PTFE BASED POLYMER LUBRICANT:TESTING OF CHARACTERSTIC PARAMETERS

SUBMITTED BY

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DECLARATION

This is to certify that the minor project entitled “PTFE based polymer lubricants:testing of characteristic parameters” which is submitted by yashovardhan mishra in the fulfillment of course unit for the award of the degree of master of technology in polymer technology to the department of applied chemistry and polymer technology,delhi technological university delhi 110042,is a bonafied record of the project work carried out by me under the supervision of dr. richa srivastava during IV semester.to the best of our knowledge and belief,this work has not been submitted to any other university or institutions for the award of any degree or diploma.

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(YASHOVARDHAN MISHRA)

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INTRODUCTION

A **lubricant** is a substance used to reduce friction between surfaces in contact, which reduces the heat generated when the surfaces move. It may also have the function of transmitting forces, transporting foreign particles, or heating or cooling the surfaces. The property of reducing friction called lubricity.

In addition to industrial applications, lubricants are used for other purposes. Other uses include cooking (oils and fats in use in frying pans, in baking to prevent food sticking), bio-medical applications on humans (e.g. lubricants for artificial joints), ultrasound examination, medical examinations, and the use ofpersonal lubricant for sexual purposes

PROPERTIES

A good lubricant should posses the following :

1.high boiling and freezing point

2. high index of viscosity

3.thermal and hydraulic stability

4.resistance for oxidation

5.demulsibility

6.corrosion resistance

PTFE(POLYTETRAFLUOROETHYLENE)

Polytetrafluoroethylene (PTFE) is a synthetic fluoropolymer of tetrafluoroethylene that has numerous applications. The best known brand name of PTFE-based formulas is Teflon by Chemours.[2] Chemours is a spin-off of DuPont Co.,[3] which discovered the compound in 1938.[2]

PTFE is a fluorocarbon solid, as it is a high-molecular-weight compound consisting wholly of carbon and fluorine. PTFE is hydrophobic: neither water nor water-containing substances wet PTFE, as fluorocarbons demonstrate mitigated London dispersion forces due to the high electronegativity of fluorine. PTFE has one of the lowest coefficients of friction of any solid.

PTFE is used as a non-stick coating for pans and other cookware. It is very non-reactive, partly because of the strength of carbon–fluorine bonds, and so it is often used in containers and pipework for reactive and corrosive chemicals. Where used as a lubricant, PTFE reduces friction, wear and energy consumption of machinery. It is commonly used as a graft material in surgical interventions. Also, it is frequently employed as coating oncatheters; this interferes with the ability of bacteria and other infectious agents to adhere to catheters and causehospital-acquired infections

PROPERTIES

PTFE is a thermoplastic polymer, which is a white solid at room temperature, with a density of about 2200 kg/m^3. According to DuPont, its melting point is 600 K (327 °C; 620 °F).[19] It maintains high strength, toughness and self-lubrication at low temperatures down to 5 K (−268.15 °C; −450.67 °F), and good flexibility at temperatures above 194 K (−79 °C; −110 °F).[20] PTFE gains its properties from the aggregate effect of carbon-fluorine bonds, as do all fluorocarbons. The only chemicals known to affect these carbon-fluorine bonds are highly reactive metals like the alkali metals, and at higher temperatures also such metals as aluminium and magnesium, and fluorinating agents such asxenon difluoride and cobalt(III) fluoride

Property	Value
Density	2200 kg/m^3
Melting point	600 K
Thermal expansion	112–125 · 10^{-6} K^{-1} [22]
Thermal diffusivity	0.124 mm^2/s [23]
Young's modulus	0.5 GPa

Yield strength	23 MPa
Bulk resistivity	10^{16} Ω·m [24]
Coefficient of friction	0.05–0.10
Dielectric constant	ε = 2.1, tan(δ) < 5(-4)
Dielectric constant (60 Hz)	ε = 2.1, tan(δ) < 2(-4)
Dielectric strength (1 MHz)	60 MV/m

APPLICATION OF PTFE

The major application of PTFE, consuming about 50% of production, is for wiring in aerospace and computer applications (e.g. hookup wire, coaxial cables). This application exploits the fact that PTFE has excellent dielectric properties. This is especially true at high radio frequencies, making it suitable for use as aninsulator in cables and connector assemblies and as a material for printed circuit boards used at microwave frequencies. Combined with its high melting temperature, this makes it the material of choice as a high-performance substitute for the weaker and lower-melting-point polyethylene commonly used in low-cost applications.

In industrial applications, owing to its low friction, PTFE is used for applications where sliding action of parts is needed: plain bearings, gears, slide plates, etc. In these applications, it performs significantly better than nylon and acetal; it is comparable to ultra-high-molecular-weight polyethylene (UHMWPE). Although UHMWPE is more resistant to wear than PTFE, for these applications, versions of PTFE with mineral oil or molybdenum disulfide embedded as additionallubricants in its matrix are being manufactured. Its extremely high bulk resistivity makes it an ideal material for fabricating long-life electrets, useful devices that are the electrostatic analogues of magnets

TESTING OF CHARACTERISTIC PARAMETERS

SAMPLE: DIESEL OIL 10 ml , lubricant (NULON E25) 2 ml

PARAMETERS TESTED:

1.DENSITY

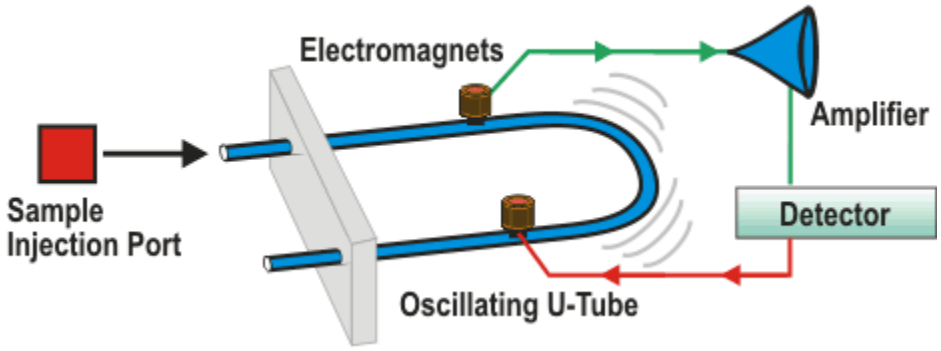
METHOD: ASTM D5002 osillating U tube density meter

