

Designation: D 3427 - 03

# Standard Test Method for Air Release Properties of Petroleum Oils<sup>1</sup>

This standard is issued under the fixed designation D 3427; 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. A superscript epsilon  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 This test method covers the ability of turbine, hydraulic, and gear oils to separate entrained air.

Note 1—This test method was developed for mineral based oils. It may be used for some synthetic fluids; however, the precision statement applies only to petroleum oils.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

## 2. Referenced Documents

2.1 ASTM Standards: <sup>2</sup>

D 1193 Specification for Reagent Water

D 1401 Test Method for Water Separability of Petroleum Oils and Synthetic Fluids

E 1 Specification for ASTM Thermometers

2.2 DIN Standard:<sup>3</sup>

DIN 51 381

# 3. Terminology

- 3.1 Definitions of Terms Specific to This Standard:
- 3.1.1 *air release time*, *n*—the number of minutes needed for air entrained in the oil to reduce in volume to 0.2 % under the conditions of this test and at the specified temperature.

## 4. Summary of Test Method

4.1 Compressed air is blown through the test oil, which has been heated to a temperature of 25, 50, or 75°C. After the air flow is stopped, the time required for the air entrained in the oil to reduce in volume to 0.2 % is recorded as the air release time.

NOTE 2—By agreement between the customer and the laboratory, the oil may be heated at other temperatures. However, the precision at these different temperatures is not known at present.

## 5. Significance and Use

- 5.1 Agitation of lubricating oil with air in equipment, such as bearings, couplings, gears, pumps, and oil return lines, may produce a dispersion of finely divided air bubbles in the oil. If the residence time in the reservoir is too short to allow the air bubbles to rise to the oil surface, a mixture of air and oil will circulate through the lubricating oil system. This may result in an inability to maintain oil pressure (particularly with centrifugal pumps), incomplete oil films in bearings and gears, and poor hydraulic system performance or failure.
- 5.2 This test method measures the time for the entrained air content to fall to the relatively low value of 0.2 % volume under a standardized set of test conditions and hence permits the comparison of the ability of oils to separate entrained air under conditions where a separation time is available. The significance of this test method has not been fully established. However, entrained air can cause sponginess and lack of sensitivity of the control of turbine and hydraulic systems. This test may not be suitable for ranking oils in applications where residence times are short and gas contents are high.

### 6. Apparatus

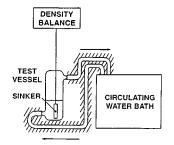
- 6.1 A schematic diagram of the apparatus is shown in Fig. 1. The component parts are described as follows:
- 6.1.1 *Test Vessel*, made of borosilicate glass as shown in Fig. 2, consisting of a jacketed sample tube fitted with an air inlet capillary, baffle plate, and air outlet tube. The two parts of each test vessel should be marked and preferably used as a pair. Interchanged parts may be used so long as the resultant test vessel conforms to the stated dimensions.
- 6.1.2 *Pressure Gage*, covering the range from 0 to 35 kPa, with divisions at least every 2 kPa, and an accuracy of 1.5 kPa.
  - 6.1.3 Thermometers:
- 6.1.3.1 *Air Thermometer*, for measuring compressed air temperature. ASTM Precision Thermometer having a range from -20 to 102°C, graduated in 0.2°C and conforming to the

 $<sup>^{\</sup>rm 1}$  This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.C0 on Turbine Oils.

Current edition approved Nov. 1, 2003. Published December 2003. Originally approved in 1975. Last previous edition approved in 2002 as D 3427–02.

<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

 $<sup>^{3}\,\</sup>mathrm{Available}$  from Beuth Verlag GmbH, Burggrafenstrasse 6, 1000 Berlin 30, Germany.



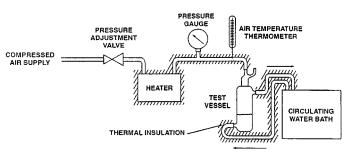


FIG. 1 Apparatus for the Determination of Air Release Time

requirements for Thermometer 12C as prescribed in Specification E 1 is suitable. A temperature sensor of at least equivalent performance is also suitable. Care shall be taken to avoid restricting the air path with the thermometer bulb or any adapter used.

