

PETROLEUM JELLY

Prepared at the 51st JECFA (1998), published in FNP 52 Add 6 (1998) superseding specifications prepared at the 49th JECFA (1997) published in FNP 52 Add 5 (1997). No ADI allocated at the 33rd JECFA in 1988.

SYNONYMS

Vaseline, Petrolatum, INS No. 905b

DEFINITION

A purified mixture of semi-solid, saturated hydrocarbons, mainly of paraffinic nature, obtained from petroleum. It may contain antioxidants approved for food use.

DESCRIPTION

White to yellowish or light amber semisolid substance. It is transparent in thin layers and has not more than a slight fluorescence.

FUNCTIONAL USES

Lubricant, release agent, protective coating, antifoaming agent.

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Insoluble in water; very soluble in carbon disulfide; soluble in ether and hexane

Drop melting point

Between 38 and 60°
See description under TESTS

Infrared absorption

The infrared absorption spectrum, obtained by spreading a thin film of the melted sample between sodium chloride plates, exhibits high intensity bands between 3000 cm^{-1} and 2800 cm^{-1} , medium intensity bands between 1500 cm^{-1} and 1300 cm^{-1} , and low intensity bands between 750 cm^{-1} and 700 cm^{-1} .

PURITY

Viscosity, 100° (Vol. 4)

Between 3 and 20 mm^2/s

Carbon number at 5% distillation point

Not more than 5% of molecules with carbon number less than 18
See description under TESTS

Average molecular weight

Between 350 and 650
See description under TESTS

Residue on ignition

Not more than 0.05%
Accurately weigh about 4 g of the sample in a tared porcelain or platinum dish and heat over a flame. The sample volatiles without emitting any acid odour. Ignite until free from carbon, without exceeding very dull redness. Cool in a desiccator and weigh.

Colour Melt about 10 g of the sample on a steam bath, and pour about 5 ml of the liquid into a 150 x 16-mm clear-glass bacteriological test tube, keeping the sample melted. The petrolatum is not darker than a solution made by mixing 3.8 ml of ferric chloride TS and 1.2 ml of cobaltous chloride TS in a similar tube, the comparison of the two being made in reflected light against a white background, holding the sample tube directly against the background at such an angle that there is no fluorescence.

Acidity or alkalinity To 10 g of the sample add 20 ml of boiling water and shake vigorously for one minute. Allow to cool and decant. To 10 ml of the aqueous layer add 0.1 ml of 1 to 10 solution of phenolphthalein TS. The solution is colourless. Not more than 0.5 ml of 0.01 N sodium hydroxide is required to change colour of the indicator to red.

Sulfur To 4.0 g add 2 ml of absolute alcohol and 2 drops of a 1 in 5 solution of sodium hydroxide previously saturated with lead monoxide, warm the mixture at about 70° for 1 min with frequently shaking and allow to cool. No dark colour is produced.

Readily carbonizable substances Use a clean, dry, ground-glass-stoppered test tube about 125 mm long and 18 mm in internal diameter, graduated at the 5- and 10-ml liquid levels. Place 5 ml of the melted sample in the test tube and add 5 ml of sulfuric acid. Insert the stopper and shake vigorously, in the longitudinal direction of the test tube, for 5 sec. Loosen the stopper, immediately place the tube in a waterbath, avoiding contact of the tube with the bottom or side of the bath, and heat for 10 min. After 2, 4, 6 and 8 min, remove the tube from the bath and shake, in the longitudinal direction of the test tube, for 5 sec. At the end of 10 min of heating, remove the tube from the waterbath and allow to stand for 10 min. Centrifuge at approximately 2000 g for 5 min: the lower layer is not more intensely coloured than a mixture of 0.5 ml of cupric sulfate TSC, 1.5 ml of cobaltous chloride TSC, 3.0 ml of ferric chloride TSC and 2 ml of 0.3 ml of 0.3 N hydrochloric acid.

Polycyclic aromatic hydrocarbons (Vol. 4) The sample shall meet the following ultraviolet absorbance when subjected to the analytical procedure as described in General methods

<u>nm</u>	<u>max. absorbance /cm path length</u>
280 - 289	0.15
290 - 299	0.12
300 - 359	0.08
360 - 400	0.02

Lead (Vol. 4) Not more than 2 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods."

TESTS

IDENTIFICATION

Drop melting point

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Apparatus

Test tubes: Standard test tubes, 25 mm in outside diameter and 150 mm long. The test tubes shall be supplied with corks grooved at the sides to permit air circulation and bored in exact center to receive the thermometer.
Bath: A transparent container of not less than 1500 ml capacity, that will permit the immersion of the test tubes to a depth of at least 100 mm and still leave a depth of 15 mm of water below the bottoms of the test tubes.
Test thermometer: ASTM 61C or equivalent
Bath thermometer: Any suitable type, accurate to 0.5° throughout the required range.

