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BUREAU OF INDIAN STANDARDS

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भारतीय मानक मसौदा
पेट्रोलियम और इसके उत्पाद – परीक्षण की विधियाँ
भाग 87 तरल पेट्रोलियम उत्पादों का स्वतः प्रज्वलन तापमान

Draft Indian Standard
PETROLEUM AND ITS PRODUCTS – METHODS OF TEST
PART 87 AUTOIGNITION TEMPERATURE OF LIQUID PETROLEUM PRODUCTS

[*First Revision* of IS 1448 Part 87]
(ICS 75. 080)

Methods of Sampling and Test for Petroleum and related Products of Natural or Synthetic Origin (excluding bitumen) Sectional Committee PCD 01	Last date for receipt of comment is **12 November 2024**

FOREWORD

(*Formal clauses will be added later*)

This standard was originally published in 1979 which was adapted from ASTM D 2155-66 "Standard Test Method for Determination of Fire Resistance of Aircraft Hydraulic Fluids by Autoignition Temperature" and ISO Standard 3988 "Liquid petroleum products — Determination of autoignition temperature"

The first revision has been taken up to align with revised ASTM D 2155-18 and to keep pace with the latest technological developments and international practices

In this revision following major changes have been made:

a) Scope has been modified.
b) Significance of autoignition incorporated.
c) Thermocouples have been modified

In this preparation of this standard, considerable assistance has been derived from the following standards:

ASTM D 2155-18 — Standard Test Method for Determination of Fire Resistance of Aircraft Hydraulic Fluids by Autoignition Temperature

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2: 2022 'Rules for rounding off numerical values (*second revision*)'.

1 SCOPE

1.1 This standard prescribes a method for the determination of the autoignition temperature of a liquid or semi-liquid petroleum product in air at atmospheric pressure using hypodermic syringe injection into a flask maintained at the test temperature.

1.2 This standard is not designed for evaluating materials which are capable of exothermic decomposition. For such materials, ignition is dependent upon the thermal and kinetic properties of the decomposition, the mass of the sample, and the heat transfer characteristics of the system.

1.3 This standard is not designed for evaluating for solid chemicals which melt and vaporize or which readily sublime at the test temperature.

1.4 This standard is not designed to measure the autoignition temperature of samples which are solids or liquids at the test temperature. Such materials will thermally degrade in the flask and the accumulated degradation products may ignite.

1.5 This standard is not designed to measure the autoignition temperature of chemicals that are gaseous at atmospheric temperature and pressure.

2 DEFINITION

2.1 Autoignition — The ignition of a material commonly in air as the result of heat liberation due to the exothermic oxidation reaction in the absence of an external ignition source such as a spark or flame.

2.2 Autoignition Temperature — It is defined as the minimum temperature at which spontaneous ignition of a petroleum product occurs in the absence of a flame, under standardized conditions.

3 OUTLINE OF THE METHOD

3.1 A small measured sample of the product to be tested is injected with a hypodermic syringe into a heated 200 ml conical borosilicate flask containing air. The contents of the flask are observed in a darkened room for 5 min following injection of the sample or until autoignition occurs, autoignition is shown by the sudden appearance of a flame inside the flask. The lowest flask temperature at which autoignition occurs for a series of prescribed sample volumes is taken to be the autoignition temperature of the petroleum product in air at atmospheric pressure.

4 SIGNIFICANCE

4.1 Autoignition is dependent on the chemical and physical properties of the sample and the method and apparatus used for its determination. The autoignition temperature by a given method does not necessarily represent the minimum temperature at which a given material will self-ignite in air. The volume of the vessel used is particularly important since lower

autoignition temperatures will be achieved in larger vessels. Vessel material can also be an important factor.

4.2 The Autoignition temperature determined by this test method are those at which air oxidation leads to ignition. These temperatures can be expected to vary with the test pressure and oxygen concentration.

5 APPARATUS

5.1 The apparatus, shown schematically in Fig. 1 to Fig. 6 shall conform to the requirements prescribed in **5.2** to **5.6.**

5.2 Furnace

Consisting of an approximately 127 mm internal diameter alundum cylinder, approximately 127 mm long, circumferentially wound with an electric heater, compressed asbestos-cement board cover, ring neck heater and flask guide ring, three neck heater supports, base heater, and suitable refractory insulating material and retaining shell. Temperature control shall be achieved by the use of suitable auto-transformers or rheostats, thermocouples and a suitable potentiometer.

5.3 Hypodermic Syringe — 0.25 ml or 1 ml capacity, graduated at every 0.01 ml and equipped with a 50 mm long stainless-steel needle of 0.15 mm maximum bore diameter, to inject the sample into the heated test flask.

5.4 Test Flask — 200 ml capacity, conical flask made of borosilicate glass. A new flask shall be used for tests on each product.