6.1.3.2 Sample Thermometer, for measuring the temperature of the sample during preparation and trial runs. ASTM Precision Thermometer having a range from -20 to 102°C, graduated in 0.2°C and conforming to the requirements for Thermometer 12C as prescribed in Specification E 1 is suitable. A temperature sensor of at least equivalent performance is also suitable.

6.1.4 *Heater*, to bring the compressed air up to measurement temperature. A coil of copper tubing immersed in the circulating bath (see 6.1.5) is suitable at 25°C, but additional heating is necessary at 50 and 75°C. This can be obtained by an additional bath, or by using a separate steam or electric heat exchanger. The temperature of the air shall be measured by a thermometer located as close as possible to the testing vessel and meeting the specifications shown in 6.1.3.

Note 3—The application of thermal insulation to the pipework carrying the heated compressed air is recommended.

6.1.5 Circulating Bath, approximately 10-L capacity with a rate of flow of 10 L/min and capable of maintaining the test cell at a temperature of 25, 50, or 75°C within  $\pm 0.1$ °C.

Note 4—Use of water in the bath has been found to minimize electrostatic effects.

Note 5—The application of thermal insulation to the pipework carrying the heated bath fluid is recommended.

(Warning—The use of glass vessels with glass host fittings for circulating 75°C (167°F) bath medium is potentially dangerous. Back pressure in excess of a gage pressure of 70 kPa (10 psi) can be generated when the bath medium is pumped at the required rate; this can cause fracture of the glass

or slippage of the hose connections. Use of a pressure relief valve set at 70 kPa (10 psi) is recommended. In addition, use of a safety shield is recommended.)

6.2 *Balance*, capable of measuring density, accurate to 0.5 kg/m<sup>3</sup>.

6.3 *Sinker*, having a round or tapered bottom of 5 or 10-mL displacement,  $80.0 \pm 1.5$ -mm length. If the sinker contains a thermometer, it shall be usable between 25 and 75°C.

6.4~ *Oven*, capable of heating samples to  $10^{\circ}$ C above the test temperature.

 $6.5\ \textit{Timer}$ , readable to  $0.1\ \text{min}$ , with an accuracy of better than  $0.1\ \%$ .

6.6 *Pump*, with a nonpulsating output and capable of maintaining an air flow of 40 L/min at a pressure of 20 kPa (optional, see 7.4).

## 7. Materials

7.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Purity of Water—Reagent water as defined by Type II of Specification D 1193.

7.3 *Acetone*, minimum reagent grade. (**Warning**—Flammable. Health hazard.)

7.4 Air, Compressed, dry and free from moisture, particulates, and oil. Air from a cylinder or a nonpulsating pump may be used. (Warning—Compressed gas under high pressure.)

7.5 Cleaning Reagent, Cleaning either in hot Nochromix<sup>5</sup> (**Warning**—Corrosive health hazard oxidizer), or a 24-h soak at room temperature in Micro<sup>6</sup> solution.

Note 6—Previously, chrome sulfuric acid was used in this procedure. Other test methods (for example, Test Method D 1401) have demonstrated acceptable, statistically equivalent results when Nochromix or Micro is used to replace sulfuric chromic acid for cleaning.

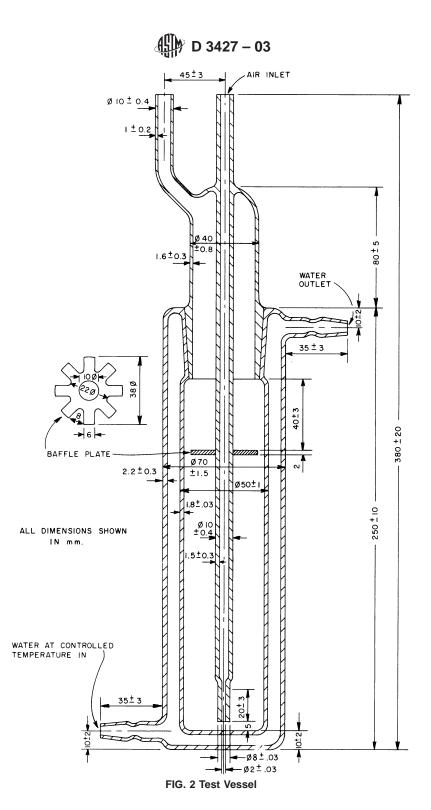
7.6 *n-Heptane*, reagent grade. (**Warning**—Flammable. Harmful if inhaled.)

