

to stand until 2 clear layers are formed. Separate the lower layer and measure its absorbance (2.2.25) between 260 nm and 420 nm, using as the compensation liquid the clear lower layer obtained by vigorously shaking 5.0 ml of *dimethyl sulphoxide R* with 25 ml of *hexane R* for 1 min. Prepare a 7.0 mg/l reference solution of *naphthalene R* in *trimethylpentane R* and measure the absorbance of the solution at the absorption maximum at 275 nm, using *trimethylpentane R* as the compensation liquid. At no wavelength between 260 nm and 420 nm does the absorbance of the test solution exceed one-third that of the reference solution at 275 nm.

Readily carbonisable substances. Use a ground-glass-stoppered tube about 125 mm long and 18 mm in internal diameter, graduated at 5 ml and 10 ml; wash with hot *water R* (temperature at least 60 °C), *acetone R*, *heptane R* and finally with *acetone R*, dry at 100-110 °C. Cool in a desiccator. Introduce 5 ml of the substance to be examined and add 5 ml of *nitrogen-free sulphuric acid R1*. Insert the stopper and shake as vigorously as possible, in the longitudinal direction of the tube, for 5 s. Loosen the stopper, immediately place the tube in a water-bath, avoiding contact of the tube with the bottom or side of the bath, and heat for 10 min. After 2 min, 4 min, 6 min and 8 min, remove the tube from the bath and shake as vigorously as possible, in the longitudinal direction of the tube for 5 s. At the end of 10 min of heating, remove the tube from the water-bath and allow to stand for 10 min. Centrifuge at 2000 *g* for 5 min. The lower layer is not more intensely coloured (2.2.2, *Method I*) than a mixture of 0.5 ml of blue primary solution, 1.5 ml of red primary solution, 3.0 ml of yellow primary solution and 2 ml of a 10 g/l solution of *hydrochloric acid R*.

Solid paraffins. Dry a suitable quantity of the substance to be examined by heating at 100 °C for 2 h and cool in a desiccator over *sulphuric acid R*. Place in a glass tube with an internal diameter of about 25 mm, close the tube and immerse in a bath of iced water. After 4 h, the liquid is sufficiently clear for a black line, 0.5 mm wide, to be easily seen against a white background held vertically behind the tube.

STORAGE

Protected from light.

01/2008:1799

PARAFFIN, WHITE SOFT

Vaselinum album

DEFINITION

Purified and wholly or nearly decolorised mixture of semi-solid hydrocarbons, obtained from petroleum. It may contain a suitable antioxidant. White soft paraffin described in this monograph is not suitable for oral use.

CHARACTERS

Appearance: white or almost white, translucent, soft unctuous mass, slightly fluorescent in daylight when melted.

Solubility: practically insoluble in water, soluble in methylene chloride, practically insoluble in alcohol and in glycerol.

IDENTIFICATION

First identification: A, B, D.

Second identification: A, C, D.

- A. The drop point is between 35 °C and 70 °C and does not differ by more than 5 °C from the value stated on the label, according to method (2.2.17) with the following modification to fill the cup: heat the substance to be examined at a temperature not exceeding 80 °C, with stirring to ensure uniformity. Warm the metal cup at a temperature not exceeding 80 °C in an oven, remove it from the oven, place on a clean plate or ceramic tile and pour a sufficient quantity of the melted sample into the cup to fill it completely. Allow the filled cup to cool for 30 min on the plate or the ceramic tile and place it in a water bath at 24-26 °C for 30-40 min. Level the surface of the sample with a single stroke of a knife or razor blade, avoiding compression of the sample.
- B. Infrared absorption spectrophotometry (2.2.24).
Comparison: *Ph. Eur. reference spectrum of white soft paraffin.*
- C. Melt 2 g and when a homogeneous phase is obtained, add 2 ml of *water R* and 0.2 ml of 0.05 M *iodine*. Shake. Allow to cool. The solid upper layer is violet-pink.
- D. It complies with the test for appearance (see Tests).

TESTS

Appearance. The substance is white. Melt 12 g on a water-bath. The melted mass is not more intensely coloured than a mixture of 1 volume of yellow primary solution and 9 volumes of a 10 g/l solution of *hydrochloric acid R* (2.2.2, *Method II*).

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

Consistency (2.9.9): 60 to 300.

Polycyclic aromatic hydrocarbons: maximum 300 ppm.

Use reagents for ultraviolet spectrophotometry. Dissolve 1.0 g in 50 ml of *hexane R* which has been previously shaken twice with 10 ml of *dimethyl sulphoxide R*. Transfer the solution to a 125 ml separating funnel with unlubricated ground-glass parts (stopper, stopcock). Add 20 ml of *dimethyl sulphoxide R*. Shake vigorously for 1 min and allow to stand until 2 clear layers are formed. Transfer the lower layer to a second separating funnel. Repeat the extraction with a further 20 ml of *dimethyl sulphoxide R*. Shake vigorously the combined lower layers with 20 ml of *hexane R* for 1 min. Allow to stand until 2 clear layers are formed. Separate the lower layer and dilute to 50.0 ml with *dimethyl sulphoxide R*. Measure the absorbance (2.2.25) over the range 260 nm to 420 nm using a path length of 4 cm and as compensation liquid the clear lower layer obtained by vigorously shaking 10 ml of *dimethyl sulphoxide R* with 25 ml of *hexane R* for 1 min. Prepare a reference solution in *dimethyl sulphoxide R* containing 6.0 mg of *naphthalene R* per litre and measure the absorbance of the solution at the maximum at 278 nm using a path length of 4 cm and *dimethyl sulphoxide R* as compensation liquid. At no wavelength in the range 260 nm to 420 nm does the absorbance of the test solution exceed that of the reference solution at 278 nm.

