

the plate, and allow to stand in ammonia gas: a spot from the sample solution and a grayish green spot from the standard solution show the same color tone and the same *R_f* value.

Containers and storage Containers—Tight containers.

Orange Peel Tincture

トウヒチンキ

Method of preparation

Bitter Orange Peel, in coarse powder	200 g
70 vol% Ethanol	a sufficient quantity
To make 1000 mL	

Prepare as directed under Tinctures, with the above ingredients. An appropriate quantity of Ethanol and Purified Water may be used in place of 70 vol% Ethanol.

Description Orange Peel Tincture is a yellowish brown liquid. It has a characteristic odor, and a bitter taste.

Specific gravity d_{20}^{20} : about 0.90

Identification To 5.0 mL of Orange Peel Tincture add 5 mL of ethanol (95), filter if necessary, and use the filtrate as the sample solution. Proceed as directed in the Identification under Bitter Orange Peel.

Alcohol number Not less than 6.6 (Method 2).

Containers and storage Containers—Tight containers.

Oriental Bezoar

Bezoar Bovis

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Oriental Bezoar is a stone formed in the gall sac of *Bos taurus* Linné var. *domesticus* Gmelin (*Bovidae*).

Description Spherical or massive stone, 1–4 cm in diameter; externally yellow-brown to red-brown; light, fragile and easily broken. Fractured surface shows yellow-brown to red-brown annular rings, often containing white granular substances or thin layers in these annular rings. Odor, slight; taste, slightly bitter, followed by slight sweetness.

Identification (1) Shake 0.1 g of pulverized Oriental Bezoar with 10 mL of petroleum ether for 30 minutes, filter, and wash the residue with 10 mL of petroleum ether. Shake 0.01 g of the residue with 3 mL of acetic anhydride for 1 to 2 minutes, add a mixture of 0.5 mL of acetic anhydride and 2 drops of sulfuric acid, and shake: a yellow-red to deep red color develops, and changes through dark red-purple to dark red-brown.

(2) Shake well 0.01 g of Oriental Bezoar with 1 mL of hydrochloric acid and 10 mL of chloroform, separate the chloroform layer when it acquires a yellow-brown color, and shake with 5 mL of barium hydroxide TS: a yellow-brown precipitate is produced.

Purity (1) Synthetic dye—To 2 mg of pulverized Oriental Bezoar add 1 mL dilute hydrochloric acid: no violet color develops.

(2) Starch—To 5 mg of pulverized Oriental Bezoar add 2 mL of water, and heat on a water bath for 5 minutes. Cool and add 2 to 3 drops of iodine TS: no blue-purple color develops.

(3) Sucrose—To 0.02 g of pulverized Oriental Bezoar add 10 mL of water, shake for 15 minutes, and filter. To 1 mL of the filtrate add 2 mL of anthrone TS, and shake: no deep blue-green to dark green color develops.

Total ash Not more than 10.0%.

Content of the active principle Weigh accurately about 0.5 g of pulverized Oriental Bezoar in a flask, add 50 mL of petroleum ether, warm under a reflux condenser on a water bath for 2 hours, and filter. Replace the residue along with the filter paper in the flask, add 2 mL of hydrochloric acid and 40 mL of chloroform, warm under a reflux condenser on a water bath for 1 hour, and filter into a tared flask. Wash the filter paper with a small quantity of chloroform, combine the washings with the filtrate, and distil off the chloroform. Dry the residue in a desiccator (silica gel) for 24 hours, and weigh: the mass of the residue is not less than 12.0%.

Compound Oxycodone Injection

Compound Hycodenone Injection

複方オキシコドン注射液

Compound Oxycodone Injection is an aqueous solution for injection.

It contains not less than 0.74 w/v% and not more than 0.86 w/v% of oxycodone hydrochloride ($C_{18}H_{21}NO_4 \cdot HCl \cdot 3H_2O$: 405.87), and not less than 0.18 w/v% and not more than 0.22 w/v% of hydrocotarnine hydrochloride ($C_{12}H_{15}NO_3 \cdot HCl \cdot H_2O$: 275.73).

Method of preparation

Oxycodone Hydrochloride	8 g
Hydrocotarnine Hydrochloride	2 g
Water for Injection	a sufficient quantity
To make 1000 mL	

Prepare as directed under Injections, with the above ingredients.

Description Compound Oxycodone Injection is a clear, colorless to pale yellow liquid.

It is affected by light.

pH: 2.5–4.0

Identification (1) To 1 mL of Compound Oxycodone Injection add 1 mL of 2,4-dinitrophenylhydrazine-ethanol TS: a yellow precipitate is formed (oxycodone).

(2) Evaporate 1 mL of Compound Oxycodone Injection on a water bath. Dissolve the residue in 2 mL of sulfuric acid: a yellow color is produced. Heat the solution: it changes to red, and then to deep orange-red (hydrocotarnine).

(3) Evaporate 1 mL of Compound Oxycodone Injection on a water bath. Dissolve the residue in 3 mL of sulfuric acid, add 2 drops of a solution of tannic acid in ethanol (95) (1 in 20), and allow to stand: a deep green color is produced (hydrocotarnine).

