

E29 White Petrolatum

Stage 2, version 2

Definition

White Petrolatum is a purified and wholly or nearly decolorized semi-solid mixture of hydrocarbons obtained from petroleum. It may contain a suitable antioxidant.

Package and storage— Preserve in well-closed containers, protected from light.

Labeling— Label to indicate the name and proportion of any added antioxidant. Label to state the drop point value of the product.

Identification

A. Infrared Absorption Spectrophotometry, obtained by spreading a thin film of melted test specimen between sodium chloride plates.

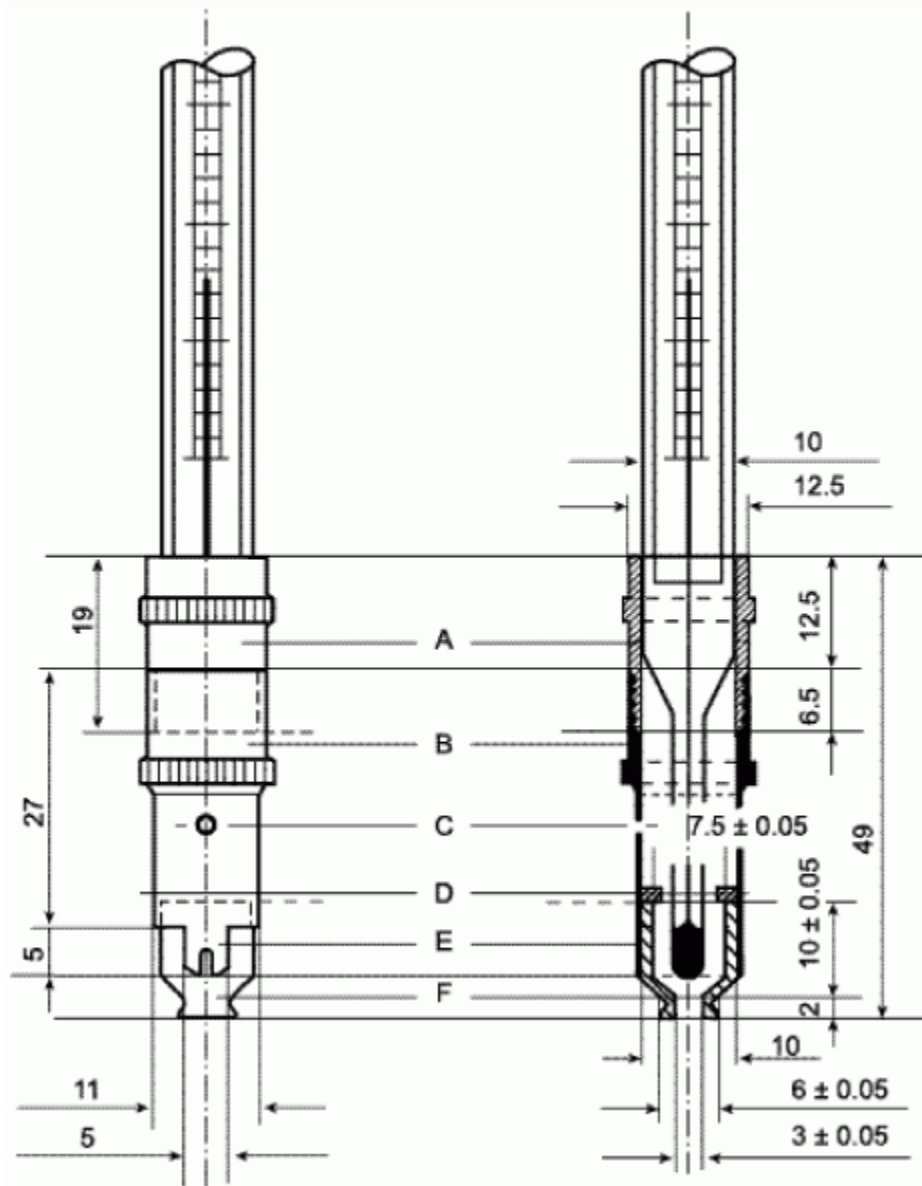
Acceptance criteria:

Record the infrared absorption spectrum and compare the spectrum with the Reference Spectrum or the spectrum of White Petrolatum Reference Standard: the transmission minima correspond in position and relative size.

B. Color— Melt about 10 g on a steam bath, and pour about 5 mL of the liquid into a clear-glass 15- × 150-mm test tube, keeping the White Petrolatum melted. The White Petrolatum is not darker than 5 mL of a mixture of Ferric Chloride Solution: 10 mg/mL hydrochloric acid (1:9) in a similar tube, the comparison of the two being made in reflected light against a white background, the White Petrolatum tube being held directly against the background at such an angle that there is no fluorescence.

