

**FED. TEST METHOD STD. NO. 175B**

**September 1, 1983**

**SUPERSEDING**

**Fed. Test Method Std. No. 175A**

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# **FEDERAL TEST METHOD STANDARD**

## **ADHESIVES: METHODS OF TESTING**

**This standard was approved by  
the Assistant Administrator**



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**Office of Federal Supply and Services  
General Services Administration**

**FSC 8040**

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## INFORMATION SHEET

ON

## FEDERAL TEST METHOD STANDARDS

This Federal Test Method Standard is issued in loose-leaf form to permit the insertion or removal of new or revised sections and test methods.

All users of Federal Test Method Standards should keep them up to date by inserting revised or new sections and test methods as issued and removing superseded and cancelled pages.

New and revised material and cancellations will be issued under Change Notices which will be numbered consecutively and will bear the date of issuance. Change Notices should be retained and filed in front of the Part I or II Index of the Standard as applicable until such time as they are superseded by a reissue of the entire Standard.



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ALPHABETICAL INDEX OF TEST METHODS OF THIS STANDARD WITH SUPERSEDED METHODS

Title	Fed. Test Method Std. No. 175		
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Adhesive for Brake Lining and Other Friction Materials			SAE J 840B
Amylaceous Matter in Adhesives			D 1488
Applied Weight per Unit Area of Dried Adhesive Solids	3011		D 898
Applied Weight per Unit Area of Liquid Adhesive	3012		D 899
Ash Content of Adhesives	4032.1		
Blocking Point of Potentially Adhesive Layers	2041		D 1146
Cleavage Strength of Metal-to-Metal Adhesives	1071.T		D 1062
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Conducting Creep Tests of Metal-to-Metal Adhesives, Recommended Practice for			D 1760
Consistency of Adhesives			D 1084
Density of Adhesives in Fluid Form			D 1875
Determining Durability of Adhesive Joints Stressed in Shear by Tension Loading			D 2919
Determining the Effect of Moisture and Temperature on Adhesive Bonds	2052.T		D 1151
Effect of Moisture and Temperature on Adhesive Bonds			D 1151
Fatigue Strength of Adhesives			D 3166
Filler Content of Phenol, Resorcinol, and Melamine Adhesives			D 1579
Flexibility of Adhesives	1081		
Grit or Lumps (or Undissolved Matter) in Adhesives	4041.1		
Hydrogen Ion Concentration of Dry Adhesive Films			D 1583
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Impact Strength of Adhesive Bonds			D 950
Impact Value of Adhesives	1051.1T		D 950
Nonvolatile Content of Aqueous Adhesives			D 1489
Odor Test for Adhesives	4051.1		

\* All ASTM Methods with the exception of one SAE (Society of Automotives Engineers) Method.

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Peel or Stripping Strength of Adhesives	1041.1		D 903
Peel Strength of Adhesives (Climbing Drum Apparatus)	1042.T		D 1781
Peel Resistance of Adhesives (T-Peel Test)			D 1876
pH of Adhesives and Bonded Assemblies	4011		D 1583
Resistance of Adhesive Bonds to Chemical Reagents	2011.1		D 896
Resistance of Adhesive Bonds to Water (Wet Strength)	2031.1T		D 1151
Resistance of Adhesives for Wood to Cyclic Laboratory Aging Conditions	2051.T		D 1183
Resistance of Adhesives to Cyclic Laboratory Aging Conditions			D 1183
Rubber Cements			D 816
Shear Strength Properties of Adhesives by Compression Loading	1031		D 905
Shear Strength Properties of Adhesives by Flextural Loading	1021		D 1184
Shear Strength Properties of Adhesives by Tension Loading	1033.1T		D 1002
Shear Strength Properties of Adhesives Determined with Single-Lap Construction Loading	1033		D 1002
Shear Strength Properties of Adhesives in Plywood-Type Construction by Tension Loading	1032 1032.1T		D 906 D 906
Strength of Adhesive Bonds on Flexural Loading			D 1184
Strength of Properties of Adhesive Bonds in Shear by Compression Loading			D 905
Shear Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading			D 906
Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal)			D 1002
Tensile Properties of Adhesives	1011.1		D 897
Tensile Properties of Adhesive Bonds			D 897
Tensile Properties of Adhesives for Rubberlike Materials	1012		D 816
Total Solids Content			D 1582

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Ash Content of Adhesives	4032.1	--
Grit or Lumps (or Undissolved Matter) in Adhesives	4041.1	--
Odor Test for Adhesives	4051.1	--