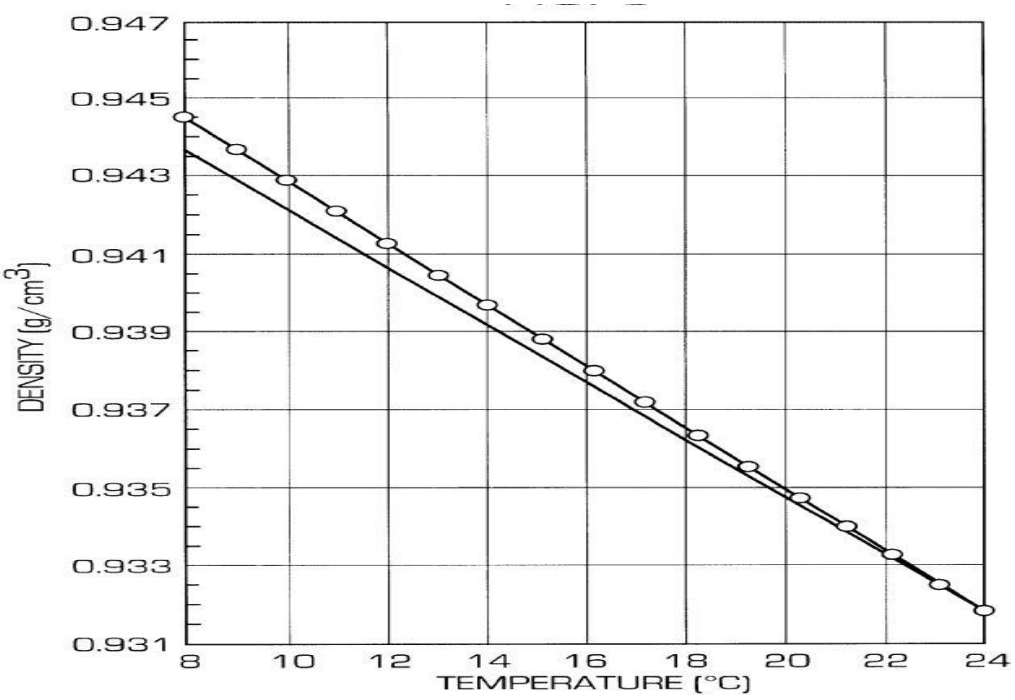
SAMPLE: DIESEL OIL 10ml,LUBRICANT 2ml

PRINCIPLE: The **oscillating U-tube** is a technique to determine the density of liquids and gases based on an electronic measurement of the frequency of oscillation, from which the density value is calculated. This measuring principle is based on the Mass-Spring Model.

The sample is filled into a container with oscillation capacity. The eigenfrequency of this container is influenced by the sample's mass. This container with oscillation capacity is a hollow, U-shaped glass tube (oscillating U-tube) which is electronically excited into undamped oscillation (at the lowest possible amplitude). The two branches of the U-shaped oscillator function as its spring elements.

The direction of oscillation is normal to the level of the two branches. The oscillator's eigenfrequency is only influenced by the part of the sample that is actually involved in the oscillation. The volume involved in the oscillation is limited by the stationary oscillation knots at the bearing points of the oscillator. If the oscillator is at least filled up to its bearing points, the same precisely defined volume always participates in the oscillation, thus the measured value of the sample's masscan be used to calculate its density.





RESULT: DENSITY 930 kg/mm3 at 24 degree celsius

2.KINEMATIC VISCOSITY

TEST METHOD: IS:1448 (P-25)1976

PRINCIPLE:

The time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured Ilow time and the calibration constant of the viscometer.

Apparartus:

1.Viscometers - Calibrated, glass capillary type, capable of measuring kinematic viscosity within the limits
2. Automated assemblies which perform as herein required are considered suitable alternatives.
3. Viscometer Holders - To enable the viscometer to be suspended in a similar position as when it was calibrated. The proper alignment of vertical parts may be confirmed by using a plumb line.

4.Viscometer Thermostat and Bath - Any transparent liquid or vapour bath may be used, provided that it is of sufficient depth so that at no time during the measurement will any portion of the sample in the viscometcr be less than 2 cm below the surface of the bath liquid or less than 2 cm above the bottom of the bath.

5.timing device

6.temperature measuring device

PROCEDURE

1. Maintain the bath at the test temperature within the limits. Apply, the necessary corrections, if any, to all thermometer readings.
2. Ascertain that the ice-point of the thermometer has been determined recently and the corrections, **if** any, applied to the calibration values. The possible change in the ice-point reading of new thermometers may require a check every week.
3. Select a clean dry, calibrated viscometer having a range covering the estimated viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time should not be less than 200 seconds.
4. When the temperature of the test is below the dew point, fit loosely packed drying tubes on to the open ends of theviscometer to prevent water condensation. Drying tubes shall fit the design of the viscometer and not restrict the~flowof the sample under test by pressure created in the instrument. At temperatures below OX, it may be advisable to charge the sample into the viscometer at ambient temperature; allow the viscometer to cool to bath temperature, keeping sample in the working capillary to prevent slight accumulation of frost on the walls of the capillary.

5. Viscometers used for silicone fluids, fluoro-carbons, and other liquids which are difficult to r9move by the use of a cleaning agent, should be reserved for the exclusive use of those fluids except when calibrating. Such viscometers should be subjected to calibration checks at frequent intervals

6. Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. Should the sample contain solid particles, filter during charging through a 75-micron IS Sieve.