C. Drop Point— *Apparatus*. The apparatus consists of 2 metal sheaths (*A* and *B*) screwed together. Sheath *A* is fixed to a mercury thermometer. A metal cup is loosely fixed to the lower part of sheath *B* by means of 2 tightening bands. Fixed supports 2 mm long determine the exact position of the cup, and in addition are used to centre the thermometer. A hole pierced in the wall of sheath *B* is used to balance the pressure. The draining surface of the cup must be flat and the edges of the outflow orifice must be at right angles to it. The lower part of the mercury thermometer has the form and size shown in the figure; it covers a range from 0 °C to 110 °C and on its scale a distance of 1 mm represents a difference of 1 °C. The mercury reservoir of the thermometer has a diameter of 3.5 ± 0.2 mm and a height of 6.0 ± 0.3 mm. The apparatus is placed in the axis of a test-tube about 200 mm long and with an external diameter of about 40 mm. It is fixed to the test-tube by means of a laterally grooved stopper through which the thermometer passes. The opening of the cup is placed about 15 mm from the bottom of the test-tube. The whole device is immersed in a beaker with a capacity of about 1 litre, filled with water. The bottom of the test-tube is placed about 25 mm from the bottom of the beaker. The water level reaches the upper part of sheath *A*. A stirrer is used to ensure that the temperature of the water remains uniform. Alternatively, a validated

44 automated method can be used.



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- | | |
|----------------------------|---------------------|
| A. upper metal sheath | D. fixed supports |
| B. lower metal sheath | E. tightening bands |
| C. pressure-balancing hole | F. metal sample cup |

46 *Figure 1. Apparatus for the determination of drop point*
 47 *Dimensions in millimeters*

48

49 Heat the substance to be examined at 100-105 °C for not more than 10 min, with stirring to ensure
 50 uniformity. Warm the metal cup at 100-105 °C in an oven, remove it from the oven, place on a
 51 clean plate or ceramic tile and pour a sufficient quantity of the melted sample into the cup to fill it
 52 completely. Allow the filled cup to cool for 30 min on the ceramic tile ($25 \pm 3^\circ\text{C}$) and place it in a

53 water-bath at 24-26 °C for a further 30-40 min. Level the surface of the sample with a single stroke
54 of a knife or razor blade, avoiding compression of the sample. Determine the drop point using a
55 starting temperature at least 10 °C below the expected drop point and increasing the temperature
56 at a rate of 1 °C/min.” Note the temperature at the fall of the first drop. Carry out at least
57 3 determinations, each time with a fresh sample of the substance. The difference between the
58 readings must not exceed 3 °C. The mean of 3 readings is the drop point of the substance. The
59 drop point is 35 °C to 70 °C and does not differ by more than 5 °C from the value stated on the
60 label

61

62 **Consistency**

63 *Apparatus*— Determine the consistency of White Petrolatum by means of a penetrometer fitted
64 with a polished cone-shaped metal plunger weighing 150 g, having a detachable steel tip of the
65 following dimensions: the tip of the cone has an angle of 30°, the point being truncated to a
66 diameter of 0.381 ± 0.025 mm, the base of the tip is 8.38 ± 0.05 mm in diameter, and the length of
67 the tip is 14.94 ± 0.05 mm. The remaining portion of the cone has an angle of 90°, is about 28 mm
68 in height, and has a maximum diameter at the base of about 65 mm. The containers for the test are
69 flat-bottom metal cylinders that are 100 ± 6 mm in diameter and not less than 65 mm in height.
70 They are constructed of at least 1.6-mm (16-gauge) metal, and are provided with well-fitting,
71 water-tight covers.

72 *Procedure*— Place the required number of containers in an oven, and bring them and a quantity of
73 White Petrolatum to a temperature of $82 \pm 2.5^\circ$, pour the White Petrolatum into one or more of
74 the containers, filling to within 6 mm of the rim. Cool to $25 \pm 2.5^\circ$ over a period of not less than
75 16 hours, protected from drafts. Two hours before the test, place the containers in a water bath at
76 $25 \pm 0.5^\circ$. If the room temperature is below 23.5° or above 26.5° , adjust the temperature of the
77 cone to $25 \pm 0.5^\circ$ by placing it in the water bath.