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Rubber Cement	D 816	--
Resistance of Adhesive Bonds to Chemical Reagents	D 896	2011.1
Tensile Properties of Adhesives	D 897	1011.1
Tensile Properties of Adhesive Bonds	D 897	--
Applied Weight per Unit Area of Dried Adhesive Solids	D 898	3011
Applied Weight per Unit Area of Liquid Adhesive	D 899	3012
Peel or Stripping Strength of Adhesive Bonds	D 903	--
Peel or Stripping Strength of Adhesives	D 903	1041.1
Shear Strength Properties of Adhesives by Compression Loading	D 905	1031
Strength Properties of Adhesive Bonds in Shear by Compression Loading	D 905	--
Shear Strength Properties of Adhesives in Plywood-Type Construction by Tension Loading	D 906	1032
Strength Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading	D 906	1032.1T
Impact Strength of Adhesives	D 906	--
Impact Strength of Adhesive Bonds	D 950	1051
Impact Value of Adhesives	D 950	--
Shear Strength Properties of Adhesives by Tension Loading	D 950	1051.1T
Shear Strength Properties of Adhesives Determined with Single-Lap-Construction by Tension Loading	D 1002	1033.1T
Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal)	D 1002	1033
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Cleavage Strength of Metal-to-Metal Adhesive Bonds	D 1062	1061
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Blocking Point of Potentially Adhesive Layers	D 1084	--
Determining the Effect of Moisture and Temperature on Adhesive Bonds	D 1146	2041
	D 1151	2052.T

\* All ASTM Methods with the exception of one  
SAE (Society of Automotive Engineers) Method.

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PART II (Cont'd)

NON-GOVERNMENT METHODS

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Resistance of Adhesives for Wood to Cyclic Laboratory Aging Conditions	D 1183	2051.T
Resistance of Adhesives to Cyclic Laboratory Aging Conditions	D 1183	--
Shear Strength Properties of Adhesives by Flexural Loading	D 1184	1021
Strength of Adhesive Bonds on Flexural Loading	D 1184	--
Amylaceous Matter in Adhesives	D 1488	--
Nonvolatile Content of Aqueous Adhesives	D 1489	--
Total Solid Content	D 1582	--
Filler Content of Phenol, Resorcinol, and Melamine Adhesives	D 1579	--
pH of Adhesives and Bonded Assemblies	D 1583	4011
Hydrogen Ion Concentration of Dry Adhesive Films	D 1583	--
Conduction Creep Tests of Metal-to-Metal Adhesives, Recommended Practice for	D 1780	--
Climbing Drum Peel Test for Adhesives	D 1781	--
Peel Strength of Adhesives (Climbing Drum Apparatus)	D 1781	1042.T
Density of Adhesives in Fluid Form	D 1875	--
Peel Resistance of Adhesives (T-Peel Test)	D 1876	--
Determining Durability of Adhesive Joints Stressed in Shear by Tension Loading	D 2919	--
Adhesives for Brake Lining and Other Friction Materials	SAE J 840B	--

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## FEDERAL TEST METHOD STANDARD

### ADHESIVES: METHODS OF TESTING

Authority. This standard is issued pursuant to the Federal Property and Administrative Services Act of 1949, as amended and its application to the purchase of commodities referred to herein is mandatory on all Federal agencies.

#### 1. SCOPE AND NUMBERING SYSTEM

1.1 Scope. This standard consists of two parts. Part I contains test methods on adhesives that are common to Government specifications and that are not covered by test methods of the non-government organizations. Part II contains non-government test methods on adhesives that are common to Government specifications. Neither part contains test methods applicable only to a specific product; such test methods are included in the material specification. In case of conflict between the provisions of this standard and those of a material specification for a particular material or product, the provisions of the latter shall take precedence.

1.2 Numbering system. The various test methods of this standard are designated by numbers for Federal or Part I test methods (see 1.2.1), and by other designations such as ASTM, SAE, etc., for Part II test methods (see 1.2.2).

1.2.1 Federal (part I) class of tests. Any given class of tests is assigned a series of basic numbers. The numbers assigned are as follows:

<u>Numbers assigned</u>	<u>Class of tests</u>
1000 to 1999	Strength properties
2000 to 2999	Permanence properties
3000 to 3999	Working properties
4000 to 4999	Analytical tests

1.2.1.1 Federal (part I) revision procedure. The number of times any given test method has been revised is indicated by the number following the decimal point. For example, method number 1061.1 has been revised once. When a revision of a Federal (part I) test method of this standard is issued, it automatically supersedes any previous number referenced in product specifications. The particular revision in force at the time of invitation for bids shall be the method used for testing.

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1.2.2 Non-Government (part II) designations. Designations of part II test methods are the same as those of the Government accepted methods of non-government standards. When a non-government method now in the standard has been revised, the revision will be coordinated; and when it has been formally accepted by the Government, a change notice to this standard will be issued, and the new method will then supersede the existing method. When any non-government method on adhesives is found to be needed in two or more Government specifications, it will be considered for coordination and addition to this standard. Non-government methods on adhesives, that are not included in this standard, however, may be referenced in Government documents, provided the review activities have concurred in the method.