7. With certain products which exhibit ‘ gel-like ’ behaviour, take care that measurements are made at sufficiently high temperatures for such materials to flow freely so that similar results will be obtained in viscome&s of different capillary diameters.

8. The viscosity of steam refined cylinder oils, black lubricating oils, residual fuel oils, and similar waxy products can beaffected by the previous thermal history. The following preheating procedure should be followed to obtain uniform results for viscosities below 95°C.

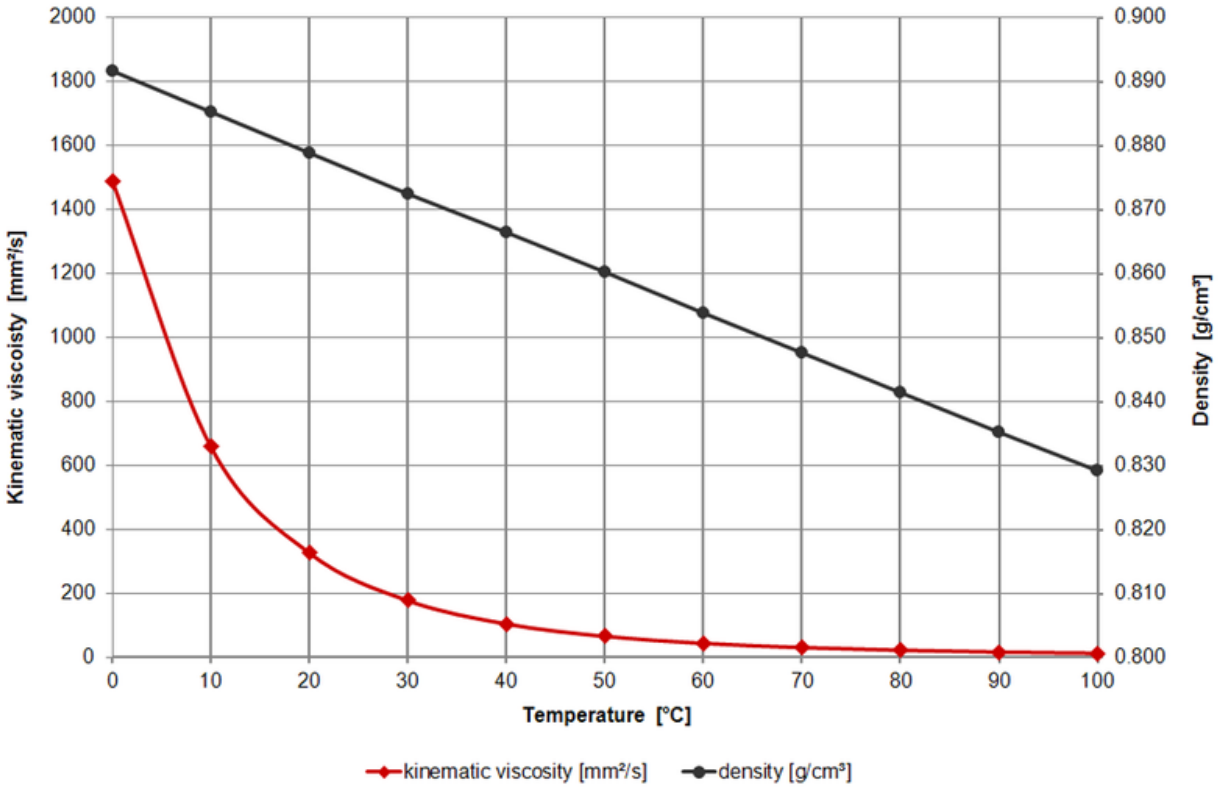
9. To obtain a representative sample, heat in the original container to about 50°C with stirring and shaking. Probe the bottom of the container with a rod to be certain that all waxy materials are in solution. Pour 100 ml into a 125-ml flask. Stopper loosely with a cork or rubber stopper. Immerse the flask in a bath of boiling water for 30 * minutes. Mix well, remove the sample from the bath, and strain it through a 75-micron IS sieve directly into the viscometer already in the thermostated bath. Complete the viscosity test within 1 hour after preheating.

10. Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Because this time will vary for the different instruments and for different temperatures, establish a safe temperature equilibrium time by trial (30 minutes should be sufficient). Where design of the viscometer requires it, adjust the volume of the test sample after the sample has reached temperature equilibrium. One bath is often used to accommodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring a flow time

11. Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 5 mm ahead of the first timing mark. With the sample flowing freely, measure in seconds, to within O-2 seconds the time required for the meniscus to pass from the first timing mark to the second. If this flow time is less than the specified minimum select a viscometer with a capillary of smaller diameter and repeat the operation.

12. For modified Ostwald and suspended level types, repeat the procedure described in -6.5 to make a second measurement of the flow time. For reverse-flow viscometers, use the same or another unit and begin at 6.3 to make the second measurement.

13. If two measurements agree within 0.2 percent, use the average for calculating the reported kinematic viscosity. time should agree within~0*35 percent. For reverse-flow types, flow If these agreements axe not obtained, reject the test results.



