-100
			Accelerated weathering test		—	CF-100
		Visual check of state	Outdoor exposure test		—	CF-100
			WW fluid resistance		CF-100	CF-100

Table 5 Storage Stability ⁽³⁾

Properties/ characteristics	Item of evaluation	Test conditions		Test method Item No.	Performance			
					Adhesive	Primer		
						For coated surf.	For glass	
Appearance	Existence of abnormality such as foreign substance, etc.	Visual check		9.25	No abnormality			
Viscosity change ratio	Rate of viscosity change to initial value %	Items of viscosity in Tables 2 and 3			50 or less	20 or less	20 or less	
Curing rate	Tack-free time min.	Item of tack-free time in Table 2			80 or less	—	—	
Characteristics of cured material	Hardness Tensile strength MPa(kgf/cm ²) Elongation %	Item of hardness, tensile strength and elongation in Table 2 . 20°C, 65% RH, 168 hr.	Hardness		35~75	—	—	
			Tensile strength		3.9 {40.0 or over}	—	—	
			Elongation		300 or over	—	—	
Peeling characteristics	Condition of failure	Item of peel strength in Table 4 20°C, 65% RH, 168 hr.			CF-100			
Pot life	Condition of failure after specified time	Same as the item of peel strength 20°C, 65% RH, 168 hr.	Primer		5°C 40% RH	3 min. and 360 min., all CF-100		
					35°C 90% RH			
			Adhesive		35°C 90% RH			—
					5 min., all CF-100			—

Note ⁽³⁾ Storage conditions shall be in sealed containers, 40°C, 168 hr. Other storage conditions shall be in accordance with agreements between parties concerned to delivery.

6. General Conditions of Testing

6.1 Standard Conditions

The standard conditions for testing shall be temperature 20°C class 2 (20±2°C) and humidity 65% class 5 (65±5% RH) as specified in JIS Z 8703 (Standard Atmospheric Conditions for Testing).

6.2 Testing Equipment and Apparatus

(1) Spatula

15 to 20 mm width and 1 to 2 mm thickness, and be made of stainless steel or of steel.

(2) Chemical balance

Reciprocal sensibility: 1 mg

(3) Dryer

Hot air circulation type, adjustable to set temperature ±2°C.

(4) Thermostatic water bath

Adjustable to set temperature ±2°.

(5) Tensile tester

Equipped with apparatus capable of recording automatically tensile load, with maximum testing load in the range of 15 to 85% of the capacity, and with tensile rate being changeable.

(6) Stopwatch

Minimum measuring unit of 0.2 seconds or less.

(7) Thermo-hygrostat bath

Maintainable to set temperature ±2°C, and adjustable to humidity 98% RH or over.

(8) Weather-o-meter (Sunshine)

As specified in JIS D 0205 (General Rules of Weatherability for Automobile Parts).

(9) Low temperature thermostatic bath

Adjustable to set temperature ±2°C.

7. Sample and Test Piece

7.1 Sampling Method

7.1.1 One part Urethane Adhesive

Sample, in amount necessary for testing, which is extracted from the product into an airtight container (Example: aluminium cartridge) shall be used for testing.

7.1.2 Primer

Sample, in amount necessary for testing, which is extracted from the product into an airtight container (Example: sealed glass bin) shall be used for testing after homogenizing by sufficient shaking.

7.2 Conditioning of Sample and Test Piece Material

Samples and test materials shall be used for testing after they are, in advance, left under specified conditions for sufficient time.

7.3 Types and Surface Treatment of Test Piece Materials

Types and surface treatment of test piece materials shall be in accordance with **Table 6**, provided that they shall be in accordance with agreements between the parties concerned to delivery if especially specified.

7.4 Conditioning of Clean Surface of Test Piece Material

The coated steel plate and glass plate shall be conditioned by cleaning with solvent ⁽⁶⁾ or by wiping with cloth into which solvent soaks. The spacer, polyethylene film and mould release paper shall be conditioned by wiping with dry cloth.

