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Oxytetracycline Veterinary Oral Powder

General Notices

Action and use

Tetracycline antibacterial.

DEFINITION

Oxytetracycline Veterinary Oral Powder is a mixture of Oxytetracycline Hydrochloride and a suitable diluent.

Content of oxytetracycline hydrochloride, $C_{22}H_{24}N_2O_9$,HCl

90.0 to 105.0% of the stated amount.

The veterinary oral powder complies with the requirements stated under Veterinary Oral Powders and with the following requirements.

IDENTIFICATION

A. Carry out the method for *thin-layer chromatography*, Appendix III A, using the following solutions.
(1) Extract a quantity of the oral powder containing 10 mg of Oxytetracycline Hydrochloride with 20 mL of *methanol*, centrifuge and use the supernatant liquid.
(2) 0.05% w/v of *oxytetracycline hydrochloride BPCRS* in *methanol*.
(3) 0.05% w/v of each of *oxytetracycline hydrochloride BPCRS* and *demeclocycline hydrochloride BPCRS* in *methanol*.

CHROMATOGRAPHIC CONDITIONS

(a) Use a *silica gel* precoated plate (Merck silica gel 60 plates are suitable). Adjust the pH of a 10% w/v solution of *disodium edetate* to 7.0 with 10M *sodium hydroxide* and spray the solution evenly onto the plate (about 10 mL for a plate 100 mm × 200 mm). Allow the plate to dry in a horizontal position for at least 1 hour. Before use, dry the plate at 110° for 1 hour.
(b) Use the mobile phase as described below.
(c) Apply 1 µL of each solution.
(d) Develop the plate to 15 cm.
(e) After removal of the plate, dry it in a current of air and examine under *ultraviolet light (365 nm)*.

MOBILE PHASE

6 volumes of *water*, 35 volumes of *methanol* and 59 volumes of *dichloromethane*.

SYSTEM SUITABILITY

The test is not valid unless the chromatogram obtained with solution (3) shows two clearly separated spots.

CONFIRMATION

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The principal spot in the chromatogram obtained with solution (1) is similar in position, colour and size to that in the chromatogram obtained with solution (2).

B. To a quantity of the powder containing 0.4 mg of Oxytetracycline Hydrochloride add 5 mL of a 1% w/v solution of *sodium carbonate*, shake and add 2 mL of *diazobenzenesulfonic acid solution*. A light brown colour is produced.
C. Shake a quantity of the powder containing 0.1 g of Oxytetracycline Hydrochloride with 10 mL of 2M *nitric acid* and filter. Decolourise the filtrate with *activated charcoal* and filter again. The filtrate yields the reactions characteristic of *chlorides*, Appendix VI.

Related substances

Carry out the method for *liquid chromatography*, Appendix III D, using the following solutions in a mixture of 20 volumes of *acetonitrile* and 80 volumes of 0.01M oxalic acid (solvent A). Prepare the solutions immediately before use.

(1) Shake a quantity of the oral powder containing 0.16 g of Oxytetracycline Hydrochloride in 150 mL of solvent A and dilute to 200 mL. Filter the resulting solution (Whatman GF/C filter is suitable).
(2) Dilute 1 volume of solution (1) to 100 volumes.
(3) 0.08% w/v of *oxytetracycline for system suitability A EPCRS*.
(4) Dilute 1 volume of solution (2) to 10 volumes.

CHROMATOGRAPHIC CONDITIONS

(a) Use a stainless steel column (15 cm × 4.6 mm) packed with *end-capped octylsilyl silica gel for chromatography* (5 µm) (Intertsil C8 is suitable).
(b) Use gradient elution and the mobile phase described below.
(c) Use a flow rate of 1.3 mL per minute.
(d) Use a column temperature of 50°.
(e) Use a detection wavelength of 254 nm.
(f) Inject 10 µL of each solution.

MOBILE PHASE

Mobile phase A 0.05% v/v *trifluoroacetic acid*.

Mobile phase B 5 volumes of *tetrahydrofuran*, 15 volumes of *methanol* and 80 volumes of *acetonitrile*.

Time (Minutes)	Mobile phase A (% v/v)	Mobile phase B (% v/v)	Comment
0-5	90	10	isocratic
5-20	90→65	10→35	linear gradient
20-21	65→90	35→10	linear gradient
21-27	90	10	re-equilibration

When the chromatograms are recorded under the prescribed conditions, the relative retentions with reference to oxytetracycline (retention time about 7 minutes) are: impurity A, about 0.9; impurity B, about 1.2; impurity C, about 1.3; impurity D, about 1.4; impurity E, about 2.2; impurity F, about 2.3.

SYSTEM SUITABILITY

The test is not valid unless, in the chromatogram obtained with solution (3), the *peak-to-valley ratio* is at least 3.0, where *Hp* is the height above the baseline of the peak due to impurity A and *Hv* is the height above the baseline of the lowest point of the curve separating this peak from the peak due to oxytetracycline.

The test is not valid unless, in the chromatogram obtained with solution (3), the *peak-to-valley ratio* is at least 3.0, where *Hp* is the height above the baseline of the peak due to impurity B and *Hv* is the height above the baseline of the lowest point of the curve separating this peak from the peak due to oxytetracycline.

LIMITS

Identify any peak corresponding to impurities A, B, C, D, E and F in the chromatogram obtained with solution (1), using the chromatogram obtained with solution (3). Multiply the areas of the peaks due to Impurity D and E by a correction factor of 0.4.

In the chromatogram obtained with solution (1):

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the area of any peaks corresponding to impurity A, impurity B and impurity C are not greater than twice the area of the principal peak in the chromatogram obtained with solution (2) (2.0% of each);

the area of any other *secondary peak* is not greater than the area of the principal peak in the chromatogram obtained with solution (2) (1.0%);

the sum of the areas of all the *secondary peaks* is not greater than 5 times the area of the principal peak in the chromatogram obtained with solution (2) (5.0%).

Disregard any peak with an area less than the area of the principal peak in the chromatogram obtained with solution (4) (0.1%).

ASSAY

Carry out the method for *liquid chromatography*, Appendix III D, using the following solutions in a mixture of 20 volumes of *acetonitrile* and 80 volumes of 0.01M *oxalic acid* (solvent A). Prepare the solutions immediately before use.

(1) Shake, with the aid of ultrasound, a quantity of the powder containing 0.16 g of Oxytetracycline Hydrochloride with 150 mL of solvent A. Dilute to 200 mL and filter (Whatman GF/C filter is suitable). Dilute 1 volume of the filtrate to 10 volumes.
(2) 0.0074% w/v of *oxytetracycline BPCRS*.

CHROMATOGRAPHIC CONDITIONS

The chromatographic conditions stated under Related substances may be used.

DETERMINATION OF CONTENT

Calculate the content of $C_{22}H_{24}N_2O_9$,HCl in the powder using the declared content of $C_{22}H_{24}N_2O_9$ in *oxytetracycline BPCRS*. Each mg of $C_{22}H_{24}N_2O_9$ is equivalent to 1.079 mg of $C_{22}H_{24}N_2O_9$,HCl.

IMPURITIES

The impurities limited by the requirements of this monograph include those listed under Oxytetracycline Hydrochloride.