RESULT: kinematic viscosity is as depicted above for different temperatures

3. 3.CLOUD POINT

TEST METHOD : IS 1448(P-10)SEC/2012

PRINCIPLE: cloud point refers to the temperature below which wax in diesel or biowax in biodiesels form a cloudy appearance. The presence of solidified waxes thickens the oil and clogs fuel filters and injectors in engines. The wax also accumulates on cold surfaces (e.g. pipeline or heat exchangerfouling) and forms an emulsion with water. Therefore, cloud point indicates the tendency of the oil to plug filters or small orifices at cold operating temperatures

Apparatus

1. Test jar, cylindrical, of clear glass, flatbottomed, 33,2 mm to 34,8 mm in outside diameter and 115 mm to 125 mm in height.The inside diameter of the jar may range from 30 mm to 32,4 mm, within the constraint that the wall thickness be no greater than I,6 mm. The jar shall be marked with a line to indicate a sample height 54 mm +- 3 mm above the inside bottom.
2. Thermometers, partial-immersion type
3. Cork, to fit the test jar, bored centrally to take the test thermometer.
4.Jacket, watertight, cylindrical, metal, flatbottomed, about 115 mm in depth, with an inside diameter of 44,2 mm to 45,8 mm. It shall be supported in a vertical position in a cooling bath so

that not more than 25 mm projects out of the cooling medium, and it shall be capable of being cleaned.
5. Disc, of cork or felt, 6 mm in thickness, to fit loosely inside the jacket.
6 Gasket, ring form, about 5 mm in thickness, to fit snugly on the outside of the test jar and loosely inside the jacket. This gasket may be made of rubber, leather or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape. The purpose of the ring gasket is to prevent the test jar from
touching the jacket.

7. Cooling baths, of a type suitable for obtaining the required temperatures.The size and shape of the baths are not specified, but a support to hold the jackets firmly in a vertical position is essential. The bath temperature shall be monitored by means of a high or low cloud and pour thermometer immersed to the correct immersion depth.
For the determination of cloud points below IO OC, two or more baths are needed. The required bath temperatures m;iy be maintained by refrigeration or by suitable freezing mixtures.

NOTE 1 The freezing mixtures commonly used are as follows:
For cloud-point temperatures down to
10 “C: ice and water
- 12 *C: crushed ice and sodium chloride crystals
-- 26 *C: crushed ice and calcium chloride crystals
- 57 “C: solid carbon dioxide and acetone or petroleum naphtha.
The CO,-based mixture may be made as follows: In a covered metal beaker, chill a suitable amount of acetone or petroleum naphtha to - 12 *C, or lower, by means of an ice/salt mixture. Then add enough solid carbon dioxide to the chilled acetone or petroleum naphtha to give the desired temperature. Solid carbon dioxide is commercially available in many areas.

Procedure

1.Bring the sample to be tested to a temperature at least 14 OC above the approximate cloud point, but not above 49 “C. Remove any moisture present by any suitable method, such as filtration through dry lintless filter paper, until the sample is perfectly clear, working at a temperature of at least 14 OC above the approximate cloud point, but not above 49 “C.
2 . Pour the clear sample into the test jar to the level mark.
3. Close the test jar tightly by the cork carrying the appropriate test thermometer . Use the high cloud and pour thermometer if the expected cloud point is at or above - 36 OC and the low cloud and pour thermometer if the expected cloud point is below - 36 OC. Adjust the position of the cork and the thermometer so that the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is resting on the bottom of the jar. Liquid-column separation of thermometers occasionally occurs and may escape detection. Thermometers shall therefore be checked immediately prior to the test and used only if the ice point is 0 OC + 1 OC, measured with the thermometer immersed to the immersion line in an ice bath and with the emergent-stem

temperature not differing significantly from 21 OC. Alternatively, immerse the thermometer to the reading level and correct for the resultant lower stem temperature.
4. Ensure that the disc , the gasket and the inside of the jacket (4.4) are clean and dry. Place the disc in the bottom of the jacket. The disc and jacket shall have been placed in the cooling medium a minimum of 10 minutes before the test jar is inserted. Place the gasket round the test jar, 25 mm from the bottom. Insert the test jar in the jacket. Never place a jar directly into the cooling medium.

NOTES: The use of a jacket cover while the empty jacket is cooling is permitted.
Failure to keep the disc, the gasket and the inside of the jacket clean and dry may lead to frost formation which may cause erroneous results.

5. Maintain the temperature of the cooling bath at - 1 OCto + 2 “C.
6 . At each test thermometer reading that is a multiple of 1 OC, remove the test jar from the jacket quickly but without disturbing the sample, inspect for cloud, and replace in the jacket. Ensure that this complete operation takes no more than 3 s. If the sample does not show a cloud when it has been cooled to 10 OC, transfer the test jar to a jacket in a second bath maintained at a temperature of - 18 “C to - 15 OC (see table2). Do not transfer the jacket. If the sample does not show a cloud when it has been cooled to - 7 OC, transfer the test jar to a jacket in a third bath maintained at a temperature of - 35 OCto - 32 “C.

7. Report as the cloud point the temperature, to the nearest 1 OC, at which any cloud is observed at the bottom of the test jar, which is confirmed by continued cooling. The wax cloud or haze is always noted first at the bottom of the test jar, where the temperature is lowest. A slight haze throughout the entire sample, which slowly becomes more apparent as the temperature is lowered, is usually due to traces of water in the sample. Generally, this water haze will not interfere with the determination of the wax cloud point. in most cases of interference, fiNration through dry lintiess filter paper such as described in is sufficient. In the case of diesel fuels, however, if the haze is very dense, a fresh portion of the sample shall be dried by shaking 100 mI with 5 g of anhydrous sodium sulfate for at least 5 min and then filtering through dry iintiess filter paper. Given sufficient contact time, this procedure will remove or sufficiently reduce the water haze so that the wax cloud can be readily discerned. Drying and filtering shall always be carried out at a temperature at least 14 OC above the approximate cloud point, but not in excess of 49 “C

RESULT: CLOUD POINT -15 DEGREE CELSIUS

4.CARBON RESIDUE PERCENTAGE

TEST METHOD : IS:1448(P-8)1967

PRINCIPLE: 1.The test Portion is weighed into a glass coking bulb having a capillary opening, and is placed in a metal furnace maintained at a temperature of approximately 550 “C. The test Portion is thus quickly heated to the Point at which all volatile matter is evaporated out of the bulb with or without decomPosition, while the heavier residue remaining in the bulb undergoes cracking and coking reactions. In the later stages of the heating period, the coke or carbon residue is subject to further slow. decomposition or slight Oxidation due to the possibility of air being drawn into the bulb. After a specified heating period, the bulb is removed from the furnace, cooled in a dessicator, and again weighed. The residue remaining is calculated as a mass percentage of the test Portion.
2. Provision is made for determining the proper operating characteristics of the furnace with a control bulb containing a thermocouple, which gives a specified time-temperature relationship.

