

Sulfated ash (2.4.14): maximum 0.1 per cent, determined on 1.0 g.

ASSAY

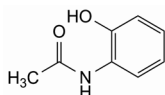
Dissolve 0.300 g in a mixture of 10 mL of *water R* and 30 mL of *dilute sulfuric acid R*. Boil under a reflux condenser for 1 h, cool and dilute to 100.0 mL with *water R*. To 20.0 mL of the solution add 40 mL of *water R*, 40 g of ice, 15 mL of *dilute hydrochloric acid R* and 0.1 mL of *ferroin R*. Titrate with 0.1 M *cerium sulfate* until a greenish-yellow colour is obtained. Carry out a blank titration.

1 mL of 0.1 M *cerium sulfate* is equivalent to 7.56 mg of $C_8H_9NO_2$.

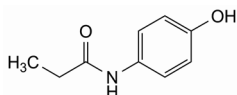
STORAGE

Protected from light.

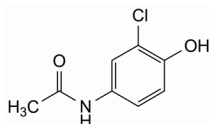
IMPURITIES



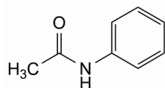
A. *N*-(2-hydroxyphenyl)acetamide,



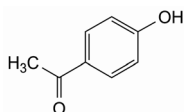
B. *N*-(4-hydroxyphenyl)propanamide,



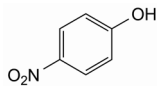
C. *N*-(3-chloro-4-hydroxyphenyl)acetamide,



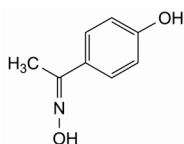
D. *N*-phenylacetamide,



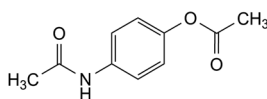
E. 1-(4-hydroxyphenyl)ethanone,



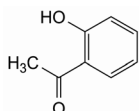
F. 4-nitrophenol,



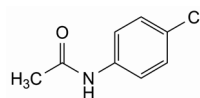
G. 1-(4-hydroxyphenyl)ethanone oxime,



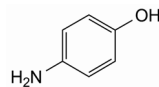
H. 4-(acetylamino)phenyl acetate,



I. 1-(2-hydroxyphenyl)ethanone,



J. *N*-(4-chlorophenyl)acetamide (chloroacetanilide),



K. 4-aminophenol.

01/2008:1034



PARAFFIN, HARD

Paraffinum solidum

DEFINITION

A purified mixture of solid saturated hydrocarbons generally obtained from petroleum. It may contain a suitable antioxidant.

CHARACTERS

Appearance: colourless or white or almost white mass; the melted substance is free from fluorescence in daylight.

Solubility: practically insoluble in water, freely soluble in methylene chloride, practically insoluble in ethanol (96 per cent).

IDENTIFICATION

First identification: A, C.

Second identification: B, C.

A. Infrared absorption spectrophotometry (2.2.24).

Comparison: hard paraffin CRS.

Preparation: place about 2 mg on a sodium chloride plate, heat in an oven at 100 °C for 10 min, spread the melted substance with another sodium chloride plate and remove one of the plates.

B. Acidity or alkalinity (see Tests).

C. Melting point (2.2.16): 50 °C to 61 °C.

TESTS

Acidity or alkalinity. To 15 g add 30 mL of boiling *water R* and shake vigorously for 1 min. Allow to cool and to separate. To 10 mL of the aqueous layer add 0.1 mL of *phenolphthalein solution R*. The solution is colourless. Not more than 1.0 mL of 0.01 M *sodium hydroxide* is required to change the colour of the indicator to red. To a further 10 mL of the aqueous layer add 0.1 mL of *methyl red solution R*. The solution is yellow. Not more than 0.5 mL of 0.01 M *hydrochloric acid* is required to change the colour of the indicator to red.