1.2.2.1 Referencing of non-government methods. Part II of this standard should not be referenced alone in Government documents as a means of designating non-government test methods. To avoid confusion, the designation and title of non-government methods should be specified with the non-government name and address where required. When a non-government method is referenced, only that version of the year of issue that has been formally accepted should be used.

## 2. GENERAL REQUIREMENTS

### 2.1 Samples.

2.1.1 Number of samples. The number of samples to be taken from each lot shall be as specified in the material specification. The number of specimens to be tested in each method of test shall be as specified in the material specification; if not so specified, the number of specimens specified in the method of test shall be used.

2.1.2 Taking samples. Samples shall be obtained, if possible, from the products to be tested, taken at random, and in such case shall be taken in accordance with the requirements of the specification covering the particular material. In case it is not practical to obtain suitable test specimens from the finished article, the manufacturer shall furnish bonded test specimens suitable for the tests specified. The manufacturer shall furnish an affidavit that the material submitted for test is made from the same materials, under the same or equivalent conditions, as those used in the manufacture of the commodity.

2.1.3 Preparation of liquid samples for test. Before using the adhesive to be tested, it shall be thoroughly agitated to uniform consistency throughout, after which a sample shall be taken of sufficient quantity for performance of the tests required. The sample shall be kept in a tightly closed container to prevent evaporation and shall be agitated or stirred to uniform consistency immediately before test or use in preparation of the test specimens.

### 2.2 Preparation of test specimens.

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2.2.1 Bonding. The accuracy of the results of strength tests of adhesive bond will depend upon the conditions under which the bonding process is carried out. Unless otherwise specified in the material specification, the bonding conditions shall be prescribed by the manufacturer of the adhesive. To insure that complete information is available to the individual conducting the tests, the manufacturer of the adhesive shall furnish numerical values and other specific information for each of the following variables:

- (1) Procedure for preparation of surface prior to application of the adhesive, including the moisture content of wood, the cleaning and drying of metal surfaces, and special surface treatments such as sanding which are not specifically limited by the pertinent test method.
- (2) Complete mixing directions for the adhesive.
- (3) Conditions for application of the adhesive including the rate of spread or thickness of film, number of coats to be applied, whether to be applied to one or both surfaces, and the conditions of drying where more than one coat is required.
- (4) Assembly conditions before application of pressure, including the room temperature, and open and closed assembly time.
- (5) Curing conditions, including the amount of pressure to be applied, the length of time under pressure, and the temperature of the assembly when under pressure. It should be stated whether this temperature is that of the adhesive layer or of the atmosphere to which the assembly is to be maintained.
- (6) Conditioning procedure before testing, including the length of time, temperature, and relative humidity.

A range may be prescribed for any variable by the manufacturer of the adhesive, if it can be assumed by the test operator that any arbitrarily chosen value within such a range or any combination of such values for several variables will be acceptable to the manufacturer and the purchaser of the adhesive.

2.2.2 Machining. When it is necessary to machine specimens, cutting tools shall be used in such a manner that the possibility of overheating is minimized. Unless otherwise specified herein, all machined surfaces shall be finished with 3/0 emery paper to eliminate all irregularities such as toolmarks, etc. Where a more perfect finish is desired, a suitable polishing compound shall be used.

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2.2.3 Treatment prior to test. Samples and specimens for tests shall not be heated, immersed in water, or subjected to any mechanical or chemical treatment prior to test except as specifically described herein or in the material specification.

2.3 Testing conditions. Unless otherwise specified in detail method of test, the atmospheric conditions surrounding the specimen prior to and during test shall be  $23^{\circ} \pm 1.1^{\circ}\text{C}$  ( $73.4^{\circ} \pm 2^{\circ}\text{C}$ ) and  $50 \pm 4$  percent relative humidity in accordance with Fed. Std. No. 1, Standard for Laboratory Atmospheric Conditions For Testing; the conditioning period prior to test shall be 48 hours for specimens of 1/8 inch or less in thickness and 96 hours for thicker specimens.

2.3.1 Testing for temperature effects. When the relation of properties to temperature is to be determined, it is recommended, as a matter of standardization, that test be performed at one or more of the following atmospheric temperatures:  $-55^{\circ}\text{C}$  ( $-67^{\circ}\text{F}$ );  $-40^{\circ}\text{C}$  ( $-40^{\circ}\text{F}$ );  $-25^{\circ}\text{C}$  ( $-13^{\circ}\text{F}$ );  $0^{\circ}\text{C}$  ( $32^{\circ}\text{F}$ );  $23^{\circ}\text{C}$  ( $73.4^{\circ}\text{F}$ );  $35^{\circ}\text{C}$  ( $95^{\circ}\text{F}$ );  $50^{\circ}\text{C}$  ( $122^{\circ}\text{F}$ );  $60^{\circ}\text{C}$  ( $140^{\circ}\text{F}$ );  $70^{\circ}\text{C}$  ( $158^{\circ}\text{F}$ );  $77^{\circ}\text{C}$  ( $171^{\circ}\text{F}$ );  $100^{\circ}\text{C}$  ( $212^{\circ}\text{F}$ );  $149^{\circ}\text{C}$  ( $300^{\circ}\text{F}$ );  $204^{\circ}\text{C}$  ( $400^{\circ}\text{F}$ );  $260^{\circ}\text{C}$  ( $500^{\circ}\text{F}$ );  $316^{\circ}\text{C}$  ( $600^{\circ}\text{F}$ ). The tolerance for test temperature shall be as follows:

