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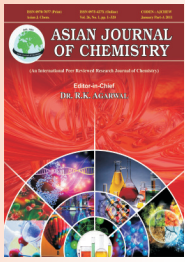

Constituents from the Root of *Lantana camara*

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Four known secondary metabolites, namely 11β,23S,24R,25-tetrahydroxyprotost-13(17)-en-3-one(alisol A-**1**), lantanilic acid (**2**), 3β-hydroxystigmast-5-en-7-one(**3**) and sitosterol (**4**), were isolated from the roots of *Lantana camara.* The structures of these compounds were established by usual spectroscopic methods. This is the first report of alisol A from *Lantana camara* roots.

Keywords: *Lantana camara*, Alisol A, Lantanilic acid, 3β-hydroxystigmast-5-en-7-one, Sitosterol.

INTRODUCTION

Lantana camara L. (Verbanaceae), commonly known as wild or red sage is the most widespread species of this genus and regarded both as a notorious weed and a popular ornamental garden plant. It is a large, evergreen, strong-smelling hairy shrub with various flower colours, red, pink, white, yellow and violet. The stems and branches are sometimes armed with prickles or spines. The plant is native to tropical and subtropical America and it is now cultivated world-wide as a hedge plant[1]. Numerous varieties and types of *L. camara* are found[1,2]. Different parts of the plant are used in traditional medicine for the treatment of various human ailments, such as ulcers, eczema, malaria and rheumatism[3,4]. Phytochemical studies undertaken by different groups of workers have resulted in the isolation of various, steroids[4,7] terpenoids[5] and alkaloids[6]. Herein, we report the isolation of compounds from the root of this plant, collected at Sana'a, Yemen. Components **1-4** were isolated from the chloroform extract of the root of this plant.

EXPERIMENTAL

TLC and preparative TLC were performed using pre-coated aluminium and glass plates with silica gel 60 F254, whereas column chromatography was carried out on silica gels 230-400 meshes. Spots and bands for compounds on TLC were detected using UV light. Proton NMR (400 MHz) andcarbon-^{13}NMR (100 MHz) spectra were recorded on JEOL JNM-ECP400 and chemical shifts in ppm were referenced to internal $CDCl_3$-CD_3OD & $CDCl_3$, respectively. CC silica gel 60 (70-230 mesh, Merck). TLC: silica gel HF_{254} and PF_{254} Merck).

Root of *Lantana camara* were collected from field in Sana'a city. A voucher specimen of JAN 002 for the plant has been deposited at the Sana'a University Herbarium.

Extraction and isolation: The air- dried powder roots (950 g) of *Lantana camara* was extracted (Soxhlet) with chloroform (2X, 26 h each) and the combined extracts evaporated to give a dark-green residue (34 g). This extract was subjected to column chromatography on silica gel with hexane containing increasing percentages of EtOAc as eluent and each collected fraction was 20 mL. Fraction 4 contain lantanilic acid (2) (10.1 mg). Fractions 8-10 (45 mg) was purified further by preparative TLC with EtOAc- acetone (6:4) to afford alisol A(1), (15.9 mg). Fractions 15-18 (85 mg) were purified further by preparative TLC with hexane-EtOAc (3:7) to afford 3-β-hydroxystigmast-5-en-7-one(3), (4.3 mg) and fractions 6-7 contain Sitosterol (4), (4.5 mg). Alisol- A (1), lantanilic acid (2), 3β-hydroxystigmast-5-en-7-one (3) and Sitosterol (4) were identified by comparison with data from previous NMR and mass spectra.

