International Journal of ChemTech Research
CODEN(USA): IJCRGG ISSN : 0974-4290
Vol.5, No.4, pp 1939-1941, April-June 2013

Crystal Structure Of Isoengelitin Isolated From Cissus quadrangularis Linn

Ayinampudi Sridhar Rao[1,2]* and Ramchander Merugu[1],

[1]Mahatma Gandhi University, Nalgonda-508 001, A.P, India
[2]University of Mississippi, MS, USA-38655.

***Corres.author: asriict@gmail.com, Tel: 8978987990.**

Abstract: The title molecule (systematic name: 5,7-dihydroxyphenyl)-3-[(2R, 3S, 4S, 5S, 6S)-3, 4, 5-trihydroxy-6-methyltetrahydro-2H-pyran-2-yloxy)chroman-4-one], $C_{21}H_{22}O_{10}$, isolated from whole plant of *Cissus quadrangularis* Linn, has it is flavanol moiety is attached with rhaminose its common name was isoengelitin. The preliminary structure of (**I**) was elucidated with the help of one-dimensional and two-dimensional NMR, and HRESIMS methods.
Key words: single-crystal X-ray study; T = 90° K; mean (C–C) = 0.003 A°.
R factor = 0.081; wR factor = 0.211; data-to-parameter ratio = 9.3

Introduction

Cissus is a genus of approximately 350 species of woody climber in the grape family Vitaceae. *Cissus quadrangularis* Linn. (Veldt Grape or Winged Treebine). *C. quadrangularis* is one of the most frequently used medicinal plants, commonly known as “bone setter” found in hotter parts of India, Ceylon, East Africa and Malaysia and Thailand. *C. quadrangularis* is used as a common food supplement in southern India. The stout fleshy *C. quadrangularis* stem is traditionally used for the treatment of gastritis, bone fractures, skin infections, constipations, eye diseases, piles, anemia, asthma, irregular menstruation, burns and wounds. *C. quadrangularis* has potent fracture healing property, antimicrobial, antiulcer, antioxidative, cholinergic activity and beneficial effect on cardiovascular diseases. The preliminary structure of (**I**) was elucidated with the help of one-dimensional and two-dimensional NMR, and HRESIMS methods.

Material and Methods:

Stems of *C. quadrangularis* Linn were obtained from University of Mississippi on 14 June 2007 and authenticated by Dr. V. Joshi at the National Center for Natural Products Research, University of Mississippi, where a voucher specimen (No. 3287**)** has been deposited. Stems powder (2.0 kg) was extracted with EtOH (3X3 L 4X4h) sonication. The combined extracts were evaporated under reduced pressure to afford a brown powder (60 g). The combined extracts were concentrated under reduced pressure to obtain dry ethanolic extract (35 g). A part of ethanol extract (30.0 g) was subjected to vacuum liquid column chromatography over RP C-18 silica gel (200 g) and eluted initially with 20/80% (W/M), 40/60% (W/M), 60/40% (W/M), 20/80% (W/M), 100% (M) and 100% DCM to give six fractions. In total fractions 20/80% (W/M) and DCM fractions having only sugars and aliphatic acids and esters mainly, remaining three fractions were interesting to subjected further chromatography, in sequence 40/60% (W/M) to gave 1 (5.0 g), 2 (6.3 g), 3 (0.5 g), 4 (X g), 5 (x.0 g) and 6 (x.0 g). Fraction 60/40% (W/M) (6.0 g) was divided into twenty fractions (60/40%-1 to 60/40%-20) after performing sephadex column with pure methanol column chromatography to yield 7 (X g), 8 (X g), 9 (x g), 10 (x g), 11 (x g), 12 (x g), 13 (x g) and 14 (x g) and Final fraction were subjected to column chromatography with normal

silica gel column with chloroform: Methanol (9: 1, 8: 2 and 7:3, V/V) to yielded 15 (x g), 16 (x g), 17 (x g), 18 (x g), 19 (x g), 20 (x g), 21 (x g) and 22(x g).

