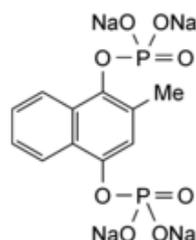




Edition: BP 2025 (Ph. Eur. 11.6 update)

Menadiol Sodium Phosphate

[General Notices](#)



$C_{11}H_8Na_4O_8P_2 \cdot 6H_2O$ 530.2 6700-42-1

Action and use

Vitamin K analogue.

Preparation

[Menadiol Phosphate Tablets](#)

DEFINITION

Menadiol Sodium Phosphate is tetrasodium 2-methylnaphthalene-1,4-diyl di(orthophosphate) hexahydrate. It contains not less than 98.0% and not more than 100.5% of $C_{11}H_8Na_4O_8P_2$, calculated with reference to the anhydrous substance.

CHARACTERISTICS

A white to pink, crystalline powder; hygroscopic.

Very soluble in [water](#); practically insoluble in [ethanol \(96%\)](#).

IDENTIFICATION

A. The [infrared absorption spectrum](#), [Appendix II A](#), is concordant with the *reference spectrum* of menadiol sodium phosphate ([RS 213](#)).

B. To 10 mL of a 2% w/v solution 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 on 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.

C. Dissolve 40 mg in 2 mL of [water](#), heat gently with 2 mL of [sulfuric acid](#) until white fumes are evolved, add [nitric acid](#) dropwise until digestion is complete and cool. Add 2 mL of [water](#), heat until white fumes are evolved again, cool, add a further 10 mL of [water](#) and neutralise to [litmus paper](#) with 5M [ammonia](#). The solution yields reaction A characteristic of [sodium salts](#) and reaction B characteristic of [phosphates](#), [Appendix VI](#).

TESTS

Inorganic phosphate

Dissolve 25 mg in 10 mL of [water](#), add 4 mL of 1M [sulfuric acid](#), 1 mL of a 10% w/v solution of [ammonium molybdate](#) and 2 mL of [methylaminophenyl-sulfite reagent](#) and allow to stand for 15 minutes. The [absorbance](#) of the solution at 730 nm, [Appendix II B](#), is not more than the [absorbance](#) of a solution prepared in the same manner but using 10 mL of a 0.0025% w/v solution of [potassium dihydrogen orthophosphate](#) in place of the solution of the substance being examined (0.7%).

Total phosphate

Dissolve 85 mg in 50 mL of [glacial acetic acid](#), add 5 mL of [mercury\(II\) acetate solution](#) and carry out Method I for [non-aqueous titration](#), [Appendix VIII A](#), determining the end point [potentiometrically](#). Not less than 7.7 mL and not more than 8.3 mL of [0.1M perchloric acid VS](#) is required.

Related substances

Carry out the method for [thin-layer chromatography](#), [Appendix III A](#), in subdued light using the following solutions in [methanol](#) (50%).

- (1) 4.0% w/v of the substance being examined.
- (2) 0.020% w/v of the substance being examined.
- (3) 0.0080% w/v of [2-methyl-1,4-naphtha-quinone](#).

CHROMATOGRAPHIC CONDITIONS

- (a) Use as the coating [silica gel GF₂₅₄](#).
- (b) Use the mobile phase as described below.
- (c) Apply 5 µL of each solution.
- (d) Develop the plate to 15 cm.
- (e) After removal of the plate, dry in air, examine under [ultraviolet light \(254 nm\)](#).

MOBILE PHASE

1.5 volumes of [diethylamine](#), 5 volumes of [butan-1-ol](#), 50 volumes of [propan-1-ol](#) and 50 volumes of a 2% w/v solution of [ammonium chloride](#).

LIMITS

Any spot corresponding to 2-methyl-1,4-naphthaquinone (menadione) in the chromatogram obtained with solution (1) is not more intense than the spot in the chromatogram obtained with solution (3) and any other [secondary spot](#) is not more intense than the spot in the chromatogram obtained with solution (2).

Water

19.0 to 21.5% w/w, [Appendix IX A](#). Use 0.25 g.

ASSAY

Dissolve 0.1 g in 25 mL of [water](#), add 25 mL of [glacial acetic acid](#) and 25 mL of [3M hydrochloric acid](#) and titrate with [0.02M cerium\(IV\) sulfate VS](#) using platinum and calomel electrodes and determining the end point [potentiometrically](#). Each mL of [0.02M cerium\(IV\) sulfate VS](#) is equivalent to 4.221 mg of $C_{11}H_8Na_4O_8P_2$.