For light products where over 90 % (VW) distils at below 370 “C, the procedure may be carried out on the residue remaining after 90 % (VW) has been distilled.

APPARATUS: 1. Glass cokinb bulb, made of heat-resistant glass, and conforming to the dimensions and tolerances Prior to use, check the diameter of the capillary to see that it is greater than 1,5 mm and not more than 2,0 mm. Pass a 1,5 mm diameter drill rod through the capillary and into the bulb; attempt to pass a 2,0 mm diameter drill rod through the capillary. Reject bulbs that do not permit the insertion of the smaller rod and those whose capillaries are larger than the larger rod.
2. Control bulb, for use in determining compliance of furnace characteristics with the Performance requirements . Made of stainless steel, containing a thermocouple and conforming to the tolerances shown in figure 2. The control bulb shall be provided with a dull finish by heating in a furnace at a temperature of 850 “C to 900 “C for 30 min, and shall not be polished thereafter, as a polished bulb has different heating characteristics from one with a dull finish.

3. Syringe, for test Portion charging. A 5 ml or IO ml glass hypodermic Syringe fitted with a needle of 1,5 mm outside diameter or a Serum needle of 1,45 mm to 1,47 mm outside diameter, for transfer of the test Portion to the glass coking bulb.
NOTE 6 A Syringe having a needle which fits on the ground glass tip of the Syringe is not recommended, as it may be blown off when pressure is applied to the Syringe plunger. Locking-type syringes are more satisfactory, as the needle locks on the bottom of the Syringe barrel, and cannot be blown off by pressure.
4. Metal coking furnace, constructed of solid metal, having coking bulb wells 25,45 mm + 0,l mm in internal diameter and 76 mm deep to the centre of the well bottom, with suitable arrangements for heating to a uniform temperature of 550 “C. The bottom of the well shall be hemispherical to accommodate the bottom of the glass coking bulb .
DO not cast or otherwise form the furnace with unnecessary voids which will impede heat transfer.
If a molten metal furnace is used, provide it with a suitable number of bulb wells, the internal

dimensions of which correspond to the internal dimensions of holes in the solid metal furnace. The bulb wells shall be immersed in the molten metal to leave not more than 3 mm of the bulb weil exposed above the molten metal at operating temperatures.

5 Temperature-measuring devices. A removable iron-constantan thermocouple with a sensitive pyrometer, or other suitable temperature-indicating device shall be used, located centrally near the bottom Portion of the furnace and arranged to measure the temperature of the furnace, thus enabling the performante tests specified in clause 5 to be carried out.

NOTES

8 When a molten bath is used, it is desirable to protect the temperature-indicating device with a quartz or thin metal sheath.

9 lt is good practice to calibrate the thermocouple or other temperature-measuring device against a Standard thermocouple or a reference Standard approximately once a week when the furnace is in constant use, the actual frequency depending on experience.

PROCEDURE:

Place a new glass coking bulb in the coking furnace (4.4) at 550 “C for approximately 20 min to decompose any foreign organic matter and to remove water. DO not re-use a glass coking bulb, as unpredictable results tan be obtained in such cases. Place the bulb in a closed desiccator over Calcium chloride or silica gel for 20 min to 30 min and then weigh to the nearest 0,l mg.

NOTE 11

For routine testing, new bulbs may be used without pre-ignition provided that they are visibly free from particles or other contamination. Shake thoroughly the oil Sample to be tested, first warming if necessary to reduce its viscosity. Strain the Sample through a 150 Pm wire sieve. By means of the hypodermic Syringe or the device, introduce into the coking bulb an amount of Sample . Ensure that no oil remains on the exterior surface or on the inside of the neck of the bulb. Reweigh the bulb and contents to the nearest 0,l mg

If difficulty is encountered in loading very viscous or asphaltic test portions, of whatever size, into the glass coking bulb, the apparatus shown in may be used. When obtaining the test Portion of oils containing Sediment (for example, used oils), it is important to make the transfer of test portions in the shortest possible time to avoid Segregation of the Sediment. Samples containing Sediment which settles quickly after stirring tan be placed in the coking bulbs more expeditiously by using an arrangement . This device consists of a three-way 2 mm stopcock to which have been fused two lengths of capillary tubing (1,5 mm to 2,0 mm internal diameter). Connect the third leg of the stopcock to a vacuum line by means of pressure tubing. Secure the glass coking bulb to the short arm of capillary tubing by a 25 mm length of hose, taking care that the capillary of the glass bulb is butted up against the capillary tubing. Immerse the long end of the capillary tubing in the Sample. After evacuating the coking bulb, manipulate the stopcock to Cause the stirred Sample to flow freely into the bulb through the two lengths of capillaty tubing. Use tubing with the same internal diameter as that of the neck of the coking bulb to prevent accumulation of any Sediment during transfer.

3. Place the coking bulb in a “Standard performante well” with the furnace at the checking temperature (see note 14) and allow to remain for 20 min + 2 min. Remove the bulb with metal tongs, the tips of which have just been heated. Reproduce the furnace and bulb condition used when standardizing that bulb well . If there is appreciable loss of oil from frothing, discard the test and repeat the determination using a smaller test Portion

NOTES

1. When carrying out a test, it is important to adhere rigorously to the temperature conditions Chosen for checking Performance of apparatus for example, if the bath was at a temperature of 553 “C & 1 “C when inserting the control bulb , then it is necessary to use similar temperature conditions in the coking test. When maintained in normal Operation, the temperature of an electrically heated furnace with automatic controls will generally fluctuate within a specific temperature range. Therefore, when making a coking test, it is generally important that the test bulbs be inserted when the furnace is at the same temperature and at the same Position in the temperature cycle as it was when the inspection test was started, unless it has been proved that the temperature variations are insignificant.
2. Frothing may be due to water which tan be removed by heating gently in a vacuum and flushing out the vapour with nitrogen Prior to filling the bulb.
3. After removal, cool the bulb in a desiccator under the same conditions (including time for weighing) used before filling the bulb . When removing the bulb from the desiccator, examine to make Sure there are no foreign particles adhering to the bulbs; if any are found, as black particles sometimes are on the capillary neck, brush them off with a piece of sized Paper or a camel hair brush. Weigh to the nearest 0,l mg. Discard the used glass coking bulb.