Procedure

Melt the sample slowly until the temperature reaches 93°. Place sufficient sample in a flat bottom container to give a sample depth of 12 ± 1 mm. Adjust the temperature to 6 to 11° above its drop melting point using any general laboratory thermometer for measurement. Chill one of the test thermometer bulbs to 4°. Wipe, dry and, quickly but carefully, immerse the chilled bulb vertically into the container (12 mm submerged) and withdraw it immediately. Hold the thermometer vertically away from the heat until the surface dulls and then place it for 5 min in water having a temperature of 16°. Prepare another specimen from the same sample using this procedure.

Securely fix the thermometers in the test tubes by means of corks so that the tip of each thermometer is 15 mm above the bottom of its test tube. Insert the test tubes in the water bath which is at 16° and adjust the height of the test tubes so that the immersion marks on the thermometers are level with the top surface of the water. Raise the temperature of the bath at a rate of 1.7°/min to 38°, then at a rate of 1°/min until the first drop of material leaves each thermometer. Record in each case the temperature at which the first drop falls from the thermometer. Calculate the average of the two temperatures.

Carbon number at 5% distillation point

(ASTM D 2887 See TEST for Drop melting point for Copyright permission)
Carbon number is the number of carbon atoms in a molecule. Determine the boiling point distribution of the sample by *gas chromatography* using the following conditions:

Detector: FID
Column:
material: glass
length: 10 m.
inside diameter: 0.53 mm

liquid phase: HT SIMDIST CB Chrompack 10, 0.17 µm film thickness or equivalent

Sample size: 1 µl

Temperature program:

70° - 200°, rate 25° / min.

200° - 325°, rate 12° min.

325° - 425°, rate 8°/min.

425° 15 min. isotherm

Detector temperature: 430° C

Carrier gas: hydrogen, 0.5 psi

Make up gas: nitrogen 25ml/min

Linearity standard:

Prepare a weighed mixture of n-paraffins covering the range between C₁₅ to as a minimum C₆₀ and dissolve the mixture in cyclohexane. It is not necessary to include every n-paraffin in this mixture, however it has to contain C₁₆, C₆₀ and at least one of every fourth n-paraffin.

Check of Solvent blank:

Inject 1 µl of the solvent. No peaks must be detected within the retention time range over which the wax elute.

Column resolution:

Inject 1 µl of a solution of 0.05% each of n-C₂₀ and n-C₂₄ in cyclohexane. The column resolution R must not be less than 30 as calculated by the following equation:

$$R = \frac{2d}{1.699(W1 + W2)}$$

where

d is the distance in mm between the peak maxima of n-C₂₀ and n-C₂₄

W1 is the peak width in mm of half height of n-C₂₀

W2 is the peak width in mm of half height of n-C₂₄

Linearity:

Analyze the linearity standard. The calculated mass response factors relative to hexadecane must be between 0.90 and 1.10.

Procedure:

Weigh 20 mg of the sample into a suitable glass vial and dissolve in 20 ml of cyclohexane. Agitate the vial until the wax is dissolved, using gentle heating if necessary. The solution should be clear when injected.

Inject 1.0 µl of the sample solution and integrate using a vertical drop to a horizontal baseline construction (Figure 1). Peaks resulting from the solvent and peaks with areas below 0.1% are not included in the calculation. For calculating and integration the common base line ends at the last C-normal peak with an area of 0.1%.

Calculation:

By definition: The non-normal hydrocarbon is followed by the normal hydrocarbon of the same carbon number.

Calculate the content of hydrocarbons of each carbon number, using the following equation:

$$C_i = \frac{A_i \times 100}{A_T}$$

where

C_i is the content in % of components having a carbon number of i

A_i is the combined peak areas of components having a carbon number of i

A_T is the total peak area

Average molecular weight (ASTM D 2502 See TEST for Drop melting point for Copyright permission)
Using the carbon number distribution obtained in the test for "Carbon number at 5% distillation point" calculate the average molecular weight by the following formula:

Average molecular weight =

$$\frac{\sum_{i=17}^{i=i} C_i (14i+2)}{100}$$

where

i is the carbon number

C_i is the content in % of components having a carbon number of i .

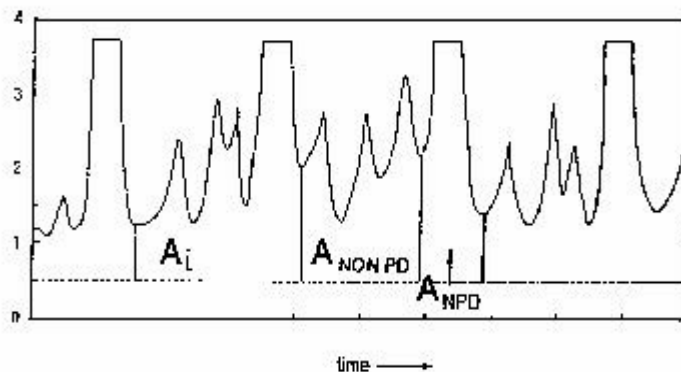


Figure 1 Carbon Number Summation (Vertical Drop to Horizontal Baseline)