Alisol A (Compound-1): Colourless crystals, $C_{30}H_{50}O_5$; m.p.: 89-90 °C; IR (KBr, ν_{max}, cm^{-1}): 3500, 1705, 1655. ^{1}H NMR ($CDC1_3$, 400 MHz): 0.95 (3H, s), 0.97 (3H, d, J = 6.8 Hz), 1.11 (6H, s), 1.03 (3H, s), 1.09 (1H, s), 1.17 (3H, s), 1.23 (3H, s), 1.63 (1H, ddd, J = 4.0, 9.6, 13.6 Hz, 22-Ha), 1.70 (1H, d, J = 10.8 Hz, 9-H), 2.76 (1H, dd, J = 6.0, 13.6 Hz, 12-Ha), 2.96 (1H,br s, 24-H), 3.72 (1H,dd, J = 2.4, 9.6 Hz, 23-H), 3.84 (1H,ddd, J = 5.6, 10.4, 10.8 Hz, 11-H); ^{13}C NMR ($CDC1_3$, 100 MHz): 220.33 (C-3), 137.45 (C-13), 135.33 (C-17), 77.60 (C-24), 74.17 (C-25), 69.88 (C-11), 69.43 (C-23), 56.99 (C-14), 49.61 (C-9), 48.57

(C-5), 47.01 (C-4), 40.50 (C-8), 40.07 (C-22), 36.98 (C-10), 34.51 (C-12), 34.39 (C-7), 33.79 (C-2), 31.07 (C-1), 30.55 (C-15), 29.61 (C-28), 29.18 (C-16), 28.33 (C-20), 27.36 (C-26), 26.28 (C-27), 25.69 (C-19), 24.18 (C-30), 23.13 (C-18), 20.25 (C-29), 20.12 (C-6, 21).

Lantanilic acid (compound-2): Colourless needles from $CHCl_3$/MeOH; m.p.: 278-280 °C; IR (KBr, ν_{max}, cm^{-1}): 3482 (OH), 1722 (C=O), 1647 (C=C); ^1H NMR (300 MHz, $CDCl_3$): δ 0.76, 0.86,0.94,0.98, 1.00, 1.13 (each3H, s, $CH_3 \times 6$), 1.83 (3H, d, *J* = 1.2 Hz, H-5'), 2.11 (3H, d, *J* = 1.2 Hz, H-4'), 3.02 (1H, dd, *J* = 9.2, 4.6 Hz, H-18), 3.88 (1H, d, *J* = 7.8 Hz, H-25a), 4.21 (1H, d, *J* = 7.8 Hz, H-25b), 5.00 (1H, m, H- 22a), 5.37 (1H, m, H-12), 5.55 (1H, m, H-2'); ^{13}C-NMR (75 MHz,$CDCl_3$-CD_3OD): δ 34.53 (t,C-1), 29.24 (t,C-2), 98.87 (s,C-3), 40.25 (s,C-4), 50.16 (d,C-5), 19.72 (t,C-6), 30.95 (t, C-7), 38.26 (s,C-8), 41.95 (d,C-9), 35.04 (s,C-10), 23.73 (t, C-11), 122.48 (d,C-12), 143.02 (s,C-13), 41.95 (s,C-14), 27.74 (t,C-15), 24.11 (t,C-16), 50.70 (s,C-17), 39.15 (d,C-18), 45.78 (t,C-19), 30.07 (s,C-20), 37.68 (t,C-21), 75.30 (d,C-22), 27.43 (q,C-23), 18.27 (q,C-24), 67.68 (t,C-25), 17.44 (q,C-26), 25.30 (q,C-27), 177.98 (s,C-28), 33.74 (q,C-29), 27.20 (q,C-30), 166.34 (s,C-1'), 115.94 (d,C-2'), 157.10 (s,C-3'), 20.22 (q, C-4'), 26.23 (q,C-5').

