



Standard Test Method for Semi-Quantitative Field Test Method for Base Number in New and Used Lubricants by Color-Indicator Titration¹

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^{ε1} NOTE—Warning notes were editorially moved into the standard text in March 2003.

1. Scope

1.1 This test method covers a procedure for determining the basic constituents in petroleum products in the field or laboratory using a pre-packaged test kit. The test uses a micro-titration resulting in a visual end point facilitated by a color indicator.

1.1.1 This test method covers base numbers from 0 to 20. It can be extended to higher ranges by diluting the sample or by using a smaller sample size; however, the precision data were obtained for base numbers up to 20.

1.2 This test method can be used to indicate relative changes that occur in an oil during use under oxidizing conditions. Although the test is performed under closely specified conditions with standardized reagents, the test method does not measure an absolute basic property that can be used to predict performance of an oil under service conditions. No general relationship between bearing corrosion and base number is known.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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:

D 1193 Specification for Reagent Water²

D 2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration³

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁴

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products⁴

D 4739 Test Method for Base Number Determination by Potentiometric Titration⁴

3. Terminology

3.1 Definitions:

3.1.1 *base number, n*—the quantity of an acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide per gram of sample, that is required to titrate a sample dissolved in the specified solvent to a specified end point (for example, Test Method D 4739).

3.1.2 *Discussion*—This test method uses fixed amounts of *isooctane* and alcoholic hydrochloric acid as the sample solvent and the end point is defined as the amount of titrant required to reach a yellow end-point with a methyl red indicator solution.

4. Summary of Test Method

4.1 To determine the base number of an oil sample, the sample is dissolved in a fixed amount of *isooctane* and alcoholic hydrochloric acid. The solution is mixed well with 7 mL of sodium chloride solution and the aqueous and organic phases are allowed to separate. The aqueous phase is then decanted off and a small amount of methyl red indicator is added. The solution is titrated with a solution of sodium hydroxide contained in a calibrated 1 mL micro-burette. When the solution changes from magenta to yellow, the titration is stopped and the base number is read off the side of the titration burette.

5. Significance and Use

5.1 New and used petroleum products can contain basic constituents that are present as additives or as degradation products formed during service. The amount of these additives in an oil can be determined by titrating against an acid. The base number is a measure of the amount of basic substance in

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 05.02.

the oil, always under the conditions of the test. A decrease in base number is often used as a measure of lubricant degradation, but any condemning limits must be empirically established.

5.2 This test method uses reagents that are considered less hazardous than most reagents used in alternate base number methods. It uses pre-packaged reagents to facilitate base number determinations in the field where scientific equipment is unavailable and quick results are at a premium.

NOTE 1—Results obtained by this test method⁵ are similar to those obtained by Test Method D 2896.

6. Apparatus⁶

6.1 *Sampling Syringe*, fixed volume of 0.35 mL.

6.2 *Test Tubes (2)*, pliable, plastic with threaded tops capable of holding at least 20 mL, one with a standard screw cap and the second with a screw cap that is equipped with a dispenser nozzle.

6.3 *Titrating Burette*, disposable, 1.0 mL.

6.4 *Filter Funnel*, capable of removing suspended oil droplets from aqueous solutions. Polypropylene wool has been found to work well for this purpose.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Pre-packaged, manufactured test kits shall use only reagent grade chemicals.

7.2 *Purity of Water*—All water will be reagent water as specified by Type III of Specification D 1193.

7.3 *Hydrochloric Acid*, 0.114 mol/L (± 0.001 mol/L) HCl in propan-2-ol (*isopropyl alcohol*). (**Warning**—Flammable.)

7.4 *Methyl Red Indicator*, 0.05 % (m/v) solution of methyl red in anhydrous ethanol. (**Warning**—Flammable.)

7.5 *Sodium Chloride Solution*, 7.0 mL of 20 % (m/m) NaCl in reagent grade, CO₂ free water.

7.6 *Sodium Hydroxide Titrant*, 1.0 mL of 0.0645 mol/L (± 0.0005 mol/L) NaOH sealed from the environment in a plastic titration burette which is indexed with base number units from 0 to 20. (**Warning**—Toxic and corrosive.)

7.7 *2,2,4-Trimethyl Pentane (isooctane)*—(**Warning**—Flammable.)

8. Procedure⁷

8.1 Obtain samples for analysis by this test method in accordance with instructions given in Practice D 4057 or D 4177.

8.2 If a sample is to be taken directly from a hot engine, follow the manufacturer's instructions for obtaining a representative sample. Allow the oil to cool to <50°C before proceeding further.

8.3 It is essential to ensure the sample is representative since any sediment can be acidic or basic or have adsorbed acidic or basic material from the sample. When necessary, samples are warmed to aid mixing. Used oils should be vigorously mixed to ensure homogeneity before sampling.

8.4 As used oils can change appreciably in storage, samples shall be tested as soon as possible after removal from the lubricating system.

8.5 Prepare the oil sample by shaking well to disperse any particulates.

8.6 Place the tip of the fixed volume sampling syringe into the oil sample. Draw back the plunger of the sampling syringe until it stops. Remove the syringe from the oil sample and wipe the outside clean of any excess oil. Dispense the entire volume of the syringe into the first test tube. Replace the cap on the test tube.

8.7 Add 1.0 mL of 2,2,4-trimethyl pentane (*isooctane*) and shake the tube well to mix the oil with the diluent. Add 1.0 mL of 0.114 mol/L alcoholic HCl and shake the tube well for 30 s, allowing the acid to react with the diluted oil sample.

8.8 Pour 7.0 mL 20 % NaCl solution into the oil mixture. Cap the tube and shake the mixture well for 30 s so that the two phases are completely dispersed. Stand the tube upside-down on its cap and allow the aqueous and organic phases to separate.

8.9 Place the plastic filter into the top of the second test tube. Keeping the first test tube upside-down and vertical, open the dispenser cap on the tube and dispense exactly 5 mL of the aqueous solution through the filter into the second test tube. Stop dispensing as soon as 5 mL has been placed in the tube.

8.10 Place the 1.0 mL micro-burette into the tube containing the 5.0 mL of aqueous extract. Seal the burette into place by screwing the threaded cap of the burette onto the test tube. Add 0.5 mL of the methyl red indicator solution and disperse the indicator throughout the solution by gently shaking. Start slowly dispensing the titrant into the solution and shake gently. As soon as the solution turns from magenta to yellow, stop the titration and read the result, in base number units, off the side of the burette barrel. The reading should be taken at the tip of the plunger.

9. Precision and Bias

9.1 The precision of this test method as determined by the statistical examination of test results from 11 samples of new and used oils, covering base numbers between 0 and 20, run in duplicate by nine different laboratories (Research Report D02-1345) is as follows:

9.1.1 *Repeatability*—The difference between successive 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 value only in one case in twenty:

$$r = 0.94 \text{ base number units (mg KOH/g sample)} \quad (1)$$

9.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1345.

⁶ A patent pending field test kit containing appropriate apparatus reagents, and materials is available from Dexsil Corp., Hamden, CT.

⁷ This procedure is covered by U.S. patent number 5,366,898, Dexsil Corp., Hamden, CT. If you are aware of alternative, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$R = 1.91 \text{ base number units (mgKOH/g sample)} \quad (2)$$

9.2 *Bias*—This procedure has no bias because the base number can be defined only in terms of the test method.

10. Keywords

10.1 base number; color-indicator titration; lubricants; test kit

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