Datasheet Melengestrol-D₃

Reference number : CEC/MAT : 14

Date of preparation: 1994.05.31

date : 17 January 2003
source : MAT - RIVM

“Bank of Reference Standards”

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Directorate General “Science, Research and Development DG XII”
Contract MAT 1 - CT92 - 0020
MELENGESTROL-D₃

Name : 17α-Hydroxy-6-trideuteromethyl-16-methylenepregna-4,6-diene-3,20-dione
Synonym : melengestrol-D₃
Molecular formula : C₂₃H₂₇O₃D₃
Cas # : not available
Molecular weight : 357.50

Long term stability tested on 1997.09.24 : 94.4 ± 1.1 %
(storage 4°C, analysis HPLC-UV, 6 tests on 2 ampoules)

Date : 1998.01.06

Methods used for characterization

I     IR spectroscopy
II    Mass spectrometry
III   HPLC-UV spectroscopy
IV    ¹H-NMR spectrometry
V     Homogeneity and stability obtained with HPLC
I IR-SPECTROSCOPY

Instrument: Bruker IFS-55 FTIR; detector DTGS
Sampling technique: KBr-tablet.

II MASS-SPECTROMETRY

Instrument: Hewlett Packard 5989 A MS
MS-spectrum, DIP = direct inlet probe

III HPLC-UV SPECTROSCOPY

Instrument: TSP spectrasystem UV2000; resolution 2nm.
HPLC eluens: methanol/water (70:30 v/v)

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HPLC column: Lichrocart 125-4 hplc cartridge

IV $^1$H-NMR SPECTROMETRY

Instrument: FT-NMR Jeol GSX; 270 MHz, 5 mm probe, solvent CDCl$_3$

<table>
<thead>
<tr>
<th>Chemical shifts (ppm)</th>
<th>Amount of protons (multiplicity)</th>
<th>Designation</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.88</td>
<td>3 (s)</td>
<td>18-CH$_3$</td>
</tr>
<tr>
<td>1.12</td>
<td>3 (s)</td>
<td>19-CH$_3$</td>
</tr>
<tr>
<td>2.32</td>
<td>3 (s)</td>
<td>21-CH$_3$</td>
</tr>
<tr>
<td>5.22</td>
<td>2 (d)</td>
<td>16-methylene</td>
</tr>
<tr>
<td>5.90</td>
<td>2 (2s)</td>
<td>4-H en 7-H</td>
</tr>
</tbody>
</table>
V HOMOGENEITY AND STABILITY

Stability and homogeneity test of Melengestrol-D₃

**CEC/ MAT: 14**

<table>
<thead>
<tr>
<th>temp.</th>
<th>t= 0 months homogeneity ( n = 10) µg (m ± SD)</th>
<th>t=1,5 months ( n = 2) µg (m ± SD)</th>
<th>t = 3 months ( n = 2) µg (m ± SD)</th>
<th>t = 6 months ( n = 2) µg (m ± SD)</th>
<th>t= 12 months ( n = 2) µg (m ± SD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4°C</td>
<td>(108 ± 2)</td>
<td>(106 ± 2)</td>
<td>(107 ± 1)</td>
<td>(107 ± 1)</td>
<td>(104 ± 4)</td>
</tr>
<tr>
<td>20°C</td>
<td>(109 ± 1)</td>
<td>(108 ± 1)</td>
<td>(107 ± 1)</td>
<td>(103 ±1)</td>
<td></td>
</tr>
<tr>
<td>37°C</td>
<td>(105 ± 2)</td>
<td>(111 ± 1)</td>
<td>(108 ± 2)</td>
<td>(104 ± 1)</td>
<td></td>
</tr>
</tbody>
</table>

Stability test Melengestrol-d3

![Graph showing stability test Melengestrol-d3](image)