Range of specified temperatures	Tolerance plus or minus
$-70^{\circ}\text{C}$ ( $-94^{\circ}\text{F}$ ) and over, but under $23^{\circ}\text{C}$ ( $73.4^{\circ}\text{F}$ )	$2.0^{\circ}\text{C}$ ( $3.6^{\circ}\text{F}$ )
$23^{\circ}\text{C}$ ( $73.4^{\circ}\text{F}$ ) through $35^{\circ}\text{C}$ ( $95^{\circ}\text{F}$ )	$1.0^{\circ}\text{C}$ ( $1.8^{\circ}\text{F}$ )
Over $35^{\circ}$ ( $95^{\circ}\text{F}$ ) but under $200^{\circ}\text{C}$ ( $392^{\circ}\text{F}$ )	$2.0^{\circ}\text{C}$ ( $3.6^{\circ}\text{F}$ )
$200^{\circ}\text{C}$ ( $392^{\circ}\text{F}$ ) through $300^{\circ}\text{C}$ ( $572^{\circ}\text{F}$ )	$3.0^{\circ}\text{C}$ ( $5.4^{\circ}\text{F}$ )
Over $300^{\circ}\text{C}$ ( $572^{\circ}\text{F}$ ) but under $350^{\circ}\text{C}$ ( $662^{\circ}\text{F}$ )	$4.0^{\circ}\text{C}$ ( $7.2^{\circ}\text{F}$ )

2.3.2 Conditioning. Unless otherwise specified, specimens shall be conditioned at the specified testing temperature and humidity for at least 24 hours immediately prior to test. The temperature, the relative humidity, and the period of time for conditioning should all be recorded.

2.4 Apparatus. Unless otherwise specified, properties shall be determined in any standard type of testing machine properly calibrated and accurate to 1 percent in the range used.

2.5 Results of tests. Unless otherwise specified, the average of the results for the specimens tested shall be used to determine conformance of materials tested under this specification. Unless otherwise specified, results of specimens that break at some obvious flaw or that do not break between the pre-determined gage marks shall be discarded. Unless otherwise

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specified, results that deviate from the mean value of all tests shall be rejected if the deviation of the doubtful value is more than 5 times the average deviation from the mean obtained by excluding the doubtful value. Additional specimens shall be tested in place of any for which the results are discarded in accordance with these provisions.

2.6 Test reports. Unless otherwise specified, the report on each test shall include the following:

- (1) The name of the Government agency requesting the test.
- (2) The name of the supplier and the number and date of the contract covering the material and/or parts.
- (3) The title, number, and date of the applicable material specification.
- (4) Description of the material, including type, source, manufacturer's code numbers, etc.
- (5) Type and dimensions of specimens.
- (6) Location and direction of specimens in the original sample.
- (7) Temperature, humidity, and length of conditioning period.
- (8) Such additional data as are stated herein under the individual test methods.
- (9) Such additional data as may be required under the material specification.
- (10) Any further information that may be considered pertinent, particularly with reference to unexpected behavior.
- (11) A brief description of the testing apparatus, sufficient to identify it.

### 3. CHANGES

3.1 When a Federal agency considers that a Federal standard does not provide for its essential needs, written request for adding to or otherwise changing the standard, supported by adequate justification, shall be sent to the Administration. This justification shall explain wherein the standard does not provide the essential needs. The request should be sent in duplicate to the General Services Administration, Federal Supply Service, Standardization Division, Washington, D.C., 20407. The administration will determine the appropriate action to be taken and will notify the agency.

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#### 4. NOTES

4.1 Obtaining specifications and standards. Federal and Military specifications and standards may be obtained as outlined under General Provisions in the Index of Specifications and Standards. Federal Government activities may obtain the Index from established distribution points within their agencies. All others may purchase the Index with cumulated monthly supplements as issued, from the Superintendent of Documents, U.S. Government Printing Office, Washington D.C., 20402.

#### 4.2 Non-government methods.

4.2.1 ASTM methods. Application for copies of ASTM methods should be made to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA, 19103.

4.2.2 SAE methods. Application for copies of SAE test methods should be addressed to the Society of Automotive Engineering, Inc., 2 Pennsylvania Plaza, New York, NY, 10001.