Note ⁽⁶⁾ Solvent to be used shall be as specified in JIS K 1522 (Isopropyl Alcohol)

Table 6

Test Piece Material		Material	Surface treatment
Coated steel plate	Steel plate	Cold rolled carbon steel sheet, 0.8 to 1.6 mm thick of class 1 as specified in JIS G 3141 (Cold Rolled Carbon Steel Sheets and Strips)	Base metal surface: Alkaline cleaning and zinc phosphate treatment shall be applied. ⁽⁴⁾ Under coat surface: Electric migration coating shall be applied. ⁽⁴⁾
	Paint	Standard top coat for finish paint Solid – white series Oil-free – polyester/melamine series Metallic – silver series acryl/melamine series	Intermediate coat surface: Intermediate coating shall be applied. ⁽⁴⁾ Top coat surface: Standard top coat shall be applied. ⁽⁴⁾
Glass plate		Float plate glass, 5 mm thick, as specified in JIS R 3202 (Float and Polished Plate Glasses)	Without masking: Ordinary float glass plate With masking: Surface treatment shall be carried out with black ceramics and so forth. ⁽⁵⁾
Spacer		Polyethylene Fluororesin Silicone resin Wood	Mould release treatment surface: Mould release treatment with pressure sensitive adhesive tape for separating.
Polyethylene film		0.15 to 0.20 mm thick	—
Release paper		Surface lubricative, treated with silicone oil or paraffin wax.	—

Notes ⁽⁴⁾ Type of coating and surface treatment conditions shall be in accordance with agreements between the parties concerned to delivery.

⁽⁵⁾ Surface treatment test pieces shall, in general, be of visible ray transmittance of 0.3% or less, and concrete method of which shall be in accordance with agreements between parties concerned to delivery.

Table 7

Test item		Unit	Test result	Number of test piece	Test method Item No.
Performance of adhesive	Appearance	—	—	1	9.1
	Specific gravity Method A	—	Down to two places of decimals	3	9.2
	Specific gravity Method B	—	Down to two places of decimals	3	9.3
	Nonvolatile matter	%	Down to one place of decimals	3	9.4
	Viscosity	Pa.{P}	Significant figure, 2 figures	1	9.5
	Sagging characteristics Method A	mm	Integer	3	9.6
	Sagging characteristics Method B	mm	Integer	3	9.7
	Curing rate Method A	min.	Integer	3	9.8
	Curing rate Method B	N{kgf}	Down to one place of decimals	3	9.9
	Curing rate Method C	MPa {kgf/cm ² }	Down to one place of decimals	3	9.10
	Pot life	—	—	3	9.11
	Coating film contamination properties	—	—	3	9.12
	Hardness	—	Integer	1	9.13
	Tensile strength	Mpa {kgf/cm ² }	Down to one place of decimals	3	
	Elongation	%	Significant figure, 2 figures	3	
	Ozone resistance	—	—	3	9.14
	Corrosion resistance	—	—	3	9.15
Performance of primer	Appearance	—	—	1	9.16
	Specific gravity	—	Down to two places of decimals	1	9.17
	Viscosity	s	Down to one place of decimals	3	9.18
	Nonvolatile matter	%	Down to one place of decimals	3	9.19
	Coating uniformity	—	—	1	9.20
	Coating film contamination	—	—	3	9.21
	Pot life	—	—	3	9.22
Adhesion performance	Shear strength	MPa {kgf/cm ² }	Down to one place of decimals	3	9.23
	Peel strength	—	—	3	9.24
Storage stability	Appearance	—	—	1	9.25
	Viscosity change ratio	%	Integer	Adhesive 1 Primer 3	
	Curing rate	min.	Integer	3	
	Hardness	—	Integer	1	
	Tensile strength	MPa {kgf/cm ² }	Down to one place of decimals	3	
	Elongation	%	Significant figure, 2 figures	3	
	Peel strength	—	—	3	
	Pot life	—	—	3	

8. Record of Test Results

Record of test results shall be as follows, provided that additional remarks, if any, shall be specified in each test item.

