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8-HYDROXYDOTRIACONTAN-30-ONE AND OTHER CONSTITUENTS FROM *DUBOISIA MYOPOROIDES*

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Key Word Index—*Duboisia myoporoides*, Solanaceae, leaves and stems, 8-hydroxydotriacontan-30-one, dotriacontanol, tetratriacontanoic acid, betulonic acid, ursolic acid

Abstract—In addition to the known constituents betulonic acid, ursolic acid, tetratriacontanoic acid and dotriacontanol, a new compound isolated from the leaves and stems of *Duboisia myoporoides*, has been characterized as 8-hydroxydotriacontan-30-one by spectral studies

INTRODUCTION

During a large-scale isolation of the tropane alkaloids hyoscyne and hyoscyamine possessing mydriatic and antispasmodic properties, it was of interest to investigate the non-alkaloidal constituents of the leaves and stems of *Duboisia myoporoides*. We have recently reported the characterization of four novel aliphatic compounds from this plant [1]. We now report a new aliphatic hydroxyketone and four other constituents.

RESULTS AND DISCUSSION

Silica gel column chromatography of the *n*-hexane extract of the plant furnished five crystalline compounds, A–E.

Compound C, mp 76–77°, obtained in traces, had IR absorption bands at 3440 (OH), 2910, 2840, 1470, 725, 715 (long chain), 1700 (CO) and 1390 cm⁻¹ (methyl) and gave a positive 2,4-dinitrophenylhydrazine test. The mass spectrum of this compound displayed a [M]⁺ ion at *m/z* 480 suggesting the molecular formula C₃₂H₆₄O₂. The location of the carbonyl group at C-30 was achieved by the prominent α -fission ions at *m/z* 451, 57, 423, and β -fission involving McLafferty rearrangement at *m/z* 72 [2]. Similarly, the hydroxyl group was placed at C-8 due to significant ions at *m/z* 129, 351, 99 and 381. These fragmentations are shown on the structure below. The absence of a [M – 15]⁺ ion indicated the straight-chain nature of the compound [3]. The above data led to the

characterization of this compound as 8-hydroxydotriacontan-30-one (1).

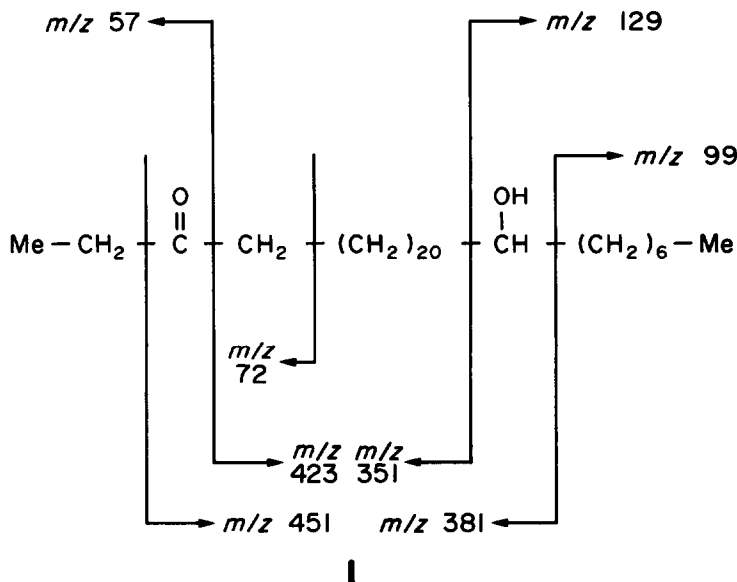
Compound A, mp 85–86°, was identified as dotriacontanol by IR, mass spectrometry, acetate, mp 76° and a comparison of the literature data [4]. Compound B, mp 90°, was identified as tetratriacontanoic acid by IR, mass spectrometry, methyl ester, mp 75° and a comparison of the literature data [5]. Compound D, mp 248–250° (dec), afforded a methyl ester, mp 165°, a sodium borohydride-reduced product, mp 285°, and was identified as betulonic acid by IR, NMR, mass spectrometry and a comparison of the literature data [6]. Compound E, mp 265°, was identified as ursolic acid by comparison with an authentic specimen (mmp, IR, MS and co-TLC).