Military agencies should make application for Part II of Fed. Test Method Std. No. 175B, containing the non-government test methods, to U.S. Naval Supply Depot (NSD 103), 5801 Tabor Ave., Philadelphia, PA 19120.

#### MILITARY INTERESTS:

#### PREPARING ACTIVITY:

##### Custodians

Army - MR

Army - MR

Navy - SH

Air Force - 11

Project No. 8040-0410

##### Review Activities

Army - GL, MI, ME, AV, PA

Navy - AS, SH

Air Force - 11, 69

##### User Activities

Army - AL

Navy - OS, SA, YD, MC

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FEDERAL TEST METHOD STANDARD

PART I

(FEDERAL METHODS)

ADHESIVES: METHODS OF TESTING



METHOD 1081  
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## FLEXIBILITY OF ADHESIVES

### 1. SCOPE

1.1 This method provides a means for determining the flexibility (elasticity) of single films or systems of films of adhesives.

### 2. TEST SPECIMENS

2.1 Test specimens shall be of the adhesive to be tested as specified in the material specification and of sufficient quantity for the preparation of the panels, as specified in 5.1.

### 3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, 5 specimens shall be tested from each sample unit.

### 4. APPARATUS

4.1 Panels. Unless otherwise specified, panels shall be not less than 2-3/4 by 5-7/8 inches and of steel not thinner than No. 30 nor thicker than No. 28 gage (approximate thickness of No. 30 gage is 0.0125 inch, of No. 28 gage 0.0156). The panels shall be capable of being bent smoothly and uniformly over a mandrel of the diameter specified in the materials specification and through an arc of 180°. The panels shall be free from oil and rust or other corrosion and one face of each panel shall be freshly sandblasted to a clean surface.

4.2 Mandrels. A series of smooth, straight steel rods ranging from 1/8 to 1 inch in diameter and held firmly by suitable supports.

4.3 Doctor blade. A doctor blade capable of casting films at least 2 inches wide and of the thickness and thickness tolerance specified in the material specification over the area of the film to be tested. When a suitable doctor blade is not available, the various conventional and automatic methods of spray, dip, flow, and brush application may be used provided the dry film thickness does not vary outside the thickness tolerance.

### 5. PROCEDURE

5.1 Apply to the clean surface of the test panel or coating system, a uniform coating of the adhesive to be tested at the required thickness, as specified in 4.3. Cure the adhesive in accordance with the instructions given in the materials specification, or if not given, in accordance with

## METHOD 1081

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recommendations by the manufacturer. Unless otherwise specified, condition the cured film at  $23^{\circ} \pm 1^{\circ}\text{C}$  ( $73.4^{\circ} \pm 1.8^{\circ}\text{F}$ ) and a relative humidity of  $50 \pm 4$  percent for at least 1/2 hour. While at this temperature, place the test panel with the adhesive side uppermost on a mandrel of the specified diameter at a point equally distant from the top and bottom edges of the panel. Bend the panel double ( $180^{\circ}$ ) or  $90^{\circ}$  as specified in the materials specification in approximately 1 second. Examine each specimen tested for cracks, flakes, and chips.

## 6. REPORT

6.1 The report shall include the data specified in paragraph 2.6 where applicable, and the following:

- (a) Material and dimensions of panels if other than that specified.
- (b) Size of mandrel.
- (c) Number of coats of adhesive applied.
- (d) Cure time for each coat, temperature, and relative humidity.
- (e) Thickness of adhesive film.
- (f) Whether panel is bent  $180^{\circ}$  or  $90^{\circ}$ .
- (g) Observation as to cracks, flakes, or chips in adhesive after bending of panel for each individual test.

METHOD 4032.1  
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## ASH CONTENT OF ADHESIVES

### 1. SCOPE

1.1 These methods cover procedures used in testing the ash content of adhesives.

1.2 Two methods are available: method A is used for a glue or starch adhesive wherein there is no danger from the nonvolatile content forming a rubbery residue when ignited; method B employs nitric acid to avoid the nonvolatile residue being transformed into a viscous foam when ignited.

Note 1 - The nitric acid treatment of method B makes for a faster, smoother oxidation of the organic matter. If a rubbery residue were merely to be heated in a muffle furnace, it might be transformed into a viscous foam. The gaseous decomposition products might cause the mass to expand, possibly overflowing the crucible. The outer skin might harden and the gases then might break through it causing loss of material by spattering. With the nitric acid treatment, most of the decomposition is done quickly at a relatively low temperature so that only the residual carbon need be burned off in the muffle furnace.

1.2.1 The methods appear in the following sections:

	Section
Procedure A	4
Procedure B	5

1.3 These standard methods of testing are not applicable to adhesives containing decomposable salts such as zinc chloride.

