
**Petroleum products — Determination
of knock characteristics of motor fuels
— Research method**

*Produits pétroliers — Détermination des caractéristiques
antidétonantes des carburants pour moteurs — Méthode de recherche*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received. www.iso.org/patents

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 28, *Petroleum products and lubricants*.

This fourth edition cancels and replaces the third edition (ISO 5164:2005). Besides improving the understanding of some of the procedures, the main revision lays in the introduction of the so-called digital detonation meter. The revision includes allowances both measurement systems:

- a) the knock measurement system based on analogue technology, and
- b) the XCP digital technology used in the digital detonation meter.

Introduction

The purpose of this International Standard is to accord ISO status to a test procedure that is already used in a standardized form all over the world. The procedure in question is published by ASTM International as Standard Test Method D 2699-12.

By publishing this International Standard, ISO recognizes that this method is used in its original text in many member countries and that the standard equipment and many of the accessories and materials required for the method are obtainable only from specific manufacturers or suppliers. To carry out the procedure in every detail requires reference to annexes and appendices of ASTM D 2699-12. The annexes detail the specific equipment and instrumentation required, the critical component settings and adjustments, and include the working tables of referenced settings. The appendices provide background and additional insight about auxiliary equipment, operational techniques and the concepts relative to proper maintenance of the engine and instrumentation items.

The accumulated motor fuel data relating to knock characteristics determined in many countries has, for many years, been based on the use of the CFR engine and the ASTM octane test methods. Accepted worldwide, petroleum industry octane number requirements for motor fuels are defined by the research method and associated CFR F-1 Octane Rating Unit¹⁾, which emphasizes the need for this method and test equipment to be standardized. The initiation of studies to use a different engine for ISO purposes has therefore been considered an unnecessary duplication of effort.

For these reasons, it has been considered desirable by ISO Technical Committee 28, *Petroleum products and lubricants*, to adopt the ASTM D 2699 standard procedures. However, this International Standard refers to annexes and appendices of ASTM D 2699 without change because of their extensive detail. These annexes and appendices are not included in this International Standard because they are available from ASTM International.

Due to identified component obsolescence issues, the original, analogue control panel has been replaced by the manufacturer by new digital panel as of 2011. Service parts availability for the analogue system will be phased out in the future. Research work was executed by ASTM International^[5] to check whether there was statistically observable systemic bias between the 501C and the new digital knock measurement system.

With respect to precision ISO and ASTM technical committees concluded that there was numerically comparable precision for repeatability between the 501C and new panel knock measurement systems, and no statistically observable difference for reproducibility between the 501C and new panel knock measurement systems. This means that the new CFR octane panel could be included in the test method.

1) The sole manufacturer of the Model CFR F-1 Octane Rating Unit is Waukesha Engine, Dresser Waukesha, Inc., 1000 West St. Paul Avenue, Waukesha, WI 53188, USA.

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WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard establishes the rating of liquid spark-ignition engine fuel in terms of an arbitrary scale of octane numbers using a standard single-cylinder, four-stroke cycle, variable compression ratio, carburetted, CFR engine operated at constant speed. Research octane number (RON) provides a measure of the knock characteristics of motor fuels in automotive engines under mild conditions of operation.

This International Standard is applicable for the entire scale range from 0 RON to 120 RON, but the working range is 40 RON to 120 RON. Typical motor fuel testing is in the range of 88 RON to 101 RON.

This International Standard is applicable for oxygenate-containing fuels containing up to 4,0 % (m/m) oxygen and for gasoline containing up to 25 % (V/V) ethanol.

NOTE 1 Although 25 % (V/V) of ethanol corresponds to approximately 9 % (m/m) oxygen, full applicability of this test method for that oxygen range has only been checked for gasoline type of fuels.

NOTE 2 Work is under way to check the possibility to use the method up to and including 85 % (V/V) ethanol.

NOTE 3 This International Standard specifies operating conditions in SI units but engine measurements may be specified in inch-pound units because these were the units used in the manufacture of the equipment, and thus some references in this International Standard include these units in parenthesis.