3β-Hydroxystigmast-5-en-7-one (Compound-3): Colourless granules from $CHCl_3$/ MeOH; m.p.: 128-129 °C; IR (KBr, ν_{max}, cm^{-1}): 3421 (OH), 1689 (C=O); ^1H NMR (400 MHz, $CDCl_3$): δ 0.67 (3H, s, H-18), 1.17 (3H, s, H-19), 3.67 (1H, m, H-3a), 5.69 (1H, d, *J* = 1.8 Hz, H-6); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.85 (C-7), 166.10 (C-5), 124.01 (C-6), 70.52 (C-3), 54.48 (C-17), 49.93 (C-9), 49.80 (C-14), 45.87 (C-24), 45.56 (C-8), 43.08 (C-13), 41.78 (C-4), 38.81 (C-12), 38.36 (C-10), 36.28 (C-1), 36.07 (C-20), 33.93 (C-22), 31.16 (C-2), 29.69 (C-25), 28.11 (C-16), 26.54 (C-15), 26.35 (C-23), 23.03 (C-28), 21.20 (C-11), 19.79 (C-27), 18.91 (C-21), 18.76 (C-26), 17.30 (C-19), 11.96 (C- 18), 11.94 (C-29); EIMS *m/z* (rel.int.): 428 $[M]^+$ (100), 410 (20), 395 (32), 287 (18), 247 (10), 192 (30).

Sitosterol (compound-4): Colourless needles, $C_{29}H_{50}O$; m.p.: 139-140 °C;CI MS *m/z*: 414, 396, 381, 273; ^1H NMR ($CDC1_3$, 400 MHz): δ 5.340 (1H, m, H-6), 3.521 (1H, m, H-3a), 1.013 (3H, s), 0.920 (3H, d, *J* = 6.8 Hz), 0.852 (3H, d, *J* = 7.8 Hz), 0.830 (3H, s), 0.809 (3H, d, *J* = 6.8 Hz), 0.682 (3H, s); ^{13}C NMR ($CDC1_3$, 100 MHz): δ 140.48 (C-5), 121.48 (C-6), 71.68 (C-3), 56.67 (C-14), 55.94 (C-17), 50.04 (C-9), 45.75 (C-24), 42.25 (C-4 and C-13), 39.71 (C-12), 37.18 (C-1), 36.44 (C-10), 36.08 (C-20), 33.88 (C-22), 31.85 (C-2 & C-7), 31.62 (C-8), 29.09 (C-25), 28.20 (C-16), 26.02 (C-23), 24.26 (C-15), 23.02 (C-28), 21.05 (C-11), 19.79 (C-27), 19.37 (C-19), 18.99 (C-26), 18.74 (C-21), 11.96 (C-29), 11.85 (C-18).

RESULTS AND DISCUSSION

Compound 1: Obtained as colourless crystals and showed IR absorptions due to hydroxyl and cyclic ketone. The ^1H, ^{13}C NMR and DEPT spectra indicated the presence of seven tertiary methyls, eight methylenes, six methines, together with one ketocabonyl carbon and a tetra-substituted olefin. The ^{13}C NMR and DEPT spectra showed the presence of three oxygenated methine carbons (77.60, 69.88 and 69.43) together with an oxygenated quaternary carbon (74.17) in the molecule. The NMR signals at 69.43 (C23), 3.72 (1H, dd, *J* = 2.4, 9.6 Hz, 23-H), 77.60 (C24), 2.96 (1H, br s, 24-H) and 74.17 (C25), indicated the absolute configuration of the side chain as 20R, 23S, 24R. The NMR data of 1 are identical to those of alisol A[6].