### 2. APPARATUS AND REAGENTS

#### 2.1 Apparatus

2.1.1 Crucibles - Crucibles, with tightly fitting lids, having a capacity of 30 ml or more shall be used. Platinum crucibles are preferred, but silica or porcelain crucibles may be used.

2.1.2 Evaporating dishes - Evaporating dishes having a capacity of 150 ml shall be used. Platinum is preferred, but silica or porcelain evaporating dishes may be used.

2.1.2.1 Watchglasses to cover evaporating dishes.

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2.1.3 Desiccator equipped with drying agent and tray.

2.1.4 Analytical balance sensitive to 1 mg.

2.1.5 Steam bath.

2.1.6 Drying oven - Drying oven, with temperature control, for maintaining temperature at 100 to 105°C (212 to 221°F).

2.1.7 Hotplate, electric.

2.1.8 Muffle furnace - An electric furnace is recommended for igniting crucibles and test specimens. An indicating pyrometer is preferable for maintaining the desired temperature.

## 2.2 Reagents

2.2.1 Nitric acid, concentrated ( $\text{HNO}_3$ ), sp. gr. 1.42.

## 3. TEST SPECIMEN

3.1 The test specimens shall consist of 5 to 6 grams of the material to be tested.

3.2 Care shall be taken to ensure that sampling is representative of the entire lot of material being tested. Two (2) specimens shall be tested from each sample unit.

## ASH

### 4. Procedure A

4.1 Ignite the empty crucible and cover over a burner or in a muffle furnace at 600°C (1112°F), cool in a desiccator, and weigh the covered crucible to the nearest milligram. Place a  $5.0 \pm 0.5$  g test specimen weighed to the nearest mg into a previously weighed crucible and with cover removed evaporate to dryness on a steam bath.

4.1.1 If the ash content is based on the total sample weight continue with section 4.2 below.

4.1.2 If the ash content is based on the nonvolatile content, then follow section 5.2 and 5.3 of procedure B before continuing with section 4.2 below.

Note 2 - Nonvolatile content: for procedure A, the percentage of ash is usually based on the total weight of the sample. If the nonvolatile content is required, however, section 5.2 and 5.3 of procedure B should be followed and then continue with the following section below.

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4.2 The crucible and its contents, with cover removed, shall be placed in the muffle furnace. Heat slowly at the start to avoid flaming and protect the crucible from strong drafts at all times to avoid mechanical loss of test specimen. Gradually increase heat until the temperature of the muffle furnace reaches  $575 \pm 25^{\circ}\text{C}$  ( $1067 \pm 45^{\circ}\text{F}$ ). Hold the ignition temperature, at 550 to  $600^{\circ}\text{C}$  ( $1022$  to  $1112^{\circ}\text{F}$ ), and allow the specimen to remain in the muffle furnace for 8 hours or until constant weight is attained. Cool in a desiccator and reweigh the covered crucible and its contents to the nearest mg.

#### 4.3 Calculations and precision of reported results.

4.3.1 Calculate the ash content, based on the total weight of the specimen as follows:

$$\text{Ash content, percent} = (W_1/W_2) \times 100$$

where:

$W_1$  = weight of ash, and  
 $W_2$  = weight of test specimen

4.3.2 Results shall be reported to two decimal places and the duplicate determinations should agree within 0.05 percent.

#### 5. PROCEDURE B

5.1 Ignite, cool in a desiccator, and weigh the evaporating dish to the nearest milligram. Place into the evaporating dish approximately 5 to 6 grams of the test specimen weighed to the nearest milligram. Place the evaporating dish and contents on a steam bath until the solvent has been evaporated.

Note 2: Caution - Care should be taken to remove as much of the solvent as is practicable by this method in order to eliminate any fire hazard which might otherwise exist when subjecting the material to the subsequent drying operation.

5.2 Place the evaporating dish and its contents in the oven at 100 to  $105^{\circ}\text{C}$  ( $212$  to  $221^{\circ}\text{F}$ ) for 5 hours, cool in the desiccator, and reweigh to the nearest milligram.

5.3 Nonvolatile content - When required, the weight of the nonvolatile content in the specimen shall be calculated as follows:

$$\text{Nonvolatile content, percent} = (w/W) \times 100$$

where:

$w$  = weight of specimen after oven heating  
 $W$  = weight of original weight of sample

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5.4 Add a 25 ml portion of the nitric acid to the evaporating dish containing the dried material. Cover the evaporating dish with a watchglass to prevent loss by spattering, and heat on the open steam bath until the vigorous reaction which first ensues has subsided. The addition of 25 ml of the nitric acid, and the heating on the steam bath shall be repeated until no further action occurs. Remove the watchglass and continue to heat on the open steam bath until all excess nitric acid has been removed.