Data collection was done by the following methods: COLLECT (Nonius, 2000); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: SCALEPACK and DENZO (Otwinowski & Minor, 1997); program(s) used to solve structure: SIR97 (Altomare et al., 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

The crystal structures of **1** were determined, using data collected at T = 90°K with Mo Ka radiation on a Nonius Kappa CCD diffractometer equipped with an Oxford Cryostreamcooler.5,7-dihydroxy-2-(4-hydroxyphenyl)-3-(3,4,5,6-tetrahydroxytetrahydro-2H-pyran-2-yloxy) chroman-4-one. $C_{21}H_{25}O_{11.50}$ Unit cell parameters: a 15.2936(17) b 7.2211(7) c 18.415(2) beta 93.041(9) space group P21 $C_{21}H_{22}O_{10}\cdot 1.5\ (H_2O)$ F_{000} = 972, *Mr* = 461.41 *D*x = 1.509 Mg m-3, Monoclinic, *P*21 Cu *K*α radiation, λ = 1.54178 Å, Hall symbol: P 2yb Cell parameters from 517 reflections *a* = 15.2936 (17) Å θ = 3.6-45.6°, *b* = 7.2211 (7) Å μ = 1.06 mm^{-1}, *c* = 18.415 (2) Å *T* = 90 K, β = 93.041 (9)° Plate, colourless, *V* = 2030.8 (4) Å3 0.34 × 0.13 × 0.01 mm, *Z* = 4, CCDC 701672.

Refinement

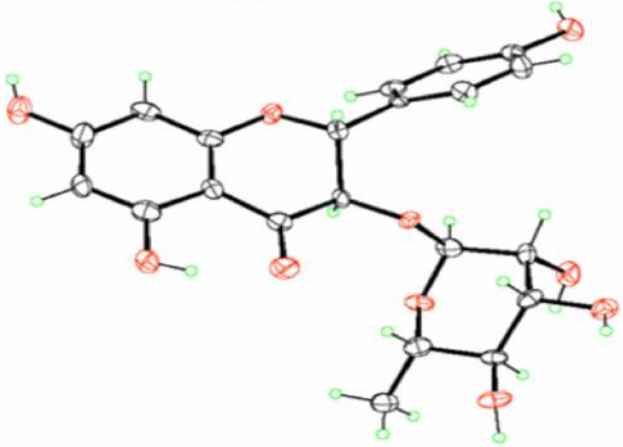
R[*F*2 > 2σ(*F*2)] = 0.080	599 parameters
w = 1/[σ2(*F*o	H-atom parameters constrained
S = 1.02	Δρmax = 0.79 e Å−3
5773 reflections	Δρmin = −0.39 e Å−3

Results and Discussion:

Crystal data

$C_{21}H_{22}O_{10}\cdot 1.5$ (H2O)	*V* = 2030.8 (4) Å3
Mr = 461.41	*Z* = 4
Monoclinic, *P*21	Cu *K*α radiation
a = 15.2936 (17) Å θ = 3.6–45.6°	0.34 × 0.13 × 0.01 mm
b = 7.2211 (7) Å μ = 1.06 mm−1	λ = 1.54178 Å
c = 18.415 (2) Å	*T* = 90 K
β = 93.041 (9)°	

The results are presented in the **Tables 1.** The Cambridge Structural Database (version 5.29, Nov. 2007) contains only one Δ5-pregnane steroid having O-substituent's at C3, C12, and C14, refcode SENKUR. SENKUR, like (I) has OH groups at C3 and C14, a benzoate at C12, and also OH groups at C8 and C17. The conformations of the A, B, and C rings in (I) and SENKUR are similar, with 18 endocyclic torsion angles differing by a mean value of 5.7°. The five-membered ring of SENKUR, however, has an envelope conformation with a different atom, C13 at the flap position. This may be a result of the fact that SENKUR has the opposite configuration at C17, with the C(O)Me substituent *α* oriented.



ORTEP Diagram of Isoengelitin

Table 1: Hydrogen-bond geometry (Å°)

D-H···A	D-H	H···A	D···A	D-H···A
O2-H2A···O11	0.84	2.04	2.743 (7)	141
O2-H2B···O11i	0.85	2.53	3.378 (6)	180
O3-H3A···O13i	0.84	2.28	3.119 (8)	177
O4-H4A···O2W	0.84	2.00	2.798 (8)	158
O7-H7A···O6	0.84	1.92	2.560 (7)	132
O10-H10···O17ii	0.84	2.00	2.776 (6)	153
O11-H11A···O2	0.84	1.91	2.743 (7)	173
O11-H11B···O12iii	0.85	1.84	2.693 (7)	180
O13-H13A···O3iii	0.84	2.48	3.119 (8)	133
O17-H17A···O16	0.84	1.83	2.563 (6)	145

Symmetry codes: (i) -x+1, y+1/2, -z+1; (ii) -x+1, y-1/2, -z+2; (iii) -x+1, y-1/2, -z+1.

References:

1. Nonius (2000). COLLECT. Nonius BV, Delft, The Netherlands.
2. Otwinowski, Z. & Minor, W. (1997). Methods in Enzymology, Vol. 276, Macromolecular Crystallography, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press
3. Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). J. Appl. Cryst. 32, 115-119.
4. Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.
5. Farrugia, L. J. (1997). J. Appl. Cryst. 30, 565.

*****