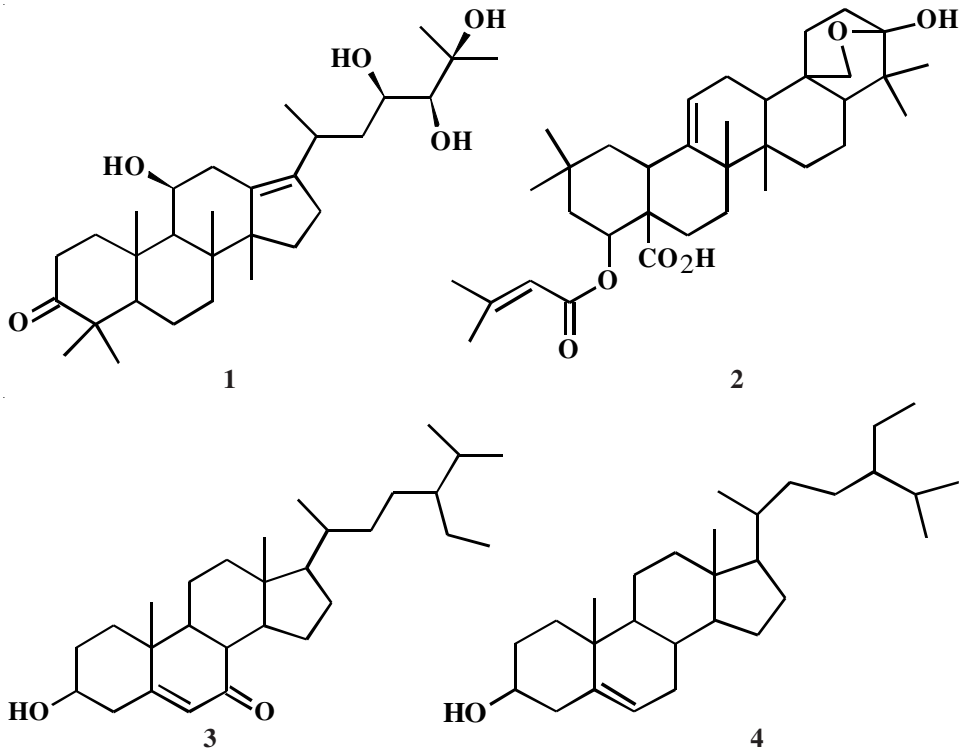

Fig. 1. Structures of compounds **1-4** isolated from *Lantana camara* roots

Compound 2: Isolated as colourless needles form. The ^{1}H NMR spectrum of **2** showed the characteristic signals for an angeloyl moiety at C- 22 [1.78 (3H, d, *J* = 1.5 Hz, H 5'), 1.97 (3H, q, *J* = 7.3 Hz, H 4'), 5.99 (1H, qq, *J* = 7.3, 1.5 Hz, H3') and 5.02 (1H, m, H 22α)]. Two signals each for one-proton doublets appeared at δ 4.20 (1H, d, *J* = 11.2 Hz) and 3.88 (1H, d, *J* = 11.2 Hz) due to two nonequivalent methylene protons. It also showed senecioyl moiety at C-22 [1.83 (3H, d, *J* = 1.2Hz, H-5'), 2.11(3H, d, *J* = 1. 2 Hz, H-4'), 5.55 (1H, m, H-3') and 5.00 (1H, m, H-22α)]. Consequently compound **2** was identified as lantanilic acid[2,5].

Compound 3: Isolated as colourless granules form. The ^{1}H NMR spectrum (400 MHz, $CDCL_3$) showed the proton of H-3 as a multiplet signal at δ 3.67 and the proton of H-6 at δ 5.69. The ^{13}C NMR showed carbonyl group signal at δ 202.85, C-3 appeared at δ 70.52 and the alkenes carbons appeared at δ 166.10 and 124.01, respectively[7].

Compound 4: Isolated as colourless needle. MS gave the molecular signal at *m/z* 414 $[M]^+$ in accordance with the molecular formula $C_{29}H_{50}O$. The ^{13}C NMR and DEPT spectra showed twenty-nine carbon signals, including six methyls, eleven methylenes, nine methines and three quaternary carbons. Compound **4** was concluded to be sitosterol by detailed comparison of NMR data with reported values for sitosterol in the literature[8] (Fig. 1).

In summary, the isolation and identification of the known compound alisol A (1) from the roots of *Lantana camara* was reported for the first time from this plant.

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