Note 7—Other alternate solvents, such as toluene, etc., may be used in place of n-heptane.

<sup>&</sup>lt;sup>4</sup> Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Annual Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>&</sup>lt;sup>5</sup> The sole source of supply of Nochromix known to the committee at this time is Godax Laboratories, Inc., 720-B Erie Ave., Takoma Park, MD 20912. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee <sup>1</sup>, which you may attend.

<sup>&</sup>lt;sup>6</sup> The sole source of supply of Micro known to the committee at this time is International Products Corp., P.O. Box 70, Burlington, NJ 08016. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee <sup>1</sup>, which you may attend.



# 8. Preparation of Apparatus

- 8.1 Clean the interior of the test vessel, including the air inlet and sinker, and all other glassware coming in contact with the sample, before each determination in the following manner:
- 8.1.1 Rinse away the oil residue with n-heptane (**Warning**—see 7.6) and acetone (**Warning**—see 7.3) and dry by air blowing.
- 8.1.2 Clean the apparatus by immersing in cleaning reagent in order to remove completely any traces of silicone.
  - 8.1.3 Rinse with reagent water.

8.1.4 Rinse with acetone (**Warning**—see 7.3) and dry with clean compressed air (**Warning**—see 7.4).

Note 8—Oil misting occurs during blowing. The test vessel should be in a hood, or the air outlet tube should be connected to a vent that removes the vapors.

# 9. Procedure

9.1 Assemble the test apparatus as shown in Fig. 1. Set the compressed air temperature to within 0.2°C of the desired test temperature. Set the circulating bath to give a specimen

temperature within 0.1°C of the desired test temperature. The required bath temperature setting and time for the specimen to equilibrate can be established for an equipment setup by making trial runs with a thermometer in the oil specimen. For oils with a viscosity at 40°C of less than 9.0 cSt, the test temperature shall be 25°C. For oils with a viscosity at 40°C between 9.0 and 90 cSt, the test temperature shall be 50°C. Oils having a viscosity at 40°C greater than 90 cSt shall be tested at 75°C.

- $9.2~{\rm Warm}$  approximately  $200~{\rm mL}$  of the oil to be tested in an oven set at a temperature of  $10^{\circ}{\rm C}$  higher than the test temperature.
- 9.3 Pour 180  $\pm$  5 mL of the heated sample into the test vessel.
- 9.4 Allow the sample to equilibrate to the desired test temperature, using the heating-up time previously established (see 9.1) or by directly measuring the temperature.
- 9.5 Warm the sinker of the balance to the test temperature in an air bath, such as a glass cylinder fitted with a suitable cover situated in the circulating bath. When the sinker has reached the test temperature, immerse it in the sample, taking care that no air bubbles cling to it. Attach the sinker to the beam of the density balance by means of the platinum wire so that the bottom of the sinker is  $10 \pm 2$  mm from the bottom of the test vessel.

Note 9—The sinker can take approximately 20 min to reach  $50^{\circ}$ C or 35 min to reach  $75^{\circ}$ C.

- 9.6 Allow the density reading to stabilize, read the density from the balance to the nearest 0.1 kg/m<sup>3</sup>, and record it as the initial density.
- 9.7 Return the sinker to the air bath and replace it with the air inlet tube as shown in Fig. 2. After 5 min, start the supply of air at a gage pressure of 20 kPa at the required temperature. Maintain the pressure and temperature of the air (25, 50, and 75°C) by readjustments, if necessary.
- 9.8 After  $7 \pm 0.1$  min, shut off the air and immediately start the timer. Quickly remove the inlet tube from the test vessel, and immerse the sinker in the oil/air dispersion. Attach the wire to the beam and maintain a distance of  $10 \pm 2$  mm between the bottom of the sinker and the bottom of the test vessel.

