Supporting Information

A Facile Green Synthesis of 16-Dehydropregnenolone Acetate (16- DPA) from Diosgenin

Diganta Baruah^a, Ram Nath Das^a and Dilip Konwar^{*a}

*^aSynthetic Organic Chemistry Division, CSIR - North East Institute of Science & Technology,
Jorhat-785006, Assam, India*

***Email: dkonwar@yahoo.co.uk**

Characterization Data

1. **16-Dehydropregnenolone acetate (16-DPA):** White solid; 267 mg, 75%; Mp.: 172°C (lit.^{8, 13}. 169-175°C); IR (CHCl₃): 1731, 1661, 1373, 1248, 1137, 1037, 943 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 1.0 (s, 3H), 1.2 (s, 3H), 2.0 (s, 3H), 2.2 (s, 3H), 4.5 (m, 1H), 5.31 (m, 1H), 6.65 (m, 1H) ppm; ; ¹³C NMR (100MHz, CDCl₃): δ 199.9, 170.4, 159.3, 146.7, 139.9, 121.4, 73.4, 54.6, 50.4, 49.6, 37.8, 36.6, 36.5, 33.2, 30.9, 30.6, 29.8, 27.4, 21.3, 20, 19, 15.6; Mass (m/z, 100%): 357 (M⁺).
2. **Pseudodiosgenin diacetate, PDA:** White solid; 488 mg, 98%; Mp. 98°C (lit.^{8, 13} 98-100°C); IR (CHCl₃): 1736 (broad), 1373, 1250, 1025 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 0.81(s, 3H), 0.97 (d, 3H, *J* = 6.3 Hz), 1.2 (s, 3H), 1.7 (s, 3H), 2.1(bs, 6H, acetate protons), 3.9 (d, 2H, *J* = 4.5 Hz), 4.80-4.45 (m, 2H), 5.62-5.31 (m, 1H)ppm; Mass (m/z, 100%): 498 (M⁺).
3. **Diosone :** IR (CHCl₃): 1733(broad), 1453, 1375,1365, 1244,1175, 1108 cm⁻¹; ¹H NMR (300 MHz) : δ = 0.84(s,3H), 0.93(d, 3H,*J*=6.3Hz), 1.1(s, 3H), 2.0(bs, 6H, acetate protons), 2.2 (s, 3H), 3.9(d,2H, *J*=4.1Hz), 4.5 (m,2H), 5.4(m,1H) ppm; ; Mass (m/z, 100%): 530 (M⁺).

Spectra (S2-S8)

1. FT-IR Spectra on 16-DPA

Description:
Instrument Model: System 2000 Data Type: PE IR SPECTRUM Instrument Serial Number: 33208
Instrument Software Version: 4.07 IR Type: Fourier Transform Resolution: 4.0 cm⁻¹

Comments: Mr. R. N. Das.
Sample: RND--142
KBr Pellet.

Analyst: Mr. P.P. Khound

2. ^1H NMR of 16-DPA:

3. ^{13}C NMR of 16-DPA:

4. Mass spectra of 16-DPA:

Analysis Info

Analysis Name D:\sir back\Data\RRLJTRKhan436.d
Method Copy of GERD Default.ms
Sample Name 16-DPA
Comment ACETONITRILE + CH₃COOH

Acquisition Date 6/18/2013 11:59:57 AM

Operator R.KHAN
Instrument esquire3000

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	off
Mass Range Mode	Std/Normal	Scan Begin	50 m/z	Scan End	700 m/z
Capillary Exit	102.4 Volt	Skim 1	30.8 Volt	Trap Drive	28.1
Accumulation Time	3252 μ s	Averages	5 Spectra	Auto MS/MS	off

5. ^1H NMR of Pseudodiosgenin diacetate, PDA

6. ^1H -NMR spectra of Diosone:

7. FT-IR spectra of Diosone:

Description:
 Instrument Model: System 2000 Data Type: PE IR SPECTRUM Instrument Serial Number: 33208
 Instrument Software Version: 4.07 IR Type: Fourier Transform Resolution: 4.0 cm⁻¹

Comments: Ashutosh Thakur
 Sample-ASH-1
 Solvent-CHCl₃

Date: 07/23/2012