Designation: D 3520 – 04

An American National Standard

Standard Test Method for Quenching Time of Heat-Treating Fluids (Magnetic Quenchometer Method)[1]

This standard is issued under the fixed designation D 3520; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for making a comparative evaluation of the quenching speed characteristics of petroleum fluids used in the quenching of metals by means of the Magnetic Quenchometer.

NOTE 1—A comparison method for testing the hardenability of steel is Test Method A 255.

NOTE 2—An additional comparison method for evaluation of the quenching speed characteristics of petroleum fluids used in the quenching of metals is Test Method D 6200.

1.2 This test method provides a measure of changes in oil chemistry due to contamination, base oil degradation, and additive drag-out during use. This test measures changes in the quenching speed characteristics of petroleum fluids at the high temperature (>354°C). This test method does not reliably predict metallurgical performance.

1.3 *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 requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

A 255 Test Method for End-Quench Test for Hardenability of Steel

D 91 Test Method for Precipitation Number of Lubricating Oils

D 92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D 97 Test Method for Pour Point of Petroleum Products

D 287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)

D 484 Specification for Hydrocarbon Drycleaning Solvents[3]

D 1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids

D 1744 Test Method for Water in Liquid Petroleum Products by Karl Fischer Reagent[3]

D 6200 Test Method for Determining Cooling Characteristics of Quench Oils by Cooling Curve Analysis

3. Summary of Test Method

3.1 This test method determines the time for cooling a chromized nickel ball from approximately 885°C (1625°F) to approximately 354°C (670°F) when quenched in 200 mL of test fluid in a metal beaker at 21 to 27°C (70 to 81°F). The quenching time is recorded by a digital timer which is energized by a photoelectric cell from light produced by the ball at 885°C (1625°F) and which is stopped when the ball becomes magnetic (Curie Point, approximately 354°C) and is attracted by a magnet to the side of the beaker, tripping a relay to stop the timer.

4. Significance and Use

4.1 The results obtained by the test method described are useful as guides in selecting fluids with respect to quenching speed characteristics desired for metal quenching applications.

NOTE 3—Although this test method has been found useful for some water-based fluids, the statistical significance of the test has been established only by round-robin testing of petroleum-based fluids.

4.2 These results will provide a measure of quenching speed (cooling rate) from approximately 885°C (1625°F) to 354°C (670°F) and are not directly proportional to hardness obtainable on metals quenched therein as many other factors are involved in the quenching process in actual plant operation with production parts.

[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.L0 on Industrial Lubricants.

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[2] 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.

[3] Withdrawn.