5.5 Thermocouples — Three calibrated iron/constantan thermocouples to be used for determining the flask temperature. These shall be mounted in the furnace so as to contact the exterior wall of the flask 25 mm to 50 mm below the bottom of the neck heater and under the base of the flask near its centre.

5.6 Timer — An electric timer or stopwatch calibrated in 0.1s or 0.2s intervals shall be used to determine the time lag before ignition.

> NOTE — Time lag is the time interval between the instant of sample injection and that of ignition as shown by the appearance of the flame.

6 PROCEDURE

6.1 Temperature Control — Adjust the temperature of the furnace so that the temperatures at the top, centre, and bottom of the test flask (*see* **5.4**) are within 1 °C of the desired test temperature.

6.2 Sample Injection — Inject 0.07 ml of the sample to be tested into the test flask with the hypodermic syringe (*see* **5.3**) and quickly withdraw the syringe (*see* Note).

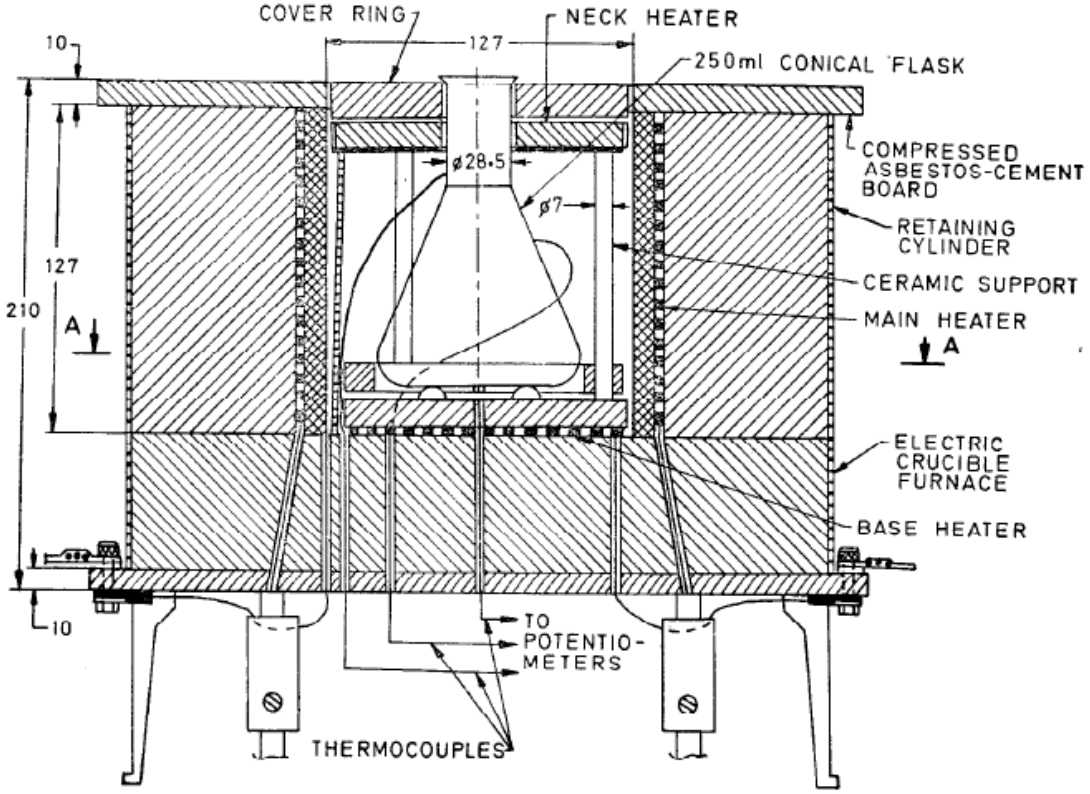
> NOTE — A new flask shall be used for tests on each product, should the flask become visibly coated with residue before the completion of tests on a product, the final series of tests shall be conducted with a new flask.

6.3 Time Measurement — Start the timer (*see* **5.6**) as the sample is injected into the test flask.

6.4 Observations — Observe the inside of the test flask in a darkened room by means of a mirror placed at an appropriate angle above the flask. If a flame is not observed in 5 min, the volume of the sample tested is considered non-flammable at the temperature of the test flask. Completely flush the flask with clean dry air and stop the timer. Then repeat the test at a higher temperature (about 30 °C). Allow at least 15 min to elapse between tests. If a flame is observed, stop the timer and record the time interval between the sample injection and the appearance of the flame to the nearest 0.2 s as the time lag. Lower the test temperature by 3 °C and repeat the entire procedure until autoignition is no longer obtained. Then raise the test temperature by about 30 °C and repeat the above procedure using 0.10 ml of the sample. If the lowest temperature at which autoignition is obtained with this quantity of sample (0.10 ml) is lower than that found in the previous test, repeat the procedure again using 0.12 ml, then 0.15 ml, etc., of the sample in 0.02 ml to 0.03 ml steps until the minimum autoignition temperature is obtained. If the lowest temperature at which autoignition is obtained with 0.10 ml of the sample is greater than that obtained with 0.07 ml of the sample, repeat the above procedure with 0.05 ml then 0.03 ml instead of 0.12 ml, 0.15 ml, etc.