The mass spectral and IR data of 1 were in complete agreement with the assigned structure. This compound has not previously been found in nature. The other compounds, except ursolic acid [7], constitute the first reports from this plant.

EXPERIMENTAL

Mps are uncorr. IR spectra were recorded in KBr and 60 MHz ¹H NMR spectrum in CDCl₃ with TMS as int. standard. TLC was carried out on silica gel G in at least three different solvent systems and the spots were visualized by exposure to I₂ vapour or DNPH spray.

Plant material was cultivated at the Experiment Farm,



Bangalore of this Institute, and a voucher specimen has been deposited at the Botany Department

Extraction and isolation of compounds Dried and powdered leaves and stems (3 kg) of *D. myoporoides* R Br were extracted with *n*-hexane (5 × 75 l). The solvent was removed from the hexane extract and the residue (40 g) was chromatographed over silica gel (1.2 kg, 60–120 mesh, BDH). Elution was carried out in hexane, hexane- C_6H_6 (3:1, 1:1, 1:1, 3), C_6H_6 , $C_6H_6-CHCl_3$ (3:1, 1:1, 1:1, 3), $CHCl_3$ and $CHCl_3-MeOH$ (19:1). Fractions (250 ml) were collected and monitored by TLC.

Compound A (dotriacontanol) Removal of solvent from the hexane- C_6H_6 (1:3) fractions (157–204) afforded a residue, 41 mg, mp 85–86° (Me_2CO -hexane). IR $\nu_{max} cm^{-1}$ 3420, 2910, 2840, 1460, 1390, 1050, 725, 715. MS m/z (rel int) 448 $[M - H_2O]^+$ (1), 420 $[M - 46]^+$ (2), 392 (1), 364 (1), 336 (1), 308 (1), 280 (1), 252 (1), 224 (2), 196 (2), 168 (3), 140 (5), 127 (8), 99 (10), 85 (35), 71 (55), 57 (100), 43 (80). Compound A (20 mg) was added to pyridine (1 ml), Ac_2O (1 ml) and left overnight at room temp. When worked up, it gave a residue, mp 76° ($CHCl_3-MeOH$). IR $\nu_{max} cm^{-1}$ 2910, 2840, 1730, 1460, 1370, 1250, 725, 715.

Compound B (tetratriacontanoic acid) The C_6H_6 fractions (205–300) when freed of solvent gave a residue, 228 mg, mp 90° ($CHCl_3-Me_2CO$). IR $\nu_{max} cm^{-1}$ 3500–3000, 2920, 2840, 1700, 1460, 1370, 930, 725, 715. MS m/z (rel int) 508 $[M]^+$ ($C_{34}H_{68}O_2$) (2), 448 $[M - 60]^+$ (2), 60 (55). Compound B (50 mg) was treated with $CH_2N_2-Et_2O$ overnight at room temp. The removal of solvent provided a residue, mp 75° ($CHCl_3-MeOH$). IR $\nu_{max} cm^{-1}$ 2940, 2870, 1740, 1470, 1390, 1180, 730, 720.

Compound C (8-hydroxydotriacontan-30-one) The removal of solvent from the $C_6H_6-CHCl_3$ (3:1, 1:1, 1:1, 3) eluates (326–391) provided a residue, 5 mg, mp 76–77° ($CHCl_3-Me_2CO$).

IR $\nu_{max} cm^{-1}$ 3440, 2910, 2840, 1700, 1460, 1390, 1170, 725, 715. MS m/z (rel int) 480 $[M]^+$ ($C_{32}H_{64}O_2$) (6), 451 (24), 423 (19), 381 (3), 351 (2), 129 (20), 99 (8), 85 (28), 72 (4), 71 (45), 57 (92), 55 (55), 43 (100).

Compound D (betulonic acid) The $CHCl_3$ eluates (392–535) when freed of solvent furnished a residue, 300 mg, mp 248–250° (dec) ($CHCl_3-MeOH$) which was identified by IR, MS, NMR, Me ester, mp 165°, and $NaBH_4$ -reduced product, mp 285°.

Compound E (ursolic acid) The eluates (551–560) from the $CHCl_3-MeOH$ (19:1) fractions yielded a residue, 30 mg, mp 265°, which was identified by mmp, IR and MS.

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