retention time of dydrogesterone after the solvent peak.

Loss on drying Not more than 0.5% (0.5 g, in vacuum, phosphorus (V) oxide, 24 hours).

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 0.05 g of Dydrogesterone, previously dried, and dissolve in methanol to make exactly 100 mL. Pipet 1 mL of this solution, and add methanol to make exactly 100 mL. Determine the absorbance A of this solution at the wavelength of maximum absorption at about 286 nm as directed under the Ultraviolet-visible Spectrophotometry.

$$\text{Amount (mg) of } C_{21}H_{28}O_2 = \frac{A}{845} \times 100{,}000$$

Containers and storage Containers—Tight containers.

Dydrogesterone Tablets

ジドロゲステロン錠

Dydrogesterone Tablets contain not less than 95% and not more than 105% of the labeled amount of dydrogesterone ($C_{21}H_{28}O_2$: 312.45).

Method of preparation Prepare as directed under Tablets, with Dydrogesterone.

Identification **(1)** To a quantity of powdered Dydrogesterone Tablets, equivalent to 0.05 g of Dydrogesterone according to the labeled amount, add 50 mL of methanol, shake well, and filter. Evaporate 5 mL of the filtrate on a water bath to dryness. Proceed with the residue as directed in the Identification (1) under Dydrogesterone.

(2) To 1 mL of the filtrate obtained in (1) add methanol to make 200 mL. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 284 nm and 288 nm.

Dissolution test Perform the test with 1 tablet of Dydrogesterone Tablets at 50 revolutions per minute according to Method 2 under the Dissolution Test, using 900 mL of water as the test solution. Take 20 mL or more of the dissolved solution 30 minutes after starting the test, and filter. Discard the first 10 mL of the filtrate, and use the subsequent as the sample solution. Separately, weigh accurately about 0.05 g of dydrogesterone for assay, previously dried in a desiccator (in vacuum, phosphorus (V) oxide) for 24 hours, and dissolve in methanol to make exactly 100 mL. Pipet 1 mL of this solution, add water to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances, A_T and A_S, of the sample solution and the standard solution at 296 nm as directed under the Ultraviolet-visible Spectrophotometry.

The dissolution rate of Dydrogesterone Tablets in 30 minutes is not less than 80%.

$$\text{Dissolution rate (\%) with respect to the labeled amount of dydrogesterone } (C_{21}H_{28}O_2) = W_S \times \frac{A_T}{A_S} \times \frac{1}{C} \times 9$$

W_S: Amount (mg) of dydrogesterone for assay.
C: Labeled amount (mg) of dydrogesterone ($C_{21}H_{28}O_2$) in 1 tablet.

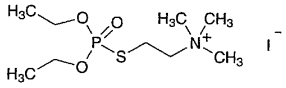
Assay Weigh accurately and powder not less than 20 Dydrogesterone Tablets. Weigh accurately a portion of the powder, equivalent to about 0.01 g of dydrogesterone ($C_{21}H_{28}O_2$), shake well with 50 mL of methanol, and add methanol to make exactly 100 mL. Filter this solution, discard the first 20 mL of the filtrate, pipet the subsequent 5 mL, add methanol to make exactly 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of dydrogesterone for assay, previously dried in a desiccator (in vacuum, phosphorus (V) oxide) for 24 hours, proceed in the same manner as the preparation of the sample solution, and use the solution as the standard solution. Determine the absorbances, A_T and A_S, of the sample solution and the standard solution at 286 nm as directed under the Ultraviolet-visible Spectrophotometry.

$$\text{Amount (mg) of dydrogesterone } (C_{21}H_{28}O_2) = \text{amount (mg) of dydrogesterone for assay} \times \frac{A_T}{A_S}$$

Containers and storage Containers—Tight containers.

Ecothiopate Iodide

ヨウ化エコチオパート



$C_9H_{23}INO_3PS$: 383.23
N-[2-(Diethoxyphosphorylsulfanyl)ethyl]-*N*,*N*,*N*-trimethylammonium iodide [*513-10-0*]

Ecothiopate Iodide contains not less than 95.0% of $C_9H_{23}INO_3PS$, calculated on the dried basis.

Description Ecothiopate Iodide occurs as white crystals or crystalline powder.

It is very soluble in water, freely soluble in methanol, slightly soluble in ethanol (95), and practically insoluble in diethyl ether.

Identification **(1)** Dissolve 0.1 g of Ecothiopate Iodide in 2 mL of water, and add 1 mL of nitric acid: a brown precipitate is formed. To 1 drop of the turbid solution containing this precipitate add 1 mL of hexane, and shake: a light red color develops in the hexane layer.

(2) Heat the suspension of the precipitate obtained in (1) until it becomes colorless, cool, add 10 mL of water, and use this solution as the sample solution. Two mL of the sample solution responds to the Qualitative Tests (2) for phosphate.

(3) Two mL of the sample solution obtained in (2) responds to the Qualitative Tests for sulfate.

pH Dissolve 0.1 g of Ecothiopate Iodide in 40 mL of water: the pH of this solution is between 3.0 and 5.0.

Melting point 116 – 122°C