Designation: D7821 – 12

Standard Test Method for Field Test Determination of Urea Concentration in Diesel Exhaust Fluid (DEF)[1]

This standard is issued under the fixed designation D7821; 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 technique for field testing the concentration of aqueous urea-based diesel exhaust fluid (DEF) prescribed for use in diesel engines equipped with selective catalytic reduction technology and is not intended to circumvent or replace the more accurate determination of urea content by refractive index laboratory method, described in ISO 22241-2, Annex C.

1.2 This test method is designed solely as a quantitative test to determine the concentration of urea in DEF and does not purport to determine the quality of DEF nor to detect trace or other contaminants therein. Biuret content of DEF creates a known bias in this test method. See section 9.2.1 for details.

1.3 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

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 *ISO Standard:*[2]

ISO 22241 22241Diesel engines—NOx reduction agent AUS 32

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *American Petroleum Institute (API), n*—a nonprofit trade association that licenses diesel exhaust fluid (DEF) marketers to use the API Diesel Exhaust Fluid Certification Mark. A marketer may be licensed to use the API Mark if its DEF is in compliance with the most recent and applicable edition of ISO 22241.

3.1.2 *diesel exhaust fluid, DEF, n*—preparation of aqueous urea [$(NH_2)_2CO$], containing 32.5 % by weight of technically pure urea in high-purity water with quality characteristics defined by ISO 22241.

3.1.3 *refractive index, n*—fundamental physical property of a substance that is related to the ratio of the velocity of light in air, to its velocity in the substance under examination, at a specified temperature and wavelength.

3.1.4 *refractometer, n*—optical instrument that measures the refractive index of a substance and can be used to characterize the concentration or mixture ratio of various substances.

3.1.5 *biuret, n*—chemical compound which appears as a characteristic impurity in aqueous urea solutions.

3.1.6 *AUS 32, n*—ISO designation for DEF also known as NOx reduction agent AUS 32 (aqueous urea solution).

4. Summary of Test Method

4.1 A digital refractometer with a special scale specific to the percent by weight of urea is used to measure the concentration of a DEF sample.[3]

4.2 To determine DEF concentration, the refractometer shall first be zero set to distilled water. Next, a sample of DEF is placed on the measuring surface of the refractometer and the temperature of the instrument, fluid, and ambient environment is allowed to equilibrate. A reading is taken by pressing a button, and the unit of measure and concentration are directly read from a digital display.

5. Apparatus

5.1 *Refractometer*—A refractometer is an optical instrument used to measure the physical properties of a solution. Refractometers suitable for use in this test method shall possess the following characteristics:

[1] This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and Related Fluids and is the direct responsibility of Subcommittee D15.25 on Diesel Exhaust Fluid.

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[2] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[3] Sources for DEF refractometers, used to establish the precision and bias of this method, MISCO Refractometer, 3401 Virginia Rd., Cleveland, OH 44122 and Reichert Technologies, 3362 Walden Avenue, Depew, NY 14043-2437.

5.1.1 *Unit of Measure*—The unit of measure for all DEF readings shall be urea percent by weight and this shall be clearly identified on the refractometer.

5.1.2 *Range*—The refractometer shall have a minimum measuring range of 0.0 to 37.0 percent by weight.

5.1.3 *Resolution*—The refractometer shall resolve readings to 0.1 % urea by weight.

5.1.4 *Precision and Bias*—The refractometer must be capable of reading with a repeatability and reproducibility equal to or better than the precision statement in Section 9 of this test method as determined through ASTM interlaboratory testing.

5.1.5 *Reference Wavelength*—Measurements shall be taken at, or referenced to, a wavelength of 589 nm (± 5 nm).

5.1.6 *Reference Temperature*—All measurements shall be referenced to a reference temperature of 20°C.

5.1.7 *Temperature Range*—The refractometer shall be capable of reading samples from 10 to 45°C.

5.1.8 *Automatic Temperature Compensation*—The refractometer shall provide automatic temperature compensation specific to aqueous urea solutions over the temperature range defined in 5.1.7.

5.1.9 *Digital Display*—The refractometer shall be equipped with a digital display capable of rendering a numeric representation of urea concentration.

5.1.10 *Zero Set*—The refractometer shall be capable of being zero set to distilled water by the user.

6. Calibration

6.1 Calibration is essential for minimizing sources of error in a measurement system. The refractometer should be zero set to distilled water before use.

6.2 Inspect the refractometer measuring surface to insure that it is free of residue from a previous test. Clean the sample surface and sample well with distilled water and tissue if needed.

6.3 The exact procedure for zero setting a refractometer varies based on the type and make of instrument. Consult the user manual for specific instructions on zero setting each make of refractometer.

6.4 For the most accurate readings, the refractometer, the calibration fluid, and the ambient temperature should be in an equilibrium state of temperature. If there is a temperature disparity, allow some time for the temperatures to equalize before taking a reading. The equilibrium state is a single temperature value between the range of 10 to 45 degrees C.

7. Procedure

7.1 *Obtaining a Test Sample:*

7.1.1 Make a record of the sample identity.

7.1.2 Great care should be taken to prevent contamination when taking samples of DEF directly from a vehicle or an original sample container. The sample container should only be opened long enough to remove a sample and should be immediately sealed.

7.1.3 A clean disposable plastic pipette should be used to transfer a sample to the refractometer and the pipette then immediately discarded.

7.2 *Measuring the Sample:*

7.2.1 The refractometer and ambient environment should be between 10 to 45°C.

7.2.2 Inspect the refractometer measuring surface to insure that it is free of residue from a previous test. Clean the sample surface and sample well with distilled water and tissue if needed.

7.2.3 Transfer a few drops of DEF sample and completely cover the measuring surface of the refractometer.

7.2.4 Allow adequate time for the temperature of the refractometer, fluid, and ambient environment to equilibrate.

7.2.5 Initiate the reading. The exact procedure for initiating a reading varies based on the type and make of instrument. Consult the user manual for specific instructions on initiating a reading for each make of refractometer.

7.2.6 Record the reading on the refractometer digital display to one decimal place in percent weight of urea.

7.2.7 Take four more readings of the same sample and average the results.

7.2.8 If the five values fluctuate by more than 0.5 %, allow a little more time for the temperature to equilibrate and retest the sample.

7.2.9 Clean measurement surfaces to remove all sample residue. Follow the manufacturer's guidelines for cleaning the instrument.

8. Report

8.1 Report the following information:

8.1.1 The sample identification for DEF analyzed (which may include such information as product name, lot number, production date, location, type of container, size, or any other information that may aid in its identification or origin).

8.1.2 The date and time of the test,

8.1.3 The name of the person completing the test, and

8.1.4 The weight percent urea found.

9. Precision and Bias

9.1 The precision of this test method as determined by statistical examination of interlaboratory results according to round robin testing[4] is as follows:

9.1.1 *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.

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 the long run, in normal and correct operation of the test method.

9.2 *Bias*—The results of bias determination will be available as part of the interlaboratory study and will be available within one year of completion of the study.

9.2.1 *Biuret Content*—A maximum limit of +0.3 % m/m biuret concentration in DEF is required by ISO 22241. This

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: DXXXX.

field-test method does not address biuret content and therefore, even when testing DEF which conforms to the ISO biuret specification, results may be biased by +0.3%. This bias is additive to any precision and bias of the test instruments.

10. Keywords

10.1 DEF; diesel exhaust fluid; refractometry; SCR; selective catalytic reduction; urea