6.5 Autoignition — Autoignition is usually shown in these tests by the appearance of a yellow or blue flame. However, pale blue, white, red, or mixed colour flames may be obtained in some cases.

6.6 Data — Record the test temperature, atmospheric pressure, quantity of sample used, and time lag before ignition. A plot of the ignition temperature against time lag before ignition may be used to determine the autoignition temperature, if desired.



All dimensions in millimeters.

FIG. 1 TEST APPARATUS ASSEMBLY

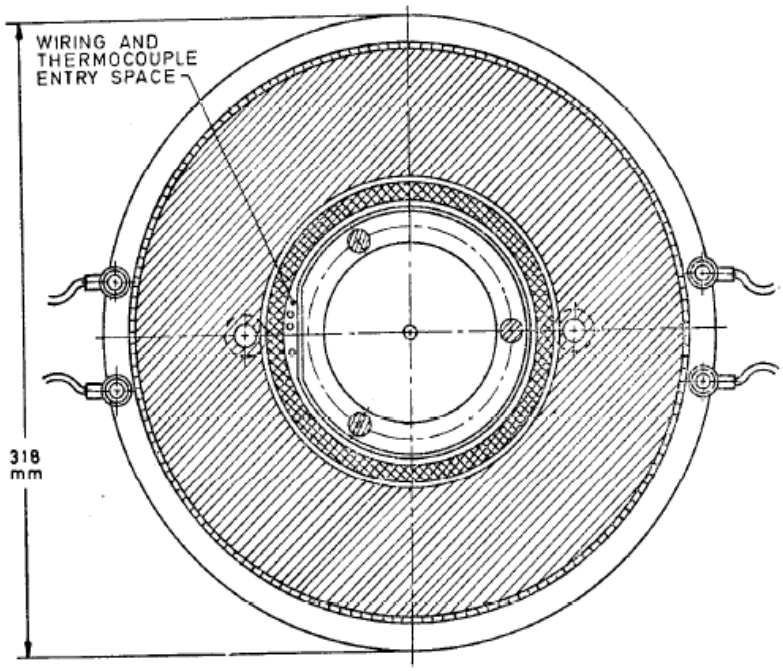
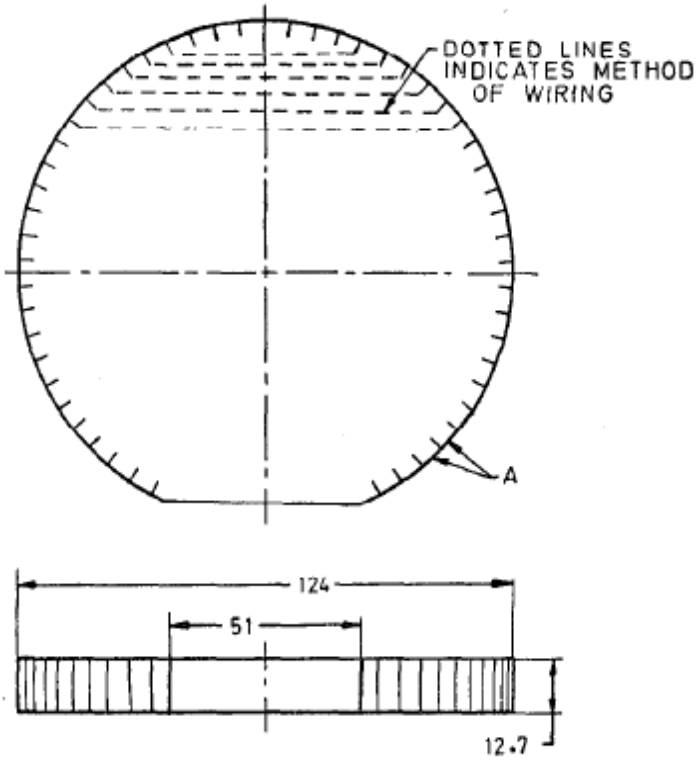


FIG. 2 SECTION AA (FLASK OMITTED)