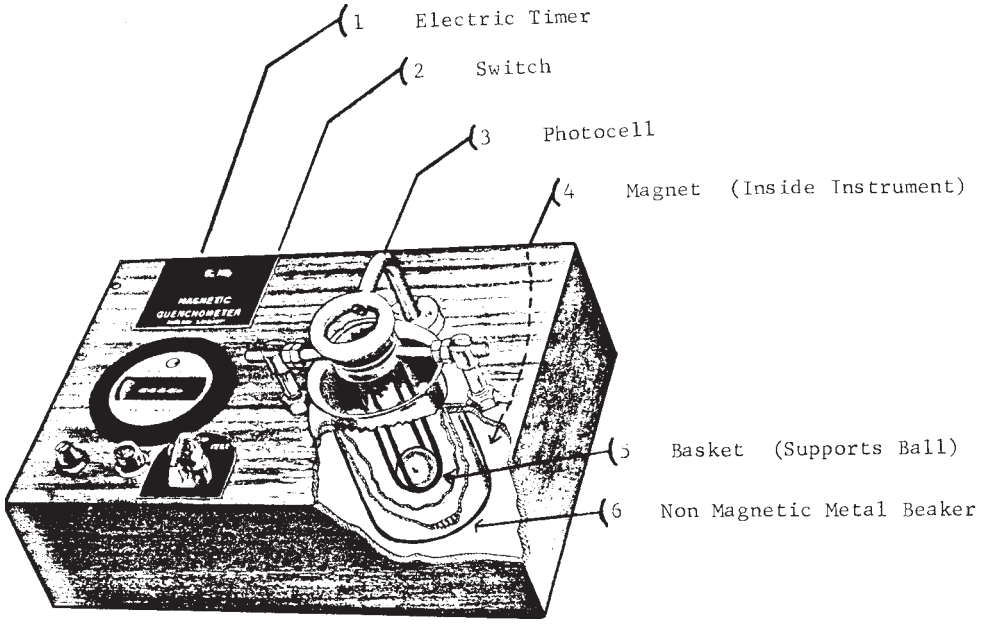

FIG. 1 Magnetic Quenchometer

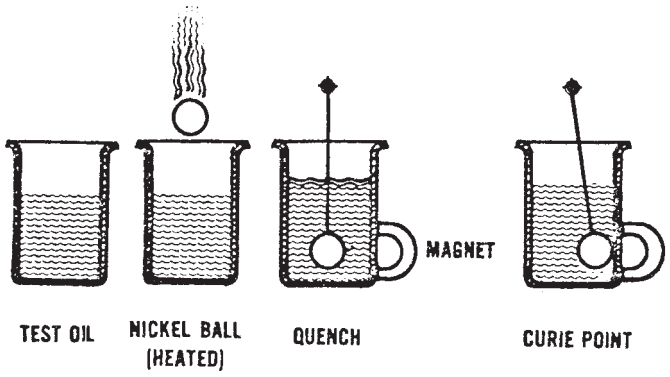

FIG. 2 Test Sequence in Beaker

NOTE 4—Test Method D 6200 describes the equipment and procedure for characterizing the time-temperature performance of a quenching oil through the entire cooling process.

5. Apparatus

5.1 *Magnetic Quenchometer,*[4] as shown in Figs. 1 and 2.

5.2 *Furnace, Electric Muffle*, with air atmosphere, capable of maintaining a temperature of 885 ± 5°C (1625 ± 9°F), as measured at the ball by the thermocouple prior to quenching, and fitted with *(1)* a clean ceramic base plate, for supporting the test ball, and *(2)* a No. 14 B&S gage Chromel-Alumel thermocouple probe (without a protection tube) inserted through a hole in a wall of the furnace, and positioned to contact the test ball.

NOTE 5—A rheostat on the power supply may be used to provide a closer control of furnace temperature, or a separate thermocouple-connected controller near the heating elements can be used to minimize temperature overrides, or both may be used. Further, 1 by 2 by 3-in. heat sinks can be placed next to the area where the ball is to be placed to act as heat shields and provide a more uniform temperature in this particular area.

6. Materials

6.1 *Ball, Chromized Nickel,*[4] having a diameter of 22.22 ± 0.13 mm (0.875 ± 0.005 in.), a weight of 50 ± 2 g, and surface finish from 0.38 to 0.76 µm (15 to 30 µin.).

6.2 *Reference Fluid.*

NOTE 6—Fluids used in cooperative testing covered in Annex A2 are suitable. Reference fluids TDL-VI-1 (35) the primary reference fluid and TDL-VI-1 (100X) the secondary standard are suitable.[4]

NOTE 7—The use of non-chromized nickel balls is not included in this test method and is not recommended since the results obtained are commonly more scattered and may be inconsistent with data properly obtained with chromized nickel balls. It is also recommended that chromized nickel balls be used with a final initialization value of 29.0 to 32.0 seconds to obtain optimal repeatability.

6.3 *Stoddard Solvent*, conforming to Specification D 484. (**Warning—**Combustible, skin irritant on repeated contact, aspiration hazard.)

6.4 *Precipitation Naphtha*, conforming to the requirement for precipitation naphtha in Test Method D 91. (**Warning—** Extremely flammable, skin irritant on repeated contact, aspiration hazard.)

6.5 *Forceps*, approximately 450 mm (18 in.) long. Weld two 20 mm (¾ in.) stainless steel washers to the tips of the forceps for greater safety in transporting the hot nickel balls.

6.6 *Tissue*, lintless.

