

## A new triterpene from *Salvia coccinea*

K. S. Mukherjee\*, B. Mukhopadhyay and G. Brahmachari

Department of Chemistry, Visva Bharati University, Santiniketan-731 235, India

E-mail : bodhitukun@yahoo.co.in Fax : 91-03463/262672

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The benzene extract of the whole plants of *Salvia coccinea* Linn. has resulted a new pentacyclic triterpene, methyl-1- $\beta$ -hydroxy-1-oxo-olean-12-en-28-carboxylate.

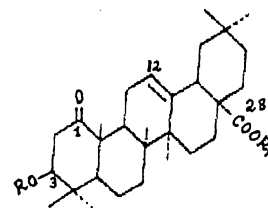
In continuation of our previous work on *Salvia coccinea*<sup>1</sup>, we report herein the isolation and characterization of a new triterpene from the benzene extract of this plant.

The concentrated benzene extract of the defatted whole plant (aerial parts and roots) of *S. coccinea* on chromatographic analysis over silica gel (60–120 mesh) using solvents with increasing polarity furnished a white solid, C<sub>31</sub>H<sub>48</sub>O<sub>4</sub> (M<sup>+</sup>, *m/z* 484), crystallized from acetone, m.p. 150–151° in chloroform eluent. It responded positively to Liebermann-Burchard test for pentacyclic triterpene. IR spectrum showed bands at  $\nu_{\max}$  (KBr) 3440 (OH), 1735 (ester carbonyl), 1720 (six membered cyclic carbonyl) and 1660 cm<sup>-1</sup> (unsaturation). <sup>1</sup>H NMR spectrum displayed signals for seven tertiary methyls at  $\delta$  0.75 (3H, s), 0.80 (3H, s), 0.85 (3H, s), 0.93 (3H, s), 0.98 (6H, s) and 1.25 (3H, s), two protons multiplet at  $\delta$  2.30 assignable to –CH<sub>2</sub>–C=O, a three proton singlet at  $\delta$  3.75 for carbomethoxy function, one proton multiplet at  $\delta$  3.52 (b., half band width 15 Hz) for a carbinol methine proton and, a vinylic proton at 5.25 (1H, m). The compound readily formed an acetate, C<sub>33</sub>H<sub>50</sub>O<sub>5</sub> (M<sup>+</sup>, *m/z* 526) (2) m.p. 140°, on treatment with acetic anhydride in pyridine and a 2,4-dinitrophenylhydrazone derivative, m.p. 170–72°. The mass spectrum of the parent triterpene is typical of  $\Delta^{12}$ -oleanene skeleton<sup>2</sup> and from the mass values of the significant peaks it is evident that the carbomethoxy group is in the D/E ring portions while keto and hydroxyl functions are in A/B ring portion.

On hydrolysis with ethanolic KOH, the parent triterpene formed an acid, C<sub>30</sub>H<sub>46</sub>O<sub>4</sub>, m.p. 225–226°,  $\nu_{\max}$  (KBr) 3400, 1720, 1695 cm<sup>-1</sup> which was found to be identical with  $\beta$ -hydroxy-1-oxo-olean-12-en-28-oic acid (virgatic acid) (3)<sup>3</sup> by direct comparison of physical and spectral data with authentic sample (m.m.p., co-IR and co-TLC. This settles the locations of keto and hydroxyl functions respectively at C-1 and C-3 positions and carbomethoxy group at C-17 position in the parent compound. These led us to formulate

this triterpene as methyl  $\beta$ -hydroxy-1-oxo-olean-12-en-28-carboxylate (1) which appears to be the first report of occurrence of this triterpene from natural source.

The structure of the compound 1 also received support from its <sup>13</sup>C NMR data (Table 1) which are comparable to those compounds having similar skeleton<sup>4</sup>.



- 1 R = H, R<sub>1</sub> = CH<sub>3</sub>  
2 R = OAc, R<sub>1</sub> = CH<sub>3</sub>  
3 R = H, R<sub>1</sub> = H

Table 1. <sup>13</sup>C NMR chemical shift of compound 1

C	$\delta_c$	C	$\delta_c$
1	218.2	17	48.4
2	36.7	18	42.6
3	75.6	19	46.6
4	47.3	20	31.5
5	55.4	21	34.8
6	19.8	22	33.7
7	34.1	23	26.8
8	40.8	24	21.1
9	50.1	25	17.5
10	37.0	26	18.1
11	23.9	27	26.3
12	123.6	28	178.2
13	144.1	29	32.6
14	42.7	30	23.8
15	28.7	OMe	51.7
16	24.9		

## Experimental

All m.p.s are uncorrected. Whole plant of *S. coccinea* was supplied by the M/s United Chemical and Allied Products, Kolkata, India. A specimen of the plant is preserved in the Natural Products Laboratory of Visva Bharati University.

Air-dried defatted powdered whole plant (1.5 kg) of *S. coccinea* was extracted with benzene in a soxhlet apparatus for 56 h. The extract was concentrated under reduced pressure and then subjected to CC on silica gel (60–120 mesh, 200 g). The chloroform eluent afforded the compound **1** (1.5 g), m.p. 150–151°, <sup>1</sup>H NMR (90 MHz, CDCl<sub>3</sub>), <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) spectral data are discussed in the text, EIMS, *m/z* 484 (M<sup>+</sup>, 20%), 469 (M-CH<sub>3</sub>, 1.5%), 425 (M-COOCH<sub>3</sub>, 25%), 262 (retro-Diels-Alder fragmentation around ring C, 100%, base peak), 221 (M-262-H, 35%), 203 (221-H<sub>2</sub>O, or 262-COOCH<sub>3</sub>, 75%), 133 (60%).

**Acetylation of triterpene 1** : The triterpene (**1**, 0.05 g) was dissolved in a solution of 5 ml acetic anhydride and 2 ml of pyridine. The reaction was allowed to stand for four days at room temperature. Then the solution was poured into cold water, extracted with ether and dried when an acetate of **1** (0.06 g), m.p. 140° was obtained.

**Hydrolysis of triterpene 1** : The triterpene (**1**, 0.5 g) was refluxed with 20% ethanolic KOH (10 ml) for 8 h, then the solvent removed, H<sub>2</sub>O added and the mixture filtered.

The residue on repeated CC over silica gel (60–120 mesh, 50 g) furnished virgatic acid (**3**, 0.07 g), C<sub>30</sub>H<sub>46</sub>O<sub>4</sub> [M]<sup>+</sup> *m/z* 470, m.p. 225–26°, *v*<sub>max</sub> (KBr) 3400, 1720, 1695 cm<sup>-1</sup>, <sup>1</sup>H NMR (90 MHz, CDCl<sub>3</sub>) δ 0.70 (3H, s), 0.85 (6H, s), 0.90 (6H, s), 0.98 (3H, s), 1.20 (3H, s) for seven tertiary methyls, δ 2.30 (2H, m) ascribed to keto methylene protons, δ 3.55 (1H, t) assignable to carbinol methine proton, and δ 5.40 (1H, m) for a vinylic proton.

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