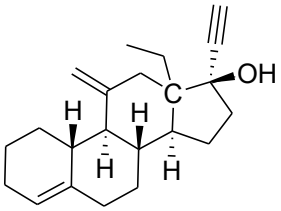
Desogestrel



$C_{22}H_{30}O$ Mol. Wt. 310.5

Desogestrel is 18,19-Dinorpregn-4-en-20-yn-17-ol,13-ethyl-11-methylene-,(17α).

Desogestrel contains not less than 98.0 per cent and not more than 102.0 per cent of $C_{22}H_{30}O$, calculated on the dried basis.

Category. Steroidal hormone.

Description. A white or almost-white, crystalline powder.

Identification

A. Determine by infrared absorption spectrophotometry (2.4.6). Compare the spectrum with that obtained with *desogestrel IPRS* or with the reference spectrum of desogestrel.

B. In the Assay, the principal peak in the chromatogram obtained with the test solution corresponds to the peak in the chromatogram obtained with reference solution (d).

Tests

Specific optical rotation (2.4.22). +53° to +57°, determined in a 1.0 per cent w/v solution in *ethanol*.

Related substances. Determine by liquid chromatography (2.4.14).

Solvent mixture. Equal volumes of *acetonitrile* and *water.*

Test solution. Dissolve 40 mg of the substance under examination in 50 ml of *acetonitrile* and dilute to 100.0 ml with *water*.

Reference solution (a). Dissolve 4 mg, each of, *desogestrel related compound A IPRS* and *desogestrel related compound D IPRS* in 50 ml of *acetonitrile*, and dilute to 100.0 ml with *water*.

Reference solution (b). Dissolve 40 mg of *desogestrel IPRS* in 50 ml of *acetonitrile*, add 1.0 ml of reference solution (a) and dilute to 100.0 ml with *water*.

Reference solution (c). Dissolve 4 mg, each of, *desogestrel IPRS*, *desogestrel related compound B IPRS* and *desogestrel related compound C IPRS* and 8 mg, each of, *desogestrel related compound A IPRS* and *desogestrel related compound D IPRS* in 50 ml of *acetonitrile*, dilute to 100.0 ml with *water*. Dilute 1.0 ml of the solution to 100.0 ml with the solvent mixture.

Reference solution (d). Dissolve 40 mg of *desogestrel IPRS* in 50 ml of *acetonitrile* and dilute to 100.0 ml with *water.*

Chromatographic system

- a stainless steel column 25 cm x 4.6 mm, packed with octadecylsilane bonded to porous silica (5 µm),
- column temperature: 50°,
- mobile phase: a mixture of 73 volumes of *acetonitrile* and 27 volumes of *water,*
- flow rate: 1 ml per minute,
- spectrophotometer set at 205 nm,
- injection volume:15 µl.

Name	Relative retention time
Desogestrel related compound B[1]	0.16

Desogestrel related compound C[2]	0.19
11-Methylene lynestrenol[3]	0.71
Desogestrel related compound A[4]	0.96
Desogestrel	1.0
Desogestrel related compound D[5]	1.06

[1] 13-Ethyl-3-hydroxy-11-methylene-18,19-dinor-17α -pregn-4-en-20-yn-17-ol,
[2] 13-Ethyl-11-methylene-18,19-dinor-17α -pregn-4-en-20-yn-17-ol-3-one,
[3] 11-Methylene-19-nor-17 α-pregn-4-en-20-yn-17-ol,
[4]13-Ethyl-11-methylene-18,19-dinor-5α,17α -pregn-3-en-20-yn-17-ol,
[5] 13-Ethyl-11-methylenegon-4-en-17-one.

Inject reference solution (b) and (c).The test is not valid unless the resolution between the peaks due to desogestrel and desogestrel related compound A is not less than 1.3, the peak to valley ratio between the peaks due to desogestrel and desogestrel related compound D is not less than 2.0 in the chromatogram obtained with reference solution (b) and the relative standard deviation for replicate injections is not more than 5.0 per cent, for all peaks in the chromatogram obtained with reference solution (c).

Inject reference solution (c) and the test solution. In the chromatogram obtained with the test solution, the area of any peak corresponding to desogestrel related compound B and desogestrel related compound C, each of, is not more than the area of the corresponding peaks in the chromatogram obtained with reference solution (c) (0.1 per cent), the area of any peak corresponding to desogestrel related compound A and desogestrel related compound D, each of, is not more than the area of the corresponding peaks in the chromatogram obtained with reference solution (c) (0.2 per cent), the area of any peak corresponding to 11-methylene lynestrenol is not more than twice the area of desogestrel peak in the chromatogram obtained with reference solution (c) (0.2 per cent), the area of any other secondary peak is not more than the area of the desogestrel peak in the chromatogram obtained with reference solution (c) (0.1 per cent) and the sum of areas of all the secondary peaks is not more than 5 times the area of the desogestrel peak in the chromatogram obtained with reference solution (c) (0.5 per cent). Ignore any peak with an area less than 0.5 times the area of the desogestrel peak in the chromatogram obtained with reference solution (c) (0.05 per cent).

Heavy metals (2.3.13). 1.0 g complies with the limit test for heavy metals, Method B (20 ppm).

Sulphated ash (2.3.18). Not more than 0.1 per cent.

Loss on drying (2.4.19). Not more than 0.5 per cent. Dry under vacuum at a pressure not exceeding 15 mm of mercury.

Assay. Determine by liquid chromatography (2.4.14), as described under Related substances.

Inject reference solution (b) and (d). The test is not valid unless the resolution between the peaks due to desogestrel and desogestrel related compound A is not less than 1.3, the peak-to-valley ratio between the peaks due to desogestrel and desogestrel related compound D is not less than 2.0 in the chromatogram obtained with reference solution (b), the tailing factor is not more than 2.0 and the relative standard deviation for replicate injections is not more than 1.0 per cent in the chromatogram obtained with reference solution (d).

Inject reference solution (d) and the test solution.

Calculate the content of the $C_{22}H_{30}O$.

Storage. Store protected from light and moisture, at a temperature not exceeding 30°.

Solubility: Very soluble in *methanol*; freely soluble in *ethanol* and in *methylene chloride;* practically insoluble in *water.*