NOTE 4 For the purposes of this standard, the terms “% (m/m)” and “% (V/V)” are used to represent the mass fraction, μ , and the volume fraction, φ , of a material respectively.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

ASTM D2699-12, *Standard Test Method for Research Octane Number of Spark-Ignition Engine Fuel*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
accepted reference value
ARV

value that serves as an agreed-upon reference for comparison, and which is derived as: a theoretical or established value, based on scientific principles, an assigned or certified value, based on experimental work of some national or international organization, or a consensus or certified value, based on collaborative experimental work under the auspices of a scientific or engineering group

3.2
check fuel

fuel of selected characteristics that has a RON accepted reference value determined by round-robin testing by multiple engines in different locations

3.3
cylinder height

relative vertical position of the CFR engine cylinder with respect to the piston at top dead center (t.d.c.) or the top machined surface of the crankcase

3.4
dial indicator reading

numerical indication of cylinder height, indexed to a basic setting when the engine is motored with the compression ratio set to produce a specified compression pressure

Note 1 to entry: The dial indicator reading is expressed in thousandths of an inch.

3.5
digital counter reading

numerical indication of cylinder height, indexed to a basic setting when the engine is motored with the compression ratio set to produce a specified compression pressure

3.6
detonation meter

knock signal conditioning instrumentation that accepts the electrical signal from the detonation pickup and produces an output signal for display

Note 1 to entry: The meter is either analogue or digital.

3.7
detonation pickup

magnetostrictive-type transducer that threads into the engine cylinder to sense combustion-chamber pressure and provide an electrical signal proportional to the rate-of-change of that cylinder pressure

3.8
firing

engine operation with fuel and ignition

3.9
fuel-air ratio for maximum knock intensity

proportion of fuel to air that produces the highest knock intensity for each fuel

3.10
guide table

tabulation of the specific relationship between cylinder height and octane number for the CFR engine operated at standard knock intensity and a specified barometric pressure

3.11
knock

abnormal combustion, often producing an audible sound, caused by auto-ignition of the air-fuel mixture

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3.12**knock intensity**

measure of engine knock

3.13**knockmeter**

indicating meter with a division scale that displays the knock intensity signal from the detonation meter

Note 1 to entry: The meter is either analogue or digital.

3.14**motoring**

engine operation without fuel and with the ignition shut off

3.15**research octane number****RON**

numerical rating of knock resistance for a fuel obtained by comparing its knock intensity with that of primary reference fuels of known research octane number when tested in a standardized CFR engine operating under conditions specified in this document

3.16**oxygenate**

oxygen-containing organic compound, such as various alcohols or ethers, used as a fuel or fuel supplement

3.17**primary reference fuel****PRF**

2,2,4-trimethylpentane (iso-octane), *n*-heptane, volumetrically proportioned mixtures of iso-octane with *n*-heptane, or blends of tetraethyl lead in iso-octane, which define the octane number scale

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3.18**spread**

sensitivity of the detonation meter expressed in knockmeter divisions per octane number

3.19**toluene standardization fuel****TSF**

volumetrically proportioned blend that has RON accepted reference value and specified rating tolerances

4 Principle

A sample fuel, operating in a CFR engine at the fuel-air ratio that maximizes its knock, is compared to primary reference fuel blends to determine that blend which, when operated at the fuel-air ratio that maximizes its knock, would result in both fuels producing the same standard knock intensity when tested at the same engine compression ratio. The volumetric composition of the primary reference fuel blend defines both its octane number and that of the sample fuel.

5 Reagents and reference materials

5.1 Cylinder-jacket coolant, consisting of water conforming to grade 3 of ISO 3696. Water shall be used in the cylinder jacket for laboratory locations where the resultant boiling temperature is $100\text{ °C} \pm 1,5\text{ °C}$ ($212\text{ °F} \pm 3\text{ °F}$). Water with commercial glycol-based antifreeze added in sufficient quantity to meet the boiling temperature requirement shall be used when laboratory altitude dictates.

A commercial multi-functional water treatment material should be used in the coolant to minimize corrosion and mineral scale that can alter heat transfer and rating results.

5.2 Carburettor coolant, if required (see 8.29), consisting of water or a water-antifreeze mixture, chilled sufficiently to prevent fuel bubbling and excessive vaporization, but neither colder than 0,6 °C nor warmer than 10 °C.