NOTE

In studies of oil characteristics, useful information tan often be obtained from a simple visual examination of the coking bulb after the test. Thus, significance tan be attached to noting, in the results, such findings as whether coke more or less fills the bulb; whether liquid material is present, either as limpid residue or drops; whether the residue is not black and flaky, but is coloured and powdery (presumably from the presence of inorganic material)

RESULT: CARBON RESIDUE PERCENTAGE 2.37%

5.ASH CONTENT PERCENTAGE

TEST METHOD: IS:1448(P-4)1984

ASH: The inorganic residue left after the ignition of the sample under prescribed conditions, calculated as the percentage by mass of the original sample.

PRINCIPLE: The sample contained in a suitable vessel is ignited and allowed to burn until only ash and carbon remain. The carbonaceous residue is reduced to an ash by heating in a muffle furnace at 7759, cooled, and weighed

APPARATUS:1. Evaporating Dish or Crucible - made of platinum, silica, or porcelain, of 90 to 120-ml capacity.

Electric Mufle Furnace - capable of maintaining a temperature of 775°C f 25:C, and preferably having suitable apertures at the front and rear so as to allow a slow natural draught of air to pass through

PROCEDURE: Heat the evaporating dish or crucible at 700°C to 800°C for 10 minutes or more. Cool to room temperature in a suitable container and weigh to the nearest 0.1 mg.

NOTE- 'The container in which the dish or crucible is cooled should not contain a desiccating agent. In addition, all weighings of the crucibles should be performed as soon as the crucibles have cooled. If it should be necessary that the crucibles remain in the desiccator for a longer period, then all subsequent'weighings should be made after allowing the crucibles and contents to remain in the desiccator for the same length of time.

2. The quantity of sample to be taken depends upon the ash content of the material. Weigh into the dish or crucible sufficient sample (up to a maximum of 100 g) to give up to 20 mg of ash. For sample mass which require more than one filling of the dish, obtain the mass from the difference between the initial and final mass of a suitable sample container. Weigh the sample to the nearest 0.1 percent. Heat the dish or crucible and sample until the contents become capable of being ignited with a flame. Maintain at such a temperature that the sample continues to burn at a uniform and moderate rate, leaving only ash and carbon when the burning ceases. The sample may contain water, which can cause spattering. The operator should heat the sample cautiously in a hood while wearing safety goggles.

NOTE- If the sample contains sufficient moisture to cause foaming and loss ol material, discard the sample and to an additional sample add 1 to 2 ml of 99 percc nt iso-propyl alcohol before heating. If this is not satisfactory, add 10 ml of an equivolume mixture of toluene and iso-propyl alcohol and mix thoroughly. Place several strips of ashless filter paper in the mixture and heat; when the paper begins *to* **bum, the greater part of the water is found to have been removed.**

3 Heat the residue in the muffle furnace at 775 f 25°C until all carbonaceous material has disappeared. Cool the dish to room temperature in a suitable container as prescribed, and weigh to the nearest O-1 mg.
4. Reheat the dish at 775°C for 20 to 30 minutes, cool in a suitable container as prescribed and reweigh. Repeat the heating and weighing until consecutive weighings differ by not more than 0.5 mg.

RESULT: ASH CONTENT 0.42%

6.FLASH POINT

TEST METHOD: IS:1448(P-69)1969

flash point
lowest temperature of the test portion, corrected to a barometric pressure of 101,3 kPa, at which application of a test flame causes the vapour of the test portion to ignite and the flame to propagate across the surface of the liquid, under the specified conditions of test.

Principle
The test test cup is filled to a specified level with the test portion. The temperature of the test portion is increased
rapidly at first and then at a slow constant rate as the flash point is approached. At specified temperature intervals,
a small test flame is passed across the test cup. The lowest temperature at which application of the test flame
causes the vapour above the surface of the liquid to ignite is taken as the flash point at ambient barometric
temperature. To determine the fire point, the test is continued until the application of the test flame causes the
vapour above the test portion to ignite and burn for at least 5 s. The flash point and fire point obtained at ambient
barometric pressure are corrected to standard atmospheric pressure using an equation.

Reagents and materials
Cleaning solvent, for removal of traces of sample from the test cup and cover. NOTE The choice of solvent will depend upon the previous material tested, and the tenacity of the residue. Low volatility aromatic (benzene free) solvents may be used to remove traces of oil, and mixed solvents such as toluene-acetone-methanol
may be efficacious for the removal of gum-type deposits.

Verification liquids

Steel wool, any grade capable of removing carbon deposits without damage to the test cup.

Apparatus

Cleveland open cup apparatus, as specified in annex A.

If automated equipment is used, ensure that it has been established that the results obtained are within the precision of this International Standard and that the test cup and test flame applicator conform to the dimensional and mechanical requirements specified in annex A. If automated testers are used, the user shall ensure that all the manufacturer's instructions for adjusting and operating the instrument are followed.

In cases of dispute, the flash point as determined manually shall be considered the referee test.

Shield, approximately 460 mm square and 610 mm high, and having an open front.

Thermometer, of the partial immersion type, conforming to the specification given in annex B.

NOTE Other types of temperature-measuring devices may be used, provided that they meet the requirements for accuracy and have the same reponse as the thermometers specified in annex B.