7. Preparation of Apparatus

7.1 *Tester*:

7.1.1 Place the instrument near the furnace and where lighting does not activate the photo cell.

7.1.2 Connect to 110 V ac.

[4] The sole source of supply of the apparatus known to the committee at this time is Testron Corp., 34153 Industrial Rd., Livonia, MI 48150. 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 [1], which you may attend.

7.1.3 Set the switch (Fig. 1, Item 2) to ON position.

NOTE 8—The shield of the photo cell should be cleaned before each test with lintless tissue to ensure proper functioning.

NOTE 9—The "horseshoe" magnet inside the instrument is positioned as close as possible to the beaker housing. Occasionally check the magnet and reposition if necessary.

7.2 *Test Ball*:

7.2.1 Apply Stoddard solvent. Dry with lintless tissue. Repeat.

7.2.2 Apply precipitation naphtha. Wipe off with lintless tissue. Repeat. Air dry, and remove any traces of lint.

NOTE 10—Do not allow the solvents to evaporate from the ball. The solvents should be absorbed by the tissue. Avoid touching the ball with hands or any other sources of contamination.

7.3 *Test Sample*:

7.3.1 Determine water content per Test Method D 1744.

7.3.2 If the water content is >0.03 %, then dry the oil sample prior to the test.

7.3.3 The oil is to be dried by heating the oil to 100°C (212°F) with agitation. Continue heating the oil at 100°C (212°F) with agitation until bubbling ceases (typically 1 to 2 h).

NOTE 11—It is not recommended that this test method be used with dirty quench oils, those oils with 0.5 % pentane insolubles, since dirty oils may lead to erroneous results and potentially damage the nickel ball.

NOTE 12—Extreme care must be used to remove carbonaceous deposits or other residues which might be difficult to remove with Stoddard solvent. Attempts with fiber brushes, etc., must be carefully viewed, and if the ball does not appear satisfactory when tested in the standard fluid TDL-VI-1 (35) (see Annex A2), it should be discarded and a new ball put into use.

8. Procedure for Making a Single Test

8.1 Using forceps, place the cleaned test ball on the clean ceramic base plate in the heated furnace, with the ball positioned to contact the exposed wires of the thermocouple.

8.2 Maintain the test ball in the furnace for a minimum of 900 s (15 min) at temperature as measured from the time the thermocouple indicates a temperature of 885 ± 5°C (1625 ± 9°F).

8.3 Charge 200 mL of the test or reference fluid at 21 to 27°C (70 to 81°F) into a clean stainless steel beaker supplied with the tester.

NOTE 13—A fresh charge of fluid should be used for each test.

NOTE 14—The presence of moisture will affect quench time values. Bright and clear-appearing fluids may still contain moisture. The degree of moisture in test fluids may be ascertained by Test Method D 1744 or appropriate accepted standard methods. If desired, moisture level may be reduced by laboratory techniques suitable for that particular test fluid.

8.4 Set the dial of the electric timer on the test instrument to read 0.

8.5 Set the switch of the timer to TEST position.

8.6 Using forceps, quickly transfer the heated test ball 885 ± 5°C (1625 ± 9°F) from the furnace to the tester, dropping the ball through the photo cell ring into the basket which suspends the ball in the fluid with the total elapsed time for transferring the ball to the tester being less than 5 s. As the test ball passes through the photo cell, the electric timer is energized. When the ball temperature is below 354°C (670°F), Curie Point, the ball becomes magnetic and is attracted toward a magnet enclosed in the instrument activating a switch and stopping the timer.

8.7 After the electric timer stops, read and record the quench time.

8.8 Record the designation number of the test ball being used.

NOTE 15—Do not disfigure the balls, as small marks change their characteristics. Instead, individually numbered envelopes or containers should be used for storage. Identification of position in the furnace should be used to identify the balls.

8.9 Set switch of the tester to OFF position.

8.10 Swing the photocell ring to the side and remove the ball by lifting the basket from the yoke.

8.11 Remove the beaker containing the fluid. Discard this used fluid.

8.12 Using fresh test fluid, repeat 8.1-8.11 while using the same ball.

8.13 Repeat 8.1-8.11 using fresh primary reference fluid.

NOTE 16—An evaluation shall consist of two tests using the same stabilized ball followed by a check test with the reference fluid using the same test ball. The evaluation is valid if the check test with the reference fluid agrees within 1.0 s of the previous reading for that ball in the reference fluid.

