CLOUD AND POUR POINT APPARATUS

WORKING – INSTRUCTIONS FOR CLOUD AND POUR POINT APPARATUS AS PER IP-15/16 AND IS 1448 (PART.i) 1970 (P-10)

SCOPE: These methods determine the cloud and pour point of petroleum products. The Method for cloud point (section 1) is intended for use only on oils which are transparent in layers 40mm in thickness and have cloud point below 49°C. The method for pour point (section II) is intended for use on any petroleum oil.

TERMINOLOGY: For the purpose of these methods, the following definitions shall apply.

CLOUD POINT: The temperature, expressed to the nearest degree Centigrade, at which a cloud or haze appears when the oil is cooled under prescribed conditions.

POUR POINT: The lowest temperature, expressed as a multiple of 30°C, at which the oil is observed to flow when cooled and examined under prescribed conditions.

OUTLINE OF METHODS: In the determination of the cloud point, the sample is cooled under prescribed conditions and is inspected at intervals of one-degree centigrade until a cloud or haze appears. In the determination of the pour point, the sample is cooled under prescribed conditions and is inspected at intervals of 3°C until it will no longer move when the plane of its surface is...
held vertical for 5 seconds. The pour point is then taken as 3°C above the temperature of cessation of flow.

SECTION .I CLOUD POINT

PROCEDURE: Bring the sample to a temperature of at least 15°C above the approximate cloud point and pour it into the jar to a height of 51 to 57 mm. Close the jar with the cork so that the thermometer bulb rest on the center of the bottom of the jar. Fit the gasket on to the jar 25 mm from the bottom and insert the jar into the jacket. The disc, the gasket, and jacket shall be kept clean and dry.

Maintain the bath temperature at minus 1°C to 2°C. Support the jacket and jar in a vertical position in the bath so that not more than 25 mm projects from the cooling medium. At each thermometer reading of one degree centigrade, remove the jar from the jacket quickly but without disturbing the oil, inspect the material for cloud, and replace the jar; this complete operation shall not take more than 3 second. If the sample does not show a cloud when it has been cooled 10°C. Place the jar and jacket in another bath maintained at a temperature of minus 15°C to minus 18°C. If the sample does not show a cloud when it has been cooled to minus 7°C place the jar and jacket in another bath maintained at a temperature of minus 32°C to minus 35°C. When an inspection of the sample first reveals a distinct cloudiness or haze at the bottom of the jar, record the reading of the thermometer as the cloud point.

REPORTING: Report the temperature recorded in corrected for thermometer errors if necessary, and express to the nearest degree Centigrade, as the cloud point.

PRECISION: RESULTS OF DUPLICATE TESTS SHALL NOT DIFFER BY MORE THAN THE FOLLOWINGS :-

REPEATABILITY 6°C

REPRODUCIBILITY 6°C

SECTION .II POUR POINT

PROCEDURE: Pour the sample, heated in a water bath if necessary, into the jar to a height of 51 to 57 mm. Close the jar with the cork-carrying thermometer No.1 so that the thermometer bulb is immersed vertically in the
sample with the beginning of the capillary 3mm below the surface. Heat the sample, without stirring, to a temperature of 46°C in a bath maintained at a temperature not higher than 48°C Cool in the sample to 32°C in air or in a water bath at approximately 25°C. If a pour point below minus 35°C is expected, cool the sample in air or in water bath to 16°C and replace the thermometer No.1 by thermometer No.2 Fit the gasket on to the jar 25 MM from the bottom and insert the jar into the jacket. The disc, the gasket, and the jacket shall be kept clean and dry.

If a pour point above 32°C is expected, heat the sample to 46°C or to a temperature 8°C above the expected pour point, and fit the jar into the jacket immediately. One the sample has cooled enough to allow the formation of wax crystals, take great care not to disturb the mass of sample nor to permit the thermometer to shift in the sample. Any disturbance of the spongy network of crystals will lead to false results. Maintain the bath temperature at minus 1°C to plus 2°C. Support the jacket and jar in a vertical position in the bath so that not more than 25mm projects form the cooling medium. Beginning at a temperature 12°C above the expected pour point, at each thermometer reading which is a multiple of 3°C, remove the jar from the jacket carefully, and tilt it just enough to see whether the oil will move and the replace it, this complete operation shall not take more than 3 seconds. If the oil has not ceased to flow when it has been cooled to 9°C, place the jar and jacket in another bath maintained at a temperature of minus 32°C to minus 35°C. If the pour point is very low, maintain additional baths with successively lower temperature differentials of about 18°C, transfer in the jar and jacket when the temperature of the sample reaches a point 27°C above the temperature of the new bath. As soon as the sample ceases to flow when the jar is tilted, hold the jar in horizontal position for exactly 5 seconds. If the sample shows any movement replace the jar in the jacket and cool down the sample another 3°C. If the oil shows no movement during the 5 second, record the reading of the thermometer.

Certain lubricating oils tend to move as a whole. The point at which they cease to flow is consequently more difficult to detect. For black oil, cylinder stock, and non-distillate fuel the result obtained by the procedure described in 9.1 and 9.2 is the upper (maximum) pour point. Determine the lower (Minimum) Pour Point by heating the sample with stirring to 105°C, pouring it into the jar, cooling it to 32°C as before.

**CALCULATION AND REPORTING :** Add 3°C to the temperature recorded above and corrected for thermometer errors if necessary, and report the result as the pour point. For black oil, cylinder stock etc report both the upper and lower pour point.

**PRECISION :** RESULTS OF DUPLICATE TESTS SHALL NOT DIFFER BY MORE THAN THE FOLLOWING AMOUNTS.

- **REPEATABILITY** 6°C
- **REPRODUCIBILITY** 6°C