5.5 Heat the evaporating dish and contents by placing on an electric hotplate at medium heat, or over a small flame. Heating may also be done with infrared lamps or by placing the evaporating dish in a cold furnace and raising the temperature at a rate slow enough to prevent spattering. Heat until all volatile matter has been driven off and the contents of the evaporating dish has been charred. The evaporating dish and contents then shall be ignited to constant weight in the muffle furnace. The temperature of the muffle furnace shall be held at  $600 \pm 25^{\circ}\text{C}$  ( $1112 \pm 45^{\circ}\text{F}$ ).

5.6 Calculation and precision of reported results.

5.6.1 Calculate the ash content of the nonvolatile matter, based on the weight of the nonvolatile matter in the sample as follows:

$$\text{Ash content of nonvolatile matter, percent} = (W_1/w) \times 100$$

where:

$W_1$  = weight of ash

$w$  = weight of nonvolatile matter in sample

5.6.2 Results shall be reported to the second decimal place and the duplicate determinations should agree within 0.05 percent.

Note 3 - If required, the ash content of the total weight of the specimen shall be calculated as specified in section 4.3.1.

## 6. REPORT

6.1 The report shall (a) include pertinent contract requirements, (b) indicate whether test will be determined by the test method according to procedure A or B, (c) indicate percentage of ash and whether the percentage of ash is based on the original weight of sample or the weight of the nonvolatile content and (d) include the temperature and length of time specimen is in the muffle furnace.



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## GRIT, LUMPS, OR UNDISSOLVED MATTER IN ADHESIVES

### 1. SCOPE.

1.1 This test covers the detection of grit, lumps, or undissolved foreign matter in liquid, water-based adhesives such as starch, dextrin, casein, latex, or resin base. It is intended to replace Federal Standard 175, Methods of Testing Adhesives, Method 4041.

### 2. APPLICABLE DOCUMENTS

#### 2.1 ASTM Standards

D 907, Standard Definitions of Terms Relating to Adhesives

D 3460, Specification for White Watermarked Bond and Unwatermarked Bond, Mimeograph, Duplicator, and Xerographic Cut-Sized Office Papers.

### 3. SUMMARY OF METHOD

3.1 A sheet of white bond paper is coated with the adhesive, placed adhesive-side down onto a glass plate, and rolled flat. The presence of grit, lumps, or undissolved matter is detected by finger contact with these discontinuities which may be felt through the paper.

### 4. SIGNIFICANCE AND USE

4.1 The test detects the presence of gross particulates in the test specimen. It is useful for the control of uniformity between lots.

### 5. TERMINOLOGY

5.1 Definitions of terms in this standard may be found in Definitions D 907.

### 6. APPARATUS

6.1 White bond paper - Sufficient quantity of white bond paper conforming to Grade No. 1, Type I of D 3460, in minimum size 8 1/2 in x 11 in (21.6 x 28.0 mm).

6.2 Glass plate - Clean plate glass, minimum size to fit the paper selected.

6.3 Roller - A rubber-covered steel roller approximately  $3.25 \pm 0.1$  in ( $82.5 \pm 2.5$  mm) diameter, and  $1.75 \pm 0.05$  in ( $44.4 \pm 1.1$  mm) wide. The rubber covering shall be approximately 0.25 in (6 mm) thick, having a Shore scale A durometer hardness of 75 to 85. The surface of the rubber covered roller shall be a true cylinder void of concave or convex deviations. The mass of the roller shall be  $4.5 \pm 0.1$  lb ( $2041 \pm 45$ g).

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## 7. SAMPLING, TEST SPECIMENS

7.1 Obtain a one-pint (500 ml) sample of adhesive representative of the lot to be tested.

7.2 Prepare a mixture of the adhesive sample in the proportions recommended by the manufacturer. In the case of ready-to-use adhesive, stir the contents of the container to achieve uniformity.

7.3 Weigh on to the test paper  $10 \pm 0.5\text{g}$  of the adhesive and spread over the surface of the paper in a thin layer or coat.

Note 1 - different weights of adhesive may be used, and a different number of specimens may be prepared if specified.

7.4 Prepare and test 3 specimens from each sample of adhesive.

## 8. PROCEDURE

8.1 While the applied adhesive is wet, lay the coated paper face down on the glass, and roll it with the roller just sufficient to eliminate air bubbles and wrinkles.

8.2 Using light pressure of the fingers, go over the sheet carefully to detect any unevenness caused by grit, lumps or undissolved matter. Estimate the number, size and range of such discontinuities.

## 9. REPORT

Include in the report the following information:

9.1 Complete identification of the adhesive tested.

9.2 Weight of adhesive in each specimen.

9.3 Any variations from standard directions, as in Note 1.

9.4 Record of grit, lumps, or undissolved matter detected in 8.2.

## 10. PRECISION AND ACCURACY

10.1 Because numerical values are not established in this method, no statement of precision or accuracy is provided.

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## ODOR TEST FOR ADHESIVES

This Standard is issued under the fixed designation ....., the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

### 1. SCOPE

1.1 This recommended practice describes a procedure for evaluating the odor of adhesives.

1.2 This recommended practice provides a means of comparing the odor of an adhesive sample to a reference material. It is not intended to give an absolute value for the odor of a sample.