9. Procedure for Stabilizing New or Unknown Ball

9.1 As indicated, follow 8.1-8.11. Repeat at least three times with the same ball using the primary reference fluid, discarding the fluid after each run and until consecutive tests yield times within 1.0 s maximum deviation. The ball is then considered to be stabilized.

NOTE 17—Since operation of this test depends upon reliable and repetitive performance of the test balls, it is advisable to maintain an individual chronological record of results obtained from each ball. Rate of degradation and possible contamination of the ball can then be observed. Different test balls frequently give slightly different quench times with the same fluid. It is important, therefore, that comparative tests on a test fluid and a reference fluid be run using the same ball. The round-robin test balls were originally stabilized by General Motors Corp. prior to the initiation of the round robin.

10. Calculations and Reports

10.1 Record (*1*) quench times and (*2*) identifications of the test balls used.

NOTE 18—A suggested form for recording the data is supplied in Appendix X1.

10.2 Calculate the average value of the two runs on the test fluid.

10.3 Calculate the average value of the tests run on the reference fluid immediately preceding and following the runs on the test fluid.

10.4 Calculate and report the relative cooling index (RCI), % as follows:

$$RCI = F_R \times 100/F_T \quad (1)$$

where:

F_R = average quench time of reference fluid, s, and

F_T = average quench time of test fluid, s.

11. Precision [5]

11.1 *Precision*—The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:

11.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in 1 case in 20:

5 % of the mean (quench time, seconds) (2)

11.1.2 *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, and in the normal and correct operation of the test method, exceed the following values only in 1 case in 20:

9 % of the mean (quench time, seconds) (3)

11.2 *Bias*—The procedure in Test Method D 3520 for measuring quenching speed characteristics of petroleum fluids has no bias because the value of quenching speed can be defined only in terms of a test method.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1025. The program was performed by eight laboratories using two oils and six test balls (see Appendix X2).

12. Keywords

12.1 Curie point; magnetic quenchometer; quench; quenchant

ANNEXES

(Mandatory Information)

A1. CHROMIZING NICKEL BALL[4,6]

A1.1 Prepare the suitable nickel ball by vapor blasting with 240 grit abrasive.

A1.2 Water rinse thoroughly; preferably scrub the surface to remove all blasting media.

A1.3 Rack balls on spline rack between two sharp-pointed spring contacts.

A1.4 Plate in conventional hard-chrome bath containing 33 oz/gal CrO_3 (**Warning**—Causes severe burns, a recognized carcinogen, strong oxidizer) and 0.26 % (0.33 oz/gal) H_2SO_4 at 57 to 59°C (135 to 138°F). (**Warning**—Causes burns, vapor harmful.)

A1.5 With dc on, place racks in plating solution and immediately apply 600 A/ft^2 for 30 s and then reduce to 225 A/ft^2.

A1.6 After 300 s (5 min) of plating, turn dc down to 1.0 V and remove the rack. *Do not* rinse parts. Change the contacts of each ball.

A1.7 Replace the racks in the plating bath and resume 225 A/ft^2.

A1.8 Repeat A1.6 twice at 900-s (15-min) intervals.

A1.9 Elapsed plating time of 2700 s (45 min) will give a uniform thickness approximately 0.13 mm (0.005 in.).

A1.10 *Diffusion Cycle*—Diffuse the chromium into nickel in a dry hydrogen atmosphere (−68°C (−90°F) or drier) at 1010°C (1850°F) for 4 h (**Warning**—Extremely flammable gas under pressure). Place the nickel balls in a stainless steel container. Purge with hydrogen. Then place the container into the furnace at 1010°C (1850°F). Remove the container from the furnace and air cool.

[6] Suitable balls, prepared and tested for proper quenching characteristics, are available from Testron Corp.