5.3 Engine crankcase-lubricating oil, comprising an SAE 30 viscosity grade oil meeting service classification API SF/CE or better.

It shall contain a detergent additive and have a kinematic viscosity of 9,3 mm²/s to 12,5 mm²/s at 100 °C (212 °F) and a viscosity index of not less than 85. Oils containing viscosity index improvers shall not be used. Multi-grade lubricating oils shall not be used.

5.4 2,2,4-trimethylpentane (iso-octane) primary reference fuel, of minimum purity 99,75 % (V/V), containing no more than 0,10 % (V/V) heptane and no more than 0,5 mg/l lead. This material shall be designated as 100 RON¹⁾.

WARNING — iso-Octane is flammable and its vapours are harmful. Vapours may cause flash fire.

5.5 n-Heptane primary reference fuel, of minimum purity 99,75 % (V/V), containing no more than 0,10 % (V/V) isooctane and no more than 0,5 mg/l lead. This material shall be designated as 0 RON²⁾.

WARNING — n-heptane is flammable and its vapours are harmful. Vapours may cause flash fire.

5.6 80-octane primary reference fuel blend, prepared using reference fuel grade iso-octane (5.4) and n-heptane (5.5); this blend shall contain 80 % (V/V) ± 0,1 % (V/V) iso-octane.

NOTE ASTM D 2699-12, Annex A3 (Reference Fuel Blending Tables), provides information for preparation of primary reference fuel blends to specific RON values.

5.7 Tetraethyl lead, dilute, (TEL dilute volume basis), consisting of a solution of aviation mix tetraethyl lead antiknock compound in a hydrocarbon diluent of 70 % (V/V) xylene and 30 % (V/V) n-heptane.

WARNING — Tetraethyl lead is poisonous and flammable. It may be harmful or fatal if inhaled, swallowed, or absorbed through the skin. May cause flash fire.

The anti-knock compound shall contain 18,23 % (m/m) ± 0,05 % (m/m) tetraethyl lead and have a relative density at 15,6 °C/15,6 °C (60 °F/60 °F) of 0,957 to 0,967.

NOTE 1 The typical composition of the compound, excluding the tetraethyl lead, is as follows:

Ethylene dibromide (scavenger):	10,6 % (m/m)
Diluent:	
xylene	52,5 % (m/m)
heptane	17,8 % (m/m)
Dye, antioxidant and inerts	0,87 % (m/m)

NOTE 2 Developments within ISO are under way in order to make less use of lead-containing PRFs.

5.8 Primary reference fuel blends for ratings over 100 RON, prepared by adding dilute tetraethyl lead (5.7), in millilitre quantities, to a 400 ml volume of iso-octane (5.4). These blends define the RON scale above 100.

1) PRFSs are commercially available, currently from Chevron Phillips Chemical Company LP, 1301 McKinney, Suite 2130, Houston, TX 77010-3030, USA or Haltermann Products—Werk Hamburg, Zweigniederlassung der DOW Olefinverbund GmbH, Schlengendeich 17, 21107 Hamburg, Germany.

NOTE ASTM D 2699–12, Annex A3 (Reference Fuel Blending Tables), provides information on the RON values for blends of tetraethyl lead in isoctane.

5.9 Methylbenzene (toluene), reference fuel grade, with a minimum purity of 99,5 % (V/V) as determined by chromatographic analysis, a peroxide number not exceeding 5 mg/kg and a water content not exceeding 200 mg/kg.

Antioxidant treatment should be added by the supplier at a rate suitable for long term stability as empirically determined with the assistance of the antioxidant supplier.

5.10 Check fuels, consisting of in-house typical spark-ignition engine fuels having RON accepted reference values, low volatility and good long-term stability.

6 Apparatus

6.1 Test engine assembly, a CFR octane rating unit consisting of a single-cylinder engine consisting of a standard crankcase, a cylinder/clamping sleeve assembly to provide continuously variable compression ratio adjustable with the engine operating, thermal-siphon recirculating jacket cooling system, a multiple fuel tank system with selector valving to deliver fuel through a single jet passage and carburettor venturi, an intake air system with controlled temperature and humidity equipment, electrical controls, and a suitable exhaust pipe.