78 Without disturbing the surface of the substance under test, place the container on the penetrometer
79 table, and lower the cone until the tip just touches the top surface of the test substance at a spot 25
80 mm to 38 mm from the edge of the container. Adjust the zero setting and quickly release the
81 plunger, then hold it free for 5 seconds. Secure the plunger, and read the total penetration from the
82 scale. Make three or more trials, each so spaced that there is no overlapping of the areas of
83 penetration. Where the penetration exceeds 20 mm, use a separate container of the test substance
84 for each trial. Read the penetration to the nearest 0.1 mm. Calculate the average of the three or
85 more readings, and conduct further trials to a total of 10 if the individual results differ from the
86 average by more than $\pm 3\%$: the final average of the trials is not less than 10.0 mm and not more
87 than 30.0 mm, indicating a consistency value between 100 and 300.

88

89 **Acidity or alkalinity**— To 10 g add 20 mL of boiling water and shake vigorously for 1 min. Allow
90 to cool and decant. To 10 mL of the aqueous layer add 0.1 mL of *phenolphthalein solution*. The
91 solution is colorless. Not more than 0.5 mL of *0.01 M sodium hydroxide* is required to change the
92 color of the indicator to pink or red.

93

94 **Residue on ignition**— Heat 2 g in an open porcelain or platinum dish: it volatilizes. Then ignite

95 at $600 \pm 50^\circ$ in the presence of sulfuric acid until constant weight and yield not more than 0.05%
96 of residue.

97

98 **UV absorbance limit for Polycyclic aromatic hydrocarbons**

99 Dissolve 1.0 g in 50 ml of hexane which has been previously shaken twice with 10 ml of *dimethyl*
100 *sulfoxide*. Transfer the Solution to a 125 ml separating funnel with unlubricated ground-glass parts
101 (stopper, stopcock). Add 20 ml of *dimethyl sulfoxide*. Shake vigorously for 1 min and allow to
102 stand until 2 clear layers are formed. Transfer the lower layer to a second separating funnel. Repeat
103 the extraction with a further 20 ml of *dimethyl sulfoxide*. Shake vigorously the combined lower
104 layers with 20 ml of *hexane* for 1 min. Allow to stand until 2 clear layers are formed. Separate the
105 lower layer and dilute to 50.0 ml with *dimethyl sulfoxide*. Measure the absorbance over the range
106 260 nm to 420 nm using a path length of 1 cm and as compensation liquid the clear lower layer
107 obtained by vigorously shaking 10 ml of *dimethyl sulfoxide* with 25 ml of *hexane* for 1 min.
108 Prepare a reference Solution in *dimethyl sulfoxide* containing 6.0 mg/L of *naphthalene* and
109 measure the absorbance of the Solution at the maximum at 278 nm using a path length of 1 cm and
110 *dimethyl sulfoxide* as compensation liquid. At no wavelength in the range 260 nm to 420 nm does
111 the absorbance of the test Solution exceed one-fourth of the reference Solution at 278 nm.

112

113 **REAGENTS**

114 **Ferric Chloride Solution (Yellow solution)**—Dissolve about 55 g of Ferric Chloride
115 ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in enough of a mixture of 25 mL of hydrochloric acid and 975 mL of water to make
116 1000 mL. Pipet 10 mL of this solution into a 250-mL iodine flask, add 15 mL of water, 3 g of
117 potassium iodide, and 5 mL of hydrochloric acid, and allow the mixture to stand for 15 minutes.
118 Dilute with 100 mL of water, and titrate the liberated iodine with 0.1 N sodium thiosulfate VS,
119 adding 3 mL of starch solution as the indicator. Perform a blank determination with the same
120 quantities of the same reagents, and make any necessary correction. Each mL of 0.1 N sodium
121 thiosulfate is equivalent to 27.03 mg of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. Adjust the final volume of the solution by the
122 addition of enough of the mixture of hydrochloric acid and water so that each mL contains 45.0
123 mg of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$.

124 **Starch Solution**—Mix 1 g of soluble starch with 10 mg of red mercuric iodide and sufficient cold
125 water to make a thin paste. Add 200 mL of boiling water, and boil for 1 minute with continuous
126 stirring. Cool, and use only the clear solution.

127 [NOTE 1—Commercially available, stabilized starch indicator solutions may be used.]

128 [NOTE 2—Starch solution can be prepared without the use of red mercuric iodide, provided the
129 solution is used immediately after preparation]

130 **Hydrogen Peroxide Solution** —Contains, in each 100 mL, not less than 2.5 g and not more than
131 3.5 g of H_2O_2 .

132 **Phenolphthalein Solution** —Dissolve 1 g of phenolphthalein in 100 mL of alcohol.