Assay Pipet 2 mL of Compound Oxycodone Injection, add exactly 10 mL of the internal standard solution, and use this solution as the sample solution. Separately, weigh accurately about 0.4 g of oxycodone hydrochloride for assay and about 0.1 g of hydrocotarnine hydrochloride for assay previously dried at 105°C for 3 hours, and dissolve in water to make exactly 50 mL. Pipet 2 mL of this solution, add exactly 10 mL of the internal standard solution, and use this solution as the standard solution. Perform the test with 10 μ L each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Calculate the ratios, Q_{Ta} and Q_{Tb} , of the peak area of oxycodone hydrochloride and hydrocotarnine hydrochloride to that of the internal standard from the sample solution, and the ratios, Q_{Sa} and Q_{Sb} , of the peak area of oxycodone hydrochloride and hydrocotarnine hydrochloride to that of the internal standard from the standard solution.

$$\begin{aligned} & \text{Amount (mg) of oxycodone hydrochloride} \\ & (\text{C}_{18}\text{H}_{21}\text{NO}_4 \cdot \text{HCl} \cdot 3\text{H}_2\text{O}) \\ & = \text{amount (mg) of oxycodone hydrochloride for assay,} \\ & \text{calculated on the anhydrous basis} \\ & \times \frac{Q_{Ta}}{Q_{Sa}} \times 1.1536 \times \frac{1}{25} \end{aligned}$$

$$\begin{aligned} & \text{Amount (mg) of hydrocotarnine hydrochloride} \\ & (\text{C}_{12}\text{H}_{15}\text{NO}_3 \cdot \text{HCl} \cdot \text{H}_2\text{O}) \\ & = \text{amount (mg) of hydrocotarnine hydrochloride for} \\ & \text{assay} \\ & \times \frac{Q_{Tb}}{Q_{Sb}} \times 1.0699 \times \frac{1}{25} \end{aligned}$$

Internal standard solution—Dissolve 0.02 g of phenacetin in 10 mL of ethanol (95), and add water to make 100 mL.

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 285 nm).

Column: A stainless steel column about 4 mm in inside diameter and about 15 cm in length, packed with octadecylsilanized polyvinyl alcohol gel polymer for liquid chromatography (5 μ m in particle diameter).

Column temperature: A constant temperature of about 25°C.

Mobile phase: To 500 mL of 0.05 mol/L disodium hydrogenphosphate TS add 0.05 mol/L sodium dihydrogenphosphate TS, and adjust the pH to 8.0. To 300 mL of this solution add 200 mL of acetonitrile, and mix.

Flow rate: Adjust the flow rate so that the retention time of oxycodone hydrochloride is about 8 minutes.

Selection of column: Proceed with 10 μ L of the standard solution under the above operating conditions, and use a column giving elution of the internal standard, oxycodone hydrochloride and hydrocotarnine hydrochloride in this order, and complete separation of their peaks.

Containers and storage Containers—Hermetic containers, and colored containers may be used.

Storage—Light-resistant.

Compound Oxycodone and Atropine Injection

Hycoato Injection

複方オキシコドン・アトロピン注射液

Compound Oxycodone and Atropine Injection is an aqueous solution for injection.

It contains not less than 0.74 w/v% and not more than 0.86 w/v% of oxycodone hydrochloride ($\text{C}_{18}\text{H}_{21}\text{NO}_4 \cdot \text{HCl} \cdot 3\text{H}_2\text{O}$: 405.87), not less than 0.18 w/v% and not more than 0.22 w/v% of hydrocotarnine hydrochloride ($\text{C}_{12}\text{H}_{15}\text{NO}_3 \cdot \text{HCl} \cdot \text{H}_2\text{O}$: 275.73), and not less than 0.027 w/v% and not more than 0.033 w/v% of atropine sulfate [$(\text{C}_{17}\text{H}_{23}\text{NO}_3)_2 \cdot \text{H}_2\text{SO}_4 \cdot \text{H}_2\text{O}$: 694.83].

Method of preparation

Oxycodone Hydrochloride	8 g
Hydrocotarnine Hydrochloride	2 g
Atropine Sulfate	0.3 g
Water for Injection	a sufficient quantity
To make 1000 mL	

Prepare as directed under Injections, with the above ingredients.

Description Compound Oxycodone and Atropine Injection is a colorless or pale yellow, clear liquid.

It is affected by light.

pH: 2.5 - 4.0

Identification (1) To 1 mL of Compound Oxycodone and Atropine Injection add 1 mL of 2,4-dinitrophenylhydrazine-ethanol TS: a yellow precipitate is formed (oxycodone).

(2) Evaporate 1 mL of Compound Oxycodone and Atropine Injection on a water bath, and dissolve the residue in 2 mL of sulfuric acid: a yellow color is produced. Heat the solution: it changes to red, and then to deep orange-red (hydrocotarnine).

(3) Evaporate 1 mL of Compound Oxycodone and Atropine Injection on a water bath. Dissolve the residue in 3 mL of sulfuric acid, add 2 drops of a solution of tannic acid in ethanol (95) (1 in 20), and allow to stand: a deep green color is produced (hydrocotarnine).

(4) To 1 mL of Compound Oxycodone and Atropine Injection add 0.5 mL of 2,4-dinitrophenylhydrazine-ethanol TS, and allow to stand for 1 hour. Centrifuge, and add acetone to the supernatant liquid until no more precipitate is produced. Allow to stand for 20 minutes, and centrifuge. To the supernatant liquid add potassium hydroxide TS until the liquid is light purple. Shake the liquid with 5 mL of dichloromethane, and separate the dichloromethane layer. Take 0.5 mL of the dichloromethane layer, and evaporate to dryness on a water bath. Add 5 drops of fuming nitric acid to the residue, and evaporate to dryness on a water bath. Cool, dissolve the residue in 1 mL of *N,N*-dimethylformamide, and add 6 drops of tetraethylammonium hydroxide TS: a red-purple color is produced (atropine).

Assay (1) Oxycodone hydrochloride and hydrocotarnine hydrochloride—Pipet 2 mL of Compound Oxycodone and