Barometer, accurate to 0,1 kPa. Barometers pre-corrected to give sea level readings, such as those used at weather stations and airports, shall not be used.

Preparation of apparatus

Location of apparatus

Place the apparatus (6.1) on a level and steady surface in a draught-free room (see notes 1 and 2 below). Shield the top of the apparatus from strong light by any suitable means, to permit detection of the flash point.

NOTE 1 When draughts cannot be avoided, it is good practice to surround the apparatus with a shield.

NOTE 2 When testing samples which produce toxic vapours, the apparatus may be located in a fume hood with an individual control of air flow, adjusted so that vapours can be withdrawn without causing air currents over the test cup

Cleaning the test cup

Wash the test cup with an appropriate solvent (5.1) to remove any traces of gum or residue remaining from a previous test. Dry the test cup using a stream of clean air to ensure complete removal of the solvent used. If any deposits of carbon are present, remove them by rubbing with steel wool .

Preparing the test cup

Before use, cool the test cup to at least 56 °C below the expected flash point.

Assembly of apparatus

Support the thermometer in a vertical position with the bottom of the bulb 6 mm from the bottom of the test cup, and located at a point halfway between the centre and side of the test cup on a

diameter perpendicular to the arc (or line) of the sweep of the test flame, and on the side opposite to the test flame applicator.

NOTE

The immersion line engraved on the thermometer will be 2 mm below the level of the rim of the test cup when the thermometer is properly positioned. An alternative method is to gently lower the thermometer until it contacts the bottom of the test cup, and then raise it 6 mm.

Verification of apparatus

Verify the correct functioning of the apparatus at least once a year by testing a certified reference material The result obtained shall be equal to or less than $R / 2$ from the certified value of the CRM, where R is the reproducibility of the method. It is recommended that more frequent verification checks are made using secondary working standards (SWSs)

NOTE A recommended procedure for apparatus verification using CRMs and SWSs, and the production of SWSs

The numerical values obtained during the verification check shall not be used to provide a bias statement, nor shall they be used to make any correction to the flash points subsequently determined using the apparatus.

Sampling

1 Unless otherwise specified, obtain samples for analysis in accordance with the procedures given in ISO 3170, ISO 3171 or an equivalent National Standard.

2 Place samples in tightly sealed containers, appropriate to the material being sampled, and for safety purposes, ensure that the sample container is only filled to between 85 % to 95 % of its capacity.

3 Store the samples in conditions to minimize vapour loss and pressure build-up. Avoid storing the samples at temperatures in excess of 30 °C.

Sample handling

1 Subsampling

Subsample at a temperature at least 56 °C below the expected flash point. If an aliquot of the original sample is to be stored prior to testing, ensure that the container is filled to more than 50 % of its capacity.

2 Samples containing undissolved water

If a sample contains undissolved water, decant an aliquot from the water prior to mixing.
NOTE Flash point results can be affected by the presence of water.

3 Samples that are liquid at ambient temperature

Mix samples by gently shaking by hand prior to the removal of the test portion, taking care to minimize the loss of volatile components, and proceed .

4 Samples that are semi-solid or solid at ambient temperature

Heat the sample in its container in a heating bath or oven at a temperature not exceeding 56 °C below the expected flash point. Avoid overheating the sample as this could lead to the loss of

volatile components. After gentle agitation, proceed .

Procedure for determining flash point

1 The results of flash point determinations may be affected if the sample volume falls below 50 % of the container capacity.

2 Record the ambient barometric pressure using a barometer (6.4) in the vicinity of the apparatus at the time of test.

NOTE It is not considered necessary to correct the barometric pressure reading to 0 °C, although some barometers are designed to make this correction automatically.

3 Fill the test cup at ambient or elevated temperature (see 9.4) so that the top of the meniscus is exactly at the filling line. If too much sample has been added to the test cup, remove the excess using a pipette or other suitable device; however, if there is sample on the outside of the apparatus, empty, clean and refill it. Destroy or remove any air bubbles or foam on the surface of the sample whilst maintaining the correct level of test portion in the test cup. If a foam persists in the final stages of the test, discard the result.

4 Light the test flame and adjust it to a diameter between 3,2 mm and 4,8 mm, the size of the comparison bead if one is mounted on the apparatus.

5 Apply heat initially so that the rate of temperature rise of the test portion is 14 °C/min to 17 °C/min. When the test portion temperature is approximately 56 °C below the expected flash point, decrease the heat so that the rate of temperature rise for the last (23 5) °C before the expected flash point is 5 °C/min to 6 °C/min. During the test, take care to avoid disturbing the vapours in the test cup by careless movements or breathing near the test cup .

6 Starting at least (23 5) °C below the exptected flash point, apply the test flame when the temperature read on the thermometer (6.3) reaches each successive 2 °C mark. With a smooth continuous motion, taking approximately 1 s, pass the test flame in one direction across the centre of the test cup, at right angles to the diameter which passes through the thermometer, either in a straight line or along the circumference of a circle having a radius of at least 150 mm. The centre of the test flame shall move in a horizontal plane not more than 2 mm above the plane of the upper edge of the test cup. For the next test flame application, pass the flame in the opposite direction. If a skin forms over the test portion, carefully move it aside and continue the determination.

7 Record as the observed flash point, the temperature of the test portion, read on the thermometer, when application of the test flame causes the vapour of the test portion to ignite and propagate across the surface of the liquid. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame.

8 When the temperature at which the flash point is observed is less than 18 °C from the temperature of the first application of the test flame, the result is not valid. Repeat the test using a fresh test portion, adjusting the temperature of the first application of the test flame until a valid determination is obtained when the flash point is 18 °C above the temperature of the first application of the test flame.