8.1 Number of Test Piece and Rounding off of Numerical Values

Number of test piece shall be in accordance with **Table 7**. Test results shall be rounded off in accordance with **JIS Z 8401** (Rules for Rounding off of Numerical Values), and be

indicated in the number of digit as given in **Table 7**.

8.2 Record of Condition of Failure

On adhesive shear strength, peel strength and pot life, the condition of failure shall be indicated in notation as described in **Table 8**, and area ratio (%) for each indicated notation shall also be given.

(Example: A1-30, CF-70; Area ratio of A1 30%, Area ratio of CF 70%)

Table 8

Division	Notation	Condition of peeling and failure	Naming
Peeling from adhesion surface	A1	Primer for coating surface does not remain on coated surface, and is peeled off.	Surface peeling
	A2	Primer for coating remains on coated surface, but is peeled off.	
	A3	Primer for glass does not remain on glass surface, and is peeled off.	
	A4	Primer for glass remains on glass surface, but is peeled off.	
Failure of test piece material	B1	Glass fails	Material failure
	B2	Coating is peeled off.	
Failure of adhesive layer	CF	Although adhesive adheres to coated surface and glass surface and remains on both, failure occurs.	Cohesive failure

9. Test Method

9.1 Appearance Test

This test shall be carried out, in order to examine existence of foreign substance, gelation and so forth in the adhesive.

9.1.1 Conditioning and Operation of Test Piece

20 to 30 g of sample shall be coated uniformly and thinly with a spatula on clean surface of glass plate, (100mm x 300mm) and the surface condition shall be observed.

9.2 Specific Gravity Test, Method A

This test shall be carried out, in order to measure the specific gravity of the adhesive with an aluminium container for specific gravity measurement.

9.2.1 Test Apparatus

(1) Aluminium container for specific gravity measurement

Made of aluminium plate, as shown in **Fig. 2**.

(2) Beaker

Such as to contain the aluminium container for specific gravity measurement.

9.2.2 Conditioning of Test Piece

Sample coated, taking care for air not to enter the groove(s) of the aluminium container for specific gravity measurement, shall be used for the test.

9.2.3 Operation

Test piece mass in air shall be measured with a chemical balance. Next, as soon as the test piece is taken out quickly after dipping into methanol ⁽⁷⁾, its apparent mass in distilled water of $20 \pm 1^\circ\text{C}$ shall be measured. Specific gravity D shall be calculated with the following formula:

$$D = \frac{(m_4 - m_2)}{(m_4 - m_2) - (m_3 - m_1)}$$

Where, m_1 : apparent mass of aluminium container for specific gravity measurement (g) in water

m_2 : mass of aluminium container for specific gravity measurement (g) in air

m_3 : apparent mass of test piece (aluminium container for specific gravity measurement and sample) (g) in water

m_4 : mass of test piece (aluminium container for specific gravity measurement and sample) (g) in air

Remarks: Mass of the aluminium container for specific gravity measurement in air and apparent mass in water shall be measured in advance.

Note (7) Grade 1 of JIS K 8891 [Methanol (Methyl Alcohol) (reagent)] is used.

9.3 Specific Gravity Test, Method B

This test shall be carried out with a hemispherical cup in order to measure specific gravity of the adhesive.

9.3.1 Test apparatus

Hemispherical cup, made of glass, as shown in Fig. 3 shall be used.

9.3.2 Operation and Record

Mass of glass plate (1) and that of hemispherical cup (2) shall be measured in advance. The adhesive shall be filled into the hemispherical cup (2) with such care that no bubble enter, and hemispherical cup is covered up with glass plate (1), with excessive adhesive being wiped off, and mass measurement shall be carried out.

Next, the hemispherical cup, which the adhesive is taken out and cleaned sufficiently, shall be filled with water of $20 \pm 1^\circ\text{C}$, and mass measurement shall be carried out in the same way. The specific gravity D shall be calculated with the following formula:

$$D = \frac{W_2 - W_1}{W_3 - W_1}$$

Fig. 2