Polycyclic aromatic hydrocarbons. Use reagents for ultraviolet absorption spectrophotometry. Dissolve 0.50 g in 25 mL of *heptane R* and place in a 125 mL separating funnel with unlubricated ground-glass parts (stopper, stopcock). Add 5.0 mL of *dimethyl sulfoxide R*. Shake vigorously for 1 min and allow to stand until 2 clear layers are formed. Transfer the lower layer to a 2nd separating funnel, add 2 mL of *heptane R* and shake the mixture vigorously. Allow to stand until 2 clear layers are formed. Separate the lower layer and measure its absorbance (2.2.25) between 265 nm and 420 nm using as the compensation liquid the clear lower layer obtained by vigorously shaking 5.0 mL of *dimethyl sulfoxide R* with 25 mL of *heptane R* for 1 min. Prepare a 7.0 mg/L reference solution of *naphthalene R* in *dimethyl sulfoxide R* and measure the absorbance of this solution at the absorption maximum at 278 nm using *dimethyl sulfoxide R* as the compensation liquid.

At wavelengths from 265 nm to 420 nm, the absorbance of the test solution is not greater than one-third that of the reference solution at 275 nm.

Sulfates (2.4.13): maximum 150 ppm.

Introduce 2.0 g of the melted substance to be examined into a 50 mL ground-glass-stoppered separating funnel. Add 30 mL of boiling *distilled water R*, shake vigorously for 1 min and filter.

STORAGE

Protected from light.



01/2008:0240

PARAFFIN, LIGHT LIQUID

Paraffinum perliquidum

DEFINITION

Purified mixture of liquid saturated hydrocarbons obtained from petroleum.

CHARACTERS

Appearance: colourless, transparent, oily liquid, free from fluorescence in daylight.

Solubility: practically insoluble in water, slightly soluble in ethanol (96 per cent), miscible with hydrocarbons.

IDENTIFICATION

First identification: A, C.

Second identification: B, C.

A. Infrared absorption spectrophotometry (2.2.24).

Comparison: Ph. Eur. reference spectrum of liquid paraffin.

B. In a test tube cautiously boil 1 mL with 1 mL of 0.1 M sodium hydroxide, with continuous shaking, for about 30 s. On cooling to room temperature, 2 phases separate. To the aqueous phase add 0.1 mL of phenolphthalein solution R. The solution becomes red.

C. Viscosity (see Tests).

TESTS

Acidity or alkalinity. To 10 mL add 20 mL of boiling *water R* and shake vigorously for 1 min. Separate the aqueous layer and filter. To 10 mL of the filtrate, add 0.1 mL of phenolphthalein solution R. The solution is colourless. Not more than 0.1 mL of 0.1 M sodium hydroxide is required to change the colour of the indicator to pink.

Relative density (2.2.5): 0.810 to 0.875.

Viscosity (2.2.9): 25 mPa·s to 80 mPa·s.

Polycyclic aromatic hydrocarbons. Use reagents for ultraviolet spectrophotometry.

Introduce 25.0 mL into a 125 mL separating funnel with unlubricated ground-glass parts (stopper, stopcock). Add 25 mL of *hexane R* which has been previously shaken twice with one-fifth its volume of *dimethyl sulfoxide R*. Mix and add 5.0 mL of *dimethyl sulfoxide R*. Shake vigorously for 1 min and allow to stand until 2 clear layers are formed. Transfer the lower layer to a 2nd separating funnel, add 2 mL of *hexane R* and shake the mixture vigorously. Allow 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 sulfoxide 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 sulfuric 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 *sulfuric 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.



01/2008:0239

PARAFFIN, LIQUID

Paraffinum liquidum

DEFINITION

Purified mixture of liquid saturated hydrocarbons obtained from petroleum.

CHARACTERS

Appearance: colourless, transparent, oily liquid, free from fluorescence in daylight.

Solubility: practically insoluble in water, slightly soluble in ethanol (96 per cent), miscible with hydrocarbons.

IDENTIFICATION

First identification: A, C.

Second identification: B, C.

A. Infrared absorption spectrophotometry (2.2.24).

Comparison: Ph. Eur. reference spectrum of liquid paraffin.

B. In a test tube cautiously boil 1 mL with 1 mL of 0.1 M sodium hydroxide, with continuous shaking, for about 30 s. On cooling to room temperature, 2 phases separate. To the aqueous phase add 0.1 mL of phenolphthalein solution R. The solution becomes red.

C. Viscosity (see Tests).

TESTS

Acidity or alkalinity. To 10 mL add 20 mL of boiling *water R* and shake vigorously for 1 min. Separate the aqueous layer and filter. To 10 mL of the filtrate, add 0.1 mL of phenolphthalein