RESULT: FLASH POINT IS 235 DEGREE CELSIUS

7.SULPHUR CONTENT PERCENTAGE

TEST METHOD: IS:1448(P-4)1984

PRINCIPLE: The sample is ignited and burned until only ash and carbon remain. After cooling, the residue is treated with sulphuric acid and heated at 775°C until oxidation of carbon is complete. The ash is then cooled,. re-treated with sulphuric acid, and heated at 775°C to constant mass.

Significance and Use - The sulphated ash may be used to indicate the concentration of known metal-containing additives in new oils. When phosphorus is absent, barium calcium, magnesium, sodium and potassium are converted to their sulphates and tin (stannic) and zinc to their oxides . Sulphur and chlorine do not interfere but when phosphorus is present with metals, it remains partially or wholly in the sulphated ash as metal phosphates.

APPARATUS: ***Dish -*** An evaporating dish or crucible made of porcelain, fused silica, or platinum of 50 to IOO-ml capacity. For samples yielding less than O-02 percent sulphated ash, a platinum evaporating dish or crucible of 120- to 150-ml capacity is specified, except for samples containing elements injurious to platinum.

NOTE - A **platinum vessel should not be used if the sample is likely to contain elements, such ar phosphorus, which attack platin~lrn under the conditions of** the test.

Furnace - furnace shall be capable of maintaining a temperature of **775°C f 25°C** and preferaljly have apertures at the front and rear to allow a slow natural draught of air to pass through the furnace.

Reagents

Low-Ash Mineral Oil - White **oil having a** sulphated ash lower than the limit capable of being determined by this method.

NOTE- Determine the sulphated ash of this oil by the procedure using 100 g of white oil weighed to the nearest 0.5 g in a 120 10 1.50 ml platinum **dish. Deduct the sulphuric acid blank.**

Sulfihuric Acid (Relative Density 1.84) - Concentrated sulphuric acid (HsSOh).

Sul~huric Acid (I : I) - Prepare by slowly adding one volume of concentrated sulphuric acid (relative density 1.84) to one volume of water.

CAUTION - Sulphuric acid is highly corrosive and has a high heat of hydration. Protective

clothing, including gloves and face mask, should be worn during manipulations involving this acid.

Procedure

1 Selectthe size of the evaporating dish or crucible according to the quantity of sample necessary.
2 Heat the evaporating dish or crucible at 775°C for at least 10 minutes. Cool to room temperature in a suitable container and weigh to the nearest 0.1 mg.

3. Do not take a quantity in excess of 80 g. In the case of lubricating oil additives yielding a sulphated ash of 2 percent or more, dilute the weighed sample in the dish with approximately 10 times its mass of low ash mineral oil.

NOTE - If the amount of sulphatcd ash found differs from the expected amount by more than a factor of two, repeat the analysis with a different mass of sample calculated from the first analysis**.**

4. Heat the dish or crucible and sample carefully until the contents can be ignited with a flame. Maintain at such a temperature that the sample continues to burn at a uniform and moderate rate. When burning ceases, continue to heat gently until no further smoke or fumes are evolved.

NOTE - If the sample contains sufficient moisture to cause foaming and loss of material from the dish, discard the sample, and to an additional sample add 1 to 2 ml of 99 percent iso-propyl alcohol before heating. If this is not satisfactory, add 10 ml of a mixture of equal volumes of toluene and iso-propyl alcohol and mix thoroughly. Place several strips of ashless filter paper in the mixture and heat; when the paper begins to burn, the greater part of the water will have been removed.

5 Allow the dish to cool to room temperature, then completely moisten the residue by the dropwise addition of sulphuric acid (relative density 1.81). Carefully heat the dish at a low temperature on a hot plate or over a gas burner, avoiding spattering, and continue heating until fumes are no longer evolved.
6 Place the dish in the furnace at 775°C f 25°C and continue heating until oxidation of tte carbon is complete or almost complete.
7 Allow the dish to cool to room temperature. Add three drops 01 water and ten drops of sulphuric acid (1 : 1). Move the dish so as to moisten the entire residue. Again heat the dish as .8 Again place the dish in the furnace at 775 f 2.5% and maintain at that temperature for 30 minutes. Cool the dish to room temperature in a suitable container
9 Weigh the dish and residue to the nearest 0.1 mg.
10 Repeat 8 and 9until two successive weighings differ by no more than 1.0 mg.

11 For samples expected to contain 0.02 percent or less of sulphated ash, determine a sulphuric

acid blank by adding 1 ml of the concentrated sulphuric acid to a tared platinum dish or crucible, heating until fumes are no longer evolved and then heating in the furnace at 775 f 25°C for 30 minutes. Cool the dish or crucible to room temperature in a suitable container and weigh to the nearest 0.1 mg. If any ash is found in th sulphuric acid, an adjustment to the mass of sulphated ash obtained is made by subtracting the mass of ash contributed by the sulphuric acid, determined from the total volume of sulphuric acid itsed and the mass of ash found for the 1 ml blank, from the total mass in grams of sulphated ash for the sample. Use this corrected mass m, in calculating the percent sulphated ash

RESULT: SULPHUR CONTENT 0.017 %

CONCLUSION

The advantage of synthetic lubricant over mineral based lubricant comes from the ability to synthesize selected molecular structure which are beneficial in lubrication.

This usually is only possible at some cost penalty and a synthetic product is only of value if it can more than repay the additional investment or provide some critical performance need which a mineral oil cannot.

Performance factors which can lead to overall cost savings are improved energy efficiency,reduced maintainence cost,improved reliability and safer operation.

THE BENEFITS OF USING POLYMER BASED LUBRICANT (NULON E25)ARE:

1.increases efficiency by reducing friction

2.improved mileage and power

3.reduces knocking in the engine

4.reduces engine noise

5.reduces maintainence cost.

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Ana Paula Pena Almeida, Ana Paula Lelis Rodrigues de Oliveira, Cynthia D'Ávila Carvalho Erbetta, Ricardo Geraldo de Sousa, Roberto Fernando de Souza Freitas, Maria Elisa Scarpelli Ribeiro e Silva*

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