Note 10—In the case of certain oils, which form a considerable volume of oil/air dispersion, the top of the sinker can initially be in foam, and hence, density readings at this time can be in error.

9.9 Record the time, to the nearest 0.1 min, for the density to return to the target of 99.8 % of the initial density  $(d_0)$ . If the time is greater than 30 min, discontinue the test.

Note 11—The test may be run for a longer period of time by agreement between the laboratory and the customer.

Note 12—For some applications, the shape of the air release time curve may be required. This can be implemented by recording the density at intervals, as required.

9.10 Certain oils may lose light components during the air saturation, thus changing their effective density. This will be noted if the time for the density to return to the initial figure is instantaneous. Where air release value information is required for such oils, an air releasing time curve may be drawn. If the

loss causes any part of the sinker to be exposed, discontinue the test and repeat, using a sample that is 10 mL larger than previously used.

## 10. Calculation of Density

10.1 Density:

Density of sample 
$$(kg/m^3) =$$
 (1)

 $\frac{\text{[Weight of sinker in air (kg) - Weight of sinker in sample (kg)]}}{\text{Volume of sinker (m}^3)}$ 

Note 13—A 10 mL sinker has a volume of  $10 \times 10^{-6}$  m<sup>3</sup>.

10.2 Target Density:

Initial density 
$$(d_0) \times 0.998$$
. (2)

10.3 If the tare key of the balance is pressed while the sinker is being weighed in air so that the reading is zeroed, then the following may be used:

Density of sample 
$$(kg/m^3) = \frac{\text{Weight of sinker in sample } (kg)}{\text{Volume of sinker } (m^3)}$$
 (3)

Note 14—Some instruments automatically make the above calculations.

10.4 The volume of the sinker may be determined by weighing it in air, then in water. The difference in weight (kg)  $\times$  10<sup>-3</sup>= volume in m<sup>3</sup>.

## 11. Reporting

11.1 Report the air release time, as recorded in 9.9, and the test temperature in °C.

## 12. Precision and Bias 7

- 12.1 *Precision*<sup>8</sup>—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:
- 12.1.1 Repeatability—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

12.1.2 Reproducibility—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

<sup>&</sup>lt;sup>7</sup> Supporting data (the results of the cooperative test program, from which these values have been derived) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1014. The program was run by six laboratories, using five samples of unused steam turbine oils and base stocks with air used as the entrained gas. Five samples were tested at one temperature and four at another. Since some reports were incomplete, this resulted in 48 pairs of replicated data.

 $<sup>^8</sup>$  The precision statement was developed using 1.1.1-trichloroethane and chrome sulfuric acid in the cleaning procedure.

(5)

1.3 times the square root of their mean

12.2 The closely related test method DIN 51 381 reports precision in another format. For ready comparison, the above

statement is shown in DIN terms in Table 1.

12.3 *Bias*—The procedure in Test Method D 3427 for measuring air release properties of petroleum oils has no bias because the value of the air bubble separation time is defined

only in terms of this test method. There is no known bias

relative to the DIN 51 381 method.

Note 15—The above precision was determined only at test temperatures of 50°C and 82°C. A new interlaboratory test program is planned.

#### TABLE 1 Comparison of ASTM and DIN Precision Data

Mean Test Result, min	Repeatability		Reproducibility	
	DIN	ASTM	DIN	ASTM
Up to 5	1	0.7	2	2.1
Over 5 to 10	2	1.3	3	3.6
Over 10 to 15	3	1.6	4	4.7

#### 13. Keywords

13.1 air entrainment; air release time; gear oil; hydraulic oil; turbine oil

#### SUMMARY OF CHANGES

Subcommittee D02.C0 has identified the location of selected changes to this standard since the last issue (D 3427–02) that may impact the use of this standard.

- (1) Added new Note 2, allowing the test to be run at other temperatures.
- (2) Changed quality of acetone required from conforming to Specification D 329 to "minimum reagent grade."
- (3) Added new Note 7 allowing the use of alternate solvents, such as toluene, in place of *n*-heptane.
- (4) Added new Note 11 allowing the test to be run longer than 30 min.

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