Edition: BP 2025 (Ph. Eur. 11.6 update)

Menadiol Phosphate Tablets

General Notices

Action and use

Vitamin K analogue.

DEFINITION

Menadiol Phosphate Tablets contain Menadiol Sodium Phosphate.

The tablets comply with the requirements stated under Tablets and with the following requirements.

Content of menadiol phosphate, $C_{11}H_{12}O_8P_2$

92.5 to 107.5% of the stated amount.

IDENTIFICATION

A. Shake a quantity of the powdered tablets containing the equivalent of 0.15 g of menadiol phosphate with 15 mL of *water*, centrifuge and filter the supernatant liquid. To 10 mL of the filtrate add 10 mL of 1M *sulfuric acid*, 10 mL of 0.1M *cerium(IV) sulfate* and 1 mL of *hydrogen peroxide solution (20 vol)* and extract with two 10 mL quantities of *chloroform*. Evaporate the combined chloroform extracts to dryness in a water bath and dry the residue at 40° at a pressure not exceeding 0.7 kPa. The *infrared absorption spectrum* of the residue, Appendix II A, is concordant with the *reference spectrum* of menadione *(RS 214)*.

B. To 50 mg of the residue obtained in test A add 5 mL of *water* followed by 75 mg of *sodium metabisulfite*, heat in a water bath, shaking vigorously, until an almost colourless solution is obtained and dilute to 50 mL with *water*. To 2 mL of the resulting solution add 2 mL of a mixture of equal volumes of 13.5M *ammonia* and *ethanol (96%)*, shake and add 0.15 mL of *ethyl cyanoacetate*. A deep purplish-blue colour is produced which changes to green on the addition of 1 mL of 10M *sodium hydroxide*.

TESTS

Related substances

Carry out in subdued light the method for *thin-layer chromatography*, Appendix III A, using *silica gel* GF_{254} as the coating substance and a mixture of 50 volumes of *propan-1-ol*, 50 volumes of a 2% w/v solution of *ammonium chloride*, 5 volumes of *butan-1-ol* and 1.5 volumes of *diethylamine* as the mobile phase. Apply separately to the plate 5 µL of each of the following solutions. For solution (1) shake a quantity of the powdered tablets containing the equivalent of 0.25 g of menadiol phosphate with 10 mL of *methanol (50%)* and filter. For solution (2) dilute 1 volume of solution (1) to 200 volumes with *methanol (50%)*. Solution (3) contains 0.0080% w/v of *2-methyl-1,4-naphthoquinone* in *methanol*. After removal of the plate, allow it to dry in air and examine under *ultraviolet light (254 nm)*. Any *secondary spot* in the chromatogram obtained with solution (1) is not more intense than the spot in the chromatogram obtained with solution (3). Examine the plate under *ultraviolet light (365 nm)*. Any *secondary spot* in the chromatogram obtained with solution (1) is not more intense than the spot in the chromatogram obtained with solution (2).

ASSAY

Weigh and powder 20 tablets. Shake a quantity of the powder containing the equivalent of 10 mg of menadiol phosphate with 100 mL of 0.1M *hydrochloric acid* for 30 minutes. Dilute to 250 mL with 0.1M *hydrochloric acid*, filter and measure the *absorbance* of the filtrate at the maximum at 290 nm, Appendix II B. Calculate the content of $C_{11}H_{12}O_8P_2$ taking the value of A(1%, 1 cm), for a solution of $C_{11}H_8Na_4O_8P_2$ in 0.1M *hydrochloric acid*, to be 138 at the maximum at 290 nm. Each mg of $C_{11}H_8Na_4O_8P_2$ (menadiol sodium phosphate) is equivalent to 0.792 mg of $C_{11}H_{12}O_8P_2$ (menadiol phosphate).

LABELLING

The quantity of active ingredient is stated in terms of the equivalent amount of menadiol phosphate.