Sulphated ash (2.4.14): maximum 0.05 per cent, determined on 2.0 g.

STORAGE

Protected from light.

LABELLING

The label states the nominal drop point.

01/2008:1554

PARAFFIN, YELLOW SOFT

Vaselinum flavum

DEFINITION

Yellow soft paraffin is a purified mixture of semi-solid hydrocarbons, obtained from petroleum. It may contain a suitable antioxidant.

CHARACTERS

A yellow, translucent, unctuous mass, slightly fluorescent in daylight when melted, practically insoluble in water, soluble in methylene chloride, practically insoluble in alcohol and in glycerol.

IDENTIFICATION

First identification: A, B, D.

Second identification: A, C, D.

- A. The drop point (2.2.17) is 40 °C to 60 °C and does not differ by more than 5 °C from the value stated on the label, with the following modification to fill the cup: heat the substance to be examined at 118 °C to 122 °C, with stirring to ensure uniformity, then cool to 100 °C to 107 °C. Warm the metal cup at 103 °C to 107 °C in an oven, remove it from the oven, place on a clean plate or ceramic tile and pour a sufficient quantity of the melted sample into the cup to fill it completely. Allow the filled cup to cool for 30 min on the ceramic tile and place it in a water-bath at 24 °C to 26 °C for a further 30 min to 40 min. Level the surface of the sample with a single stroke of a knife or razor blade, avoiding compression of the sample.
- B. Examine by infrared absorption spectrophotometry (2.2.24) comparing with the *Ph. Eur. reference spectrum of yellow soft paraffin*. The spectrum obtained shows major peaks similar in position and intensity to the *Ph. Eur. reference spectrum*.
- C. Melt 2 g and when a homogenous phase is obtained, add 2 ml of *water R* and 0.2 ml of 0.05 M *iodine*. Shake. Allow to cool. The solid upper layer is violet-pink.
- D. It complies with the test for appearance (see Tests).

TESTS

Appearance. The substance is yellow. Melt 12 g on a water-bath. The melted mass is not more intensely coloured than a mixture of 7.6 volumes of yellow primary solution and 2.4 volumes of red primary solution (2.2.2, *Method II*).

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

Consistency (2.9.9). The consistency is 100 to 300.

Polycyclic aromatic hydrocarbons. Use reagents for *ultraviolet absorption spectrophotometry*. Dissolve 1.0 g in 50 ml of *hexane R* which has been previously shaken

twice with one-fifth its volume of *dimethyl sulphoxide R*. Transfer the solution to a 125 ml separating funnel with unlubricated ground-glass parts (stopper, stopcock). Add 20 ml of *dimethyl sulphoxide R*. Shake vigorously for 1 min and allow to stand until two clear layers are formed. Transfer the lower layer to a second separating funnel. Repeat the extraction with a further 20 ml of *dimethyl sulphoxide R*. Shake vigorously the combined lower layers with 20 ml of *hexane R* for 1 min. Allow to stand until two clear layers are formed. Separate the lower layer and dilute to 50.0 ml with *dimethyl sulphoxide R*. Measure the absorbance (2.2.25) between 260 nm and 420 nm using a path length of 4 cm and using as the compensation liquid the clear lower layer obtained by vigorously shaking 10 ml of *dimethyl sulphoxide R* with 25 ml of *hexane R* for 1 min. Prepare a 9.0 mg/l reference solution of *naphthalene R* in *dimethyl sulphoxide R* and measure the absorbance of this solution at the maximum at 278 nm using a path length of 4 cm and using *dimethyl sulphoxide R* as the compensation liquid. At no wavelength in the range of 260 nm to 420 nm does the absorbance of the test solution exceed that of the reference solution at 278 nm.

Sulphated ash (2.4.14). Not more than 0.05 per cent, determined on 2.0 g.

STORAGE

Store protected from light.

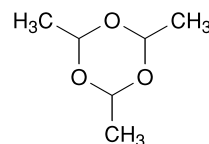
LABELLING

The label states the nominal drop point.

01/2008:0351

PARALDEHYDE

Paraldehydum



$C_6H_{12}O_3$
[123-63-7]

M_r 132.2

DEFINITION

2,4,6-Trimethyl-1,3,5-trioxane (cyclic trimer of acetaldehyde). It may contain a suitable quantity of an antioxidant.

CHARACTERS

Appearance: colourless or slightly yellow, transparent liquid. It solidifies on cooling to form a crystalline mass.

Solubility: soluble in water, but less soluble in boiling water, miscible with ethanol (96 per cent) and with essential oils.

IDENTIFICATION

- A. Solution S (see Tests) is clear (2.2.1) but becomes turbid on warming.
- B. To 5 ml add 0.1 ml of *dilute sulphuric acid R* and heat. Acetaldehyde, recognisable by its odour, is evolved.
- C. To 5 ml of solution S in a test-tube add 5 ml of *ammoniacal silver nitrate solution R* and heat in a water-bath. Silver is deposited as a mirror on the wall of the tube.