### 2. SIGNIFICANCE

2.1 The results from this determination may be useful in determining the suitability of adhesives in applications where the odor level associated with the end use is critical.

### 3. APPARATUS

3.1 Wide mouth glass bottles with screw caps.

Note: It is important that the bottles and caps be kept clean and odor free. Any bottle which cannot be made visibly clean or odor free should be discarded. Bottles and caps should be washed with a suitable cleaning powder, rinsed thoroughly with tap water, rinsed with distilled water, drained and dried in an oven. All cleaning should be done in a relatively odor free atmosphere.

3.2 Oven or incubator for aging samples at elevated temperatures.

3.3 Aluminum foil.

3.4 Balance capable of measuring to nearest 0.1g.

### 4. TEST SPECIMENS AND SAMPLE

4.1 The test specimen should be a representative sample of the adhesive being tested.

4.2 Place  $20.0 \pm 0.1$  grams of the sample into the wide mouth glass bottle, place aluminum foil over the top, and then place the cap on the bottle and tighten.

Note: Always use new aluminum foil, never reuse it.

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## 5. CONDITIONING

5.1 The bottles containing the adhesive samples should be aged for at least 24 hours at  $23 \pm 1^{\circ}\text{C}$  and  $50 \pm 5\%$  relative humidity prior to testing.

5.1.1 If the effect of temperature aging on the odor of the adhesive is desired, place the bottles containing the adhesive samples in an oven at the desired aging temperature and condition for 2 hours (longer conditioning times may be used, if desired).

Note: Do not tightly close the caps on the bottles before heating or before the bottles have cooled. Either of these could cause the glass bottles to rupture.

5.1.2 Samples aged at elevated temperatures should be evaluated within a two-hour period after reaching room temperature.

## 6. REAGENTS AND MATERIALS

6.1 A reference material which has an acceptable odor should be selected from prior experience or knowledge.

6.2 The reference material should be stored in a tightly closed container and in a relatively odor free atmosphere.

6.3 Care should be taken against contamination of the reference material. If it is suspected that the reference material has been contaminated, then it should be discarded and a new reference material obtained.

## 7. PROCEDURE

7.1 The odor testing should be conducted by an odor panel consisting of at least five people. It is preferable that panel members be nonsmokers. Panel members should not smoke or eat for approximately one hour prior to evaluating samples. Panel members may find that the sense of smell is sharpened if samples are rated with the eyes closed. A person should not be a panel member if he has a cold or has been working with chemicals with strong odors.

7.2 The odor testing should be performed in a room as free of odors as possible. All samples for one panel members should be evaluated in the same room at the same time. If possible, all panel members should use the same room.

7.3 Pair each adhesive sample to be tested with a sample of the reference material that has undergone identical conditioning.

7.4 Each panel member should open the bottles and sniff the odor. The bottles should be kept closed except for the short period necessary to sniff the sample.

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Note: Odor fatigue can occur if too many samples are evaluated at one time or if too many rechecks are required to reach a decision on the same sample. If this occurs, allow a few minutes for the nose to regain its former sensitivity.

7.5 For each pair of samples, the panel members should indicate whether the odor of the adhesive sample is less than, equal to, or greater than the reference material.

## 8. INTERPRETATION OF RESULTS

8.1 The odor testing results from the five panel members should be compiled and the odor level receiving the majority of the votes should be reported as the odor level for the sample.

8.2 If no odor level receives a majority, then the test should be repeated on a different sample of the adhesive. If after a second testing no majority is obtained, then the sample should be rated as having odor equal to the reference material.

## 9. REPORT

9.1 The report should include the following:

9.1.1 Sample identification,

9.1.2 Conditioning procedure for sample,

9.1.3 Reference material used,

9.1.4 Odor of samples as less than, equal to, or greater than the reference material,

9.1.5 Date of evaluation.



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FEDERAL TEST METHOD STANDARD

PART II

(ASTM METHODS)

ADHESIVES: METHODS OF TESTING

FED. TEST METHOD STD. NO. 175B





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# NUMERICAL INDEX OF NON-GOVERNMENT TEST METHODS

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SAE J 840B	Adhesives for Brake Lining and Other Friction Materials

\* All ASTM Methods with the exception of one  
 SAE (Society of Automotive Engineers) Method.



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FTMS No. 175B**2. DOCUMENT TITLE**  
ADHESIVES: METHODS OF TESTING**3a. NAME OF SUBMITTING ORGANIZATION****b. ADDRESS** *(Street, City, State, ZIP Code)***4. TYPE OF ORGANIZATION** *(Mark one)*☐

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