The engine flywheel shall be connected by a belt to a special electric power-absorption motor that acts as a motor driver to start the engine and as a means to absorb power at constant speed when combustion is occurring (engine firing).

NOTE Test engine assembly is available from the single source manufacturer, GE Waukesha gas engine, Dresser, Inc., 1000 West St. Paul Avenue, Waukesha, WI 53188, USA. This information is given for the convenience of users of this International Standard but does not constitute an endorsement by ISO of this product.

6.2 Instrumentation, consisting of electronic detonation metering instrumentation, including a detonation pickup and knockmeter to measure and display the intensity of combustion knock, as well as conventional hardware, tubing, fasteners, electrical and electronic items.

NOTE Instrumentation is available from multiple sources. In some cases, selection of specific dimensions or specification criteria are important to achieve proper conditions for the knock testing unit, and these are included in ASTM D2699–12, Appendix X1 when applicable.

6.3 Reference and standardization fuel dispensing equipment, consisting of calibrated burettes or volumetric ware having a capacity of 200 ml to 500 ml and a maximum volumetric tolerance of $\pm 0,2$ %.

Calibration shall be verified in accordance with ISO 4787. Burettes shall be outfitted with a delivery valve and delivery tip to accurately control dispensed volumes. The delivery tip shall be of such size and design that shut-off tip discharge does not exceed 0,5 ml. The rate of delivery from the dispensing system shall not exceed 400 ml/min. The installation shall be in such a manner and be supplied with fluids such that all components of each batch or blend are dispensed at the same temperature.

6.4 Gravimetric blending of reference fuels, use of blending systems that allow preparation of the volumetrically-defined blends by gravimetric (mass) measurements based on the density of the individual components is also permitted, provided the system meets the requirement for maximum 0.2 % blending tolerance limits.

Calculate the mass equivalents of the volumetrically-defined blend components from the densities of the individual components at 15,56°C (60°F).

6.5 Tetraethyl lead (TEL) dispensing equipment, consisting of a calibrated burette, pipette assembly, or other liquid-dispensing apparatus, having a capacity not exceeding 4,0 ml, and a critically controlled tolerance for dispensing dilute TEL into 400 ml batches of iso-octane.

Calibration shall be verified in accordance with ISO 4787.

NOTE ASTM D 2699-12, Appendix X2 (Volumetric Reference Fuel Blending Apparatus and Procedures), provides additional information for application.

6.6 Special maintenance tools, consisting of a number of speciality tools and measuring instruments available for easy, convenient and effective maintenance of the engine and testing equipment.

NOTE Lists and descriptions of these tools and instruments are available from the manufacturers of the engine equipment and those organizations offering engineering and service support for this International Standard.

7 Sampling and sample preparation

7.1 Unless otherwise specified in the commodity specification, samples shall be taken as described in ISO 3170 or ISO 3171 and/or in accordance with the requirements of national regulations for the sampling of the product under test, or an equivalent national standard.

7.2 Cool samples to 2 °C to 10 °C (35 °F to 50 °F) in the container in which they are received and before the container is opened.

7.3 Minimize the sample's exposure to light before pouring it into the engine carburettor fuel bowl, because of possible sensitivity to light that can affect fuel characteristics. Collect and store samples in an opaque container.

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8 Basic engine and instrument settings and standard operating conditions

8.1 Installation of engine equipment and instrumentation

Locate the octane test engine in an area where it will not be affected by certain gases and fumes that may have a measurable effect on the RON test result (see [Clause 1](#)).

Installation of the engine and instrumentation requires placement of the engine on a suitable foundation and hook-up of all utilities. Engineering and technical support for this function is required, and the user shall be responsible for complying with all local and national codes and installation requirements. Proper operation of the CFR engine requires assembly of a number of engine components and adjustment of a series of engine variables to prescribed specifications. Some of these settings are established by component specifications, others are established at the time of engine assembly or after overhaul, and still others are engine running conditions that must be observed or determined by the operator during the testing process. [Annex A](#) gives further information on the test variable characteristics.

8.2 Engine speed

Engine speed shall be 600 r/min \pm 6 r/min when the engine is operating with combustion with a maximum variation of 6 r/min occurring during a rating.

Engine speed when combustion is occurring shall not be more than 3 r/min greater than for motoring without combustion.