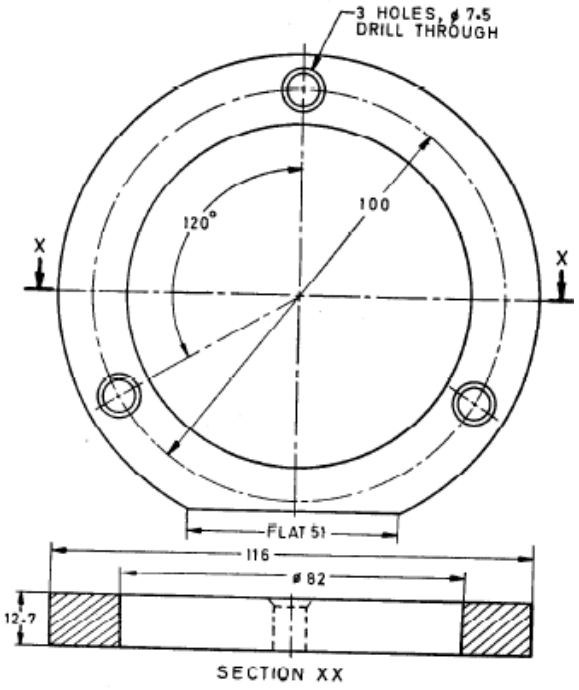


All dimensions in millimeters.

FIG. 3 BASE HEATER (COMPRESSED ASBESTOS-CEMENT BOARD)

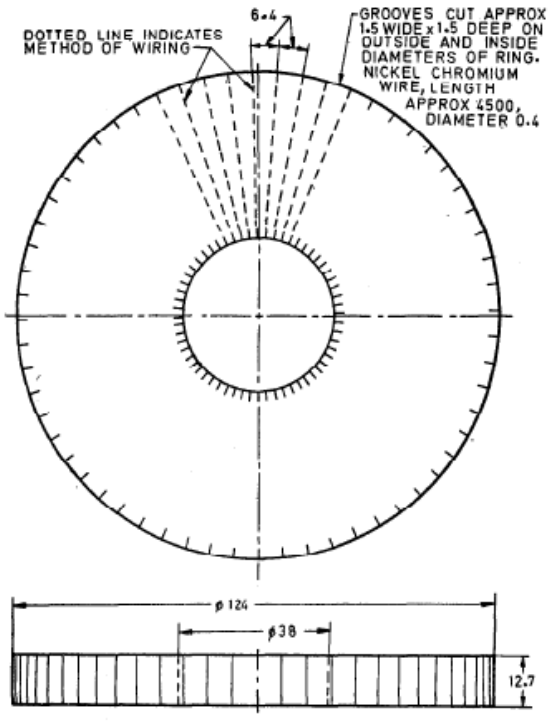
A = Grooves cut approximately 1.5 wide × 1.5 deep on outside diameter of disc. Nickel-chromium wire, length approximately 2 500, diameter 0.4

Note — The diameter of the nickel-chromium wire is based on a 110 V electricity supply. The diameter should be adjusted to suit the supply voltage, or a transformer used to convert the supply at the terminals to 110 V.



All dimensions in millimeters.

FIG. 4 FLASK GUIDE RING (COMPRESSED ASBESTOS-CEMENT BOARD)



All dimensions in millimeters.

FIG. 5 NECK HEATER (COMPRESSED ASBESTOS-CEMENT BOARD)

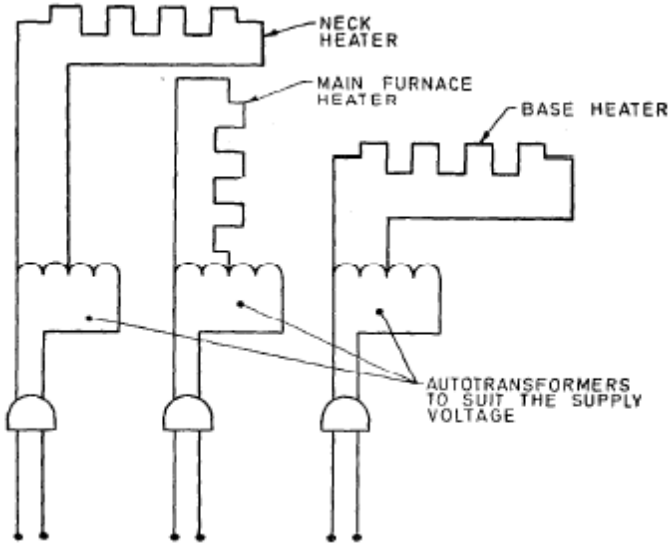


FIG. 6 FURNACE HEATER (SCHEMATIC WIRING DIAGRAM)

7 REPORT

7.1 Report the autoignition temperature as the lowest flask temperature at which autoignition was obtained, rounded to the nearest 3 °C. Report the corresponding time lag before ignition and barometric pressure as the time lag and pressure, respectively.

8 PRECISION

8.1 Results of duplicate tests shall not differ by more than the following amounts:

Temperature	*Repeatability*	*Reproducibility*
Below 316 °C	3	10
Above 316 °C	5	20