A2. QUENCH OIL STANDARDS[4]

A2.1 The primary reference fluid is a USP white oil with the characteristics shown below (identified as TDL-VI-1 (35)):

ASTM Method	Test	Typical Results	Supplier Specification
D 92	Flash, Cleveland open cup, °C (°F)	218 (425)	204 (400) min
D 97	Pour point, °C (°F)	−27 (−15)	−18 (0) max
D 445	Viscosity at 40°C (104°F), cSt	78	76 to 80
D 445	Viscosity at 100°C (212°F), cSt	7.8	...
D 287	Gravity, °API at 16°C (60°F)	28.4	...
D 1218	Refractive index at 20°C (68°F)	1.4814	...

A2.2 The secondary standard is a proprietary quenching oil, with the characteristics shown below (identified as TDL-VI-1 (100X)):

ASTM Method	Test	Typical Results	Supplier Specification
D 92	Flash, Cleveland open cup, °C (°F)	180 (355)	171 (340) min
D 97	Pour point, °C (°F)	−30 (−20)	...
D 445	Viscosity at 40°C (104°F), cSt	19	17 to 20
D 445	Viscosity at 100°C (212°F), cSt	3.85	...
D 287	Gravity, °API at 16°C (60°F)	30	...

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLE OF TEST REPORT
Results of Magnetic Quenchometer Test

ASTM D 3520

Date of Test _____; By Operator _____
Type of Ball Chromized Nickel; Ball Designation(s) _____
TEST FLUID _____

		Reference Fluid	Test Fluid
A	Time into furnace		
B	Time[A] when 885°C (1625°F) is reached		
C	Time[A] out of furnace		
D	Time to reach 885°C (1625°F) min (B − A)		
E	Time at 885°C (1625°F) min (C − B)		
F	Quench time, 885-354°C, s		

Remarks[B] Test No. _____
Test No. _____
Test No. _____
Test No. _____

$$\text{Relative cooling index (RCI), \%} = \frac{\text{Average Quench Time, Ref. fluid} \times 100}{\text{Average Quench Time, Test fluid}}$$

[A] Clock time.
[B] Note anything unusual or any necessary deviations in procedure, time, or temperature.

X2. EXAMPLES OF DATA AND CALCULATION OF RCI % VALUES

X2.1 *Data and Calculation of RCI % Values*—See Table X2.1.

TABLE X2.1 Range of Tests—Seconds and RCI Values[A]

Cooperating Laboratory	Example of Test Fluid	Primary Standard	RCI Value[B]
	100×	35	
No. 1[C]	12.0–14.6	27.4–34.1	206–254
2	13.0–13.8	31.7–33.7	241–245
3	9.2–11.7	29.3–30.0	262–274
4	12.3–14.0	30.6–32.6	235–247
5	13.2–14.5	32.6–34.7	234–246
6	12.1–14.5	32.0–35.2	241–266
7	11.6–13.9	30.5–34.0	246–265
8[C]	32.9–35.6	32.9–36.3	91–98
9	10.6–13.4	26.9–33.5	243–261
10[C]		26.1–33.0	

[A] From data used to determine precision data as submitted by round-robin testing of two oils with three new chromized nickel balls purchased by each cooperator and three "traveling" balls used for "life" tests.

[B] Representative calculation of % RCI Value.

Example using average data from Laboratory No. 2 above when testing the RCI % value of the 100× fluid.

$$RCI\,\% = \frac{\text{avg time reference fluid} \times 100}{\text{avg time test fluid}} = \frac{2 \times (31.7 + 33.7) \times 100}{2 \times (13.0 + 13.8)} = 244\ \%$$

[C] Laboratory No. 8 was deleted from statistics due to use of a contaminated sample of oil 100× and instrument problems. Laboratory no. 1 tested both the primary and the secondary standards with the three balls (X, Y, and Z) used for the traveling set and tested each A, B, and C ball to stabilization in the primary reference fluid only prior to sale to each cooperator. On return of the traveling set, Laboratory No. 1 (now Laboratory 10) tested each ball (A, B, and C) again in the primary standard only to check degradation during the round-robin program.