



ISOLATION OF PENTANDRAONE FROM METHANOLIC EXTRACT OF AERIAL PART OF *ZALEYA PENTANDRA*

Samina Afzal¹, Bashir Ahmad Chaudhary¹, Muhammad Uzair¹, Khurram Afzal², Tasveer Zahra Bokhari³

¹Faculty of Pharmacy, Bahauddin Zakariya University, Multan- Pakistan

²Ph.D. Scholar Bahauddin Zakariya University, Multan- Pakistan

³Institute of Biology, Bahauddin Zakariya University, Multan- Pakistan

*Corresponding Author Email: tzb_5@hotmail.com

Article Received on: 20/08/13 Revised on: 07/09/13 Approved for publication: 12/09/13

DOI: 10.7897/2230-8407.041006

IRJP is an official publication of Moksha Publishing House. Website: www.mokshaph.com

© All rights reserved.

ABSTRACT

Zaleya pentandra is a well known species in the genus *Zaleya*. It has wide traditional medicinal uses. *Zaleya pentandra* is being used for treatment of stomach diseases, respiratory tract infection and cough. For the purpose of isolation from the dried aerial parts of *Zaleya pentandra* was subjected to extraction with methanol, this combined extract was concentrated and column chromatography was then carried out. The isolation and purification yielded amorphous solid which was subjected to physical, chemical, and spectral techniques like UV, IR, ¹H-NMR, ¹³C-NMR and HREI-MS for the structure elucidation of compound. The compound was concluded as pentandraone, a novel compound isolated for the first time from the methanolic extract of aerial part of *Zaleya pentandra*.

Keywords: *Zaleya pentandra*, Column chromatography, Extraction, Pentandraone

INTRODUCTION

Zaleya pentandra, which belongs to family Aizoaceae, has about 1170 species and 128 genera, many of which are cultivated in tropical Africa, South America, West India, Mediterranean and tropical Asia. *Zaleya* is widely distributed prostrate and branched herbs. A genus of about 6 species found in Africa, Asia and Australia, only one species, *Zaleya pentandra*, found in Pakistan¹. The genus is enriched with pharmacological properties. *Trianthema decandra* showed hepatoprotective², antidiabetic activity³, antioxidant activity⁴, antibacterial activity⁵, antimicrobial activity⁶, antipyretic, analgesic and anti-inflammatory activity⁷. *Trianthema Portulacastrum* also displayed hepatoprotective activity⁸, anti-cancer property⁹, hypoglycemic, anti-hyperglycemic and hypolipidemic¹⁰, anthelmintic activity¹¹ antifungal activity and use in renal disorder¹². Literature survey of genus revealed phytochemical constituent Trianthemine, Trianthenol and Ecdysteroid reported from chloroform extract of *Zaleya portulacastrum*^{13,14} along with Flavonoids and phytoserolines and Ketone¹⁵. Many species of this genus have been reported to act as folk medicine for treating bronchial diseases, cathartic, irritant, fever, constipation, urinary tract infection as well as dissolve the kidney and bladder stone. The *Zaleya portulacastrum* is being used to decrease the size distribution, incidence and multiplicity of all the apparent cancerous cells. It can also be used as a cathartic, irritant, diuretic and also break the kidney and bladder stone¹⁶. The plant *Zaleya pentandra* is used as an astringent in snake bite and as a fodder for cattle. It's also used for malaria¹⁷. *Zaleya pentandra* is being used for stomach diseases¹⁸, respiratory tract infection and cough¹⁹. The genus *Zaleya* has diverse medicinal application that motivated us to carry out the phytochemical investigations on this species. Herein we submit the report about the isolation and characterization of a novel steroid hormone named as Pentandraone.

MATERIALS AND METHODS

General

Column chromatography has been used for the purpose of isolation with silica gel of 70-230, 230-400 mesh along with sephadex LH-20. For TLC purpose, aluminum sheets pre-coated silica gel 60 F₂₅₄ (20 × 20 cm, 0.2 mm thick; E-Merck) have been used to check the percentage purity of the compounds. The visualization of components was observed under ultraviolet light (254 and 366 nm) followed by Godine reagent and 10 % sulphuric acid were used as spraying reagent. IR spectrum was recorded by using Bruker vector-200 spectrophotometer (ν in cm⁻¹). EI-MS spectrum was recorded on Jeol JMS-600H spectrometer and HREI-MS was recorded on MET-95-XP. The ¹H-NMR spectrum was recorded by Bruker Avon-300 MHz instruments by using TMS as internal standard. The values of chemical shift were reported in ppm (δ) units and the coupling constants (J) they were recorded in Hz. The ¹³C-NMR spectrum was also recorded on Bruker Avon-300 MHz instruments.

Plant collection

The whole plant of *Zaleya Pentandra* was collected from Peruwal (District Khanewal) and identified by Professor Dr. Altaf Ahmed Dasti, plant Taxonomist, Institute of pure and applied biology, Bahauddin Zakariya University, Multan, Pakistan; whereas voucher specimen fl. P.235/5 for *Zaleya pentandra* was deposited.

Isolation

The freshly collected whole plant material of *Zaleya Pentandra* (1000 g) was shade dried ground and extracted successively with methanol (3 × 6 L) at room temperature for 24 hours. The combined methanolic extract was concentrated under vacuum on Rota vapor model no. Buchi-rotavapor R.200 to yield dark brown crude extracts (35 g) which was labeled as ZPM. The methanolic extract (35 g) was then fractionated with column chromatography over silica gel using step wise elution with chloroform : methanol : H₂O (80 : 20 : 2) by increasing the polarity of mobile phase-Five fractions

(1-5) were obtained. The fraction 3 (245 mg) from extract of *Zaleya pentandra* was further fractionated by using column chromatography where mixture of Chloroform: methanol: water. (85: 15: 1) was used as eluent. Nine fractions were obtained. The analysis of fraction 5 affords a pure compound Pentandraone (11.4 mg). The results of the extraction along with the abbreviations used for methanolic extracts are given in Table 4.

Physical and spectroscopical data of the isolated compound Pentandraone

Amorphous solid (11.4 mg), UV (MeOD) λ_{\max} nm (log ϵ) = 275(0.17) IR ν_{\max} (KBR) cm^{-1} = 2957, 2926, 2855, 1729, 1282 $^1\text{H-NMR}$ (MeOD, 300 MHz): δ 7.59 (1H, d, J = 2.7 Hz, H-1), 7.60 (1H, dd, J = 3.0, 6.0 Hz, H-2), 7.62 (1H, dd, J = 1.2, 3.1, Hz, H-3), 7.61(1H, d, J = 3.3 Hz, H-4) 2.27 (1H, t, J

= 7.2, Hz, H-9), 1.55 (2H, dt, J = 9.0, 8.1 Hz, H-11), 1.58 (2H, t, J = 8.2 Hz, H-12), 1.60 (2H, dt, J = 6.9, 8.3 Hz, H-15), 1.63 (2H, dt, J = 6.8, 4.2, Hz, H-16), 2.32 (2H, q, J = 7.5 Hz, H-21), 0.97 (3H, t, J = 0.9, Hz, H-22), 4.20 (1H, dt, J = 5.4, 6.6 Hz, H-23), 2.25 (2H, dd, J = 5.9, 6.2 Hz, H-24), 4.16 (1H, q, J = 12.0 Hz, H-25), 3.89 (2H, q, J = 1.5 Hz, H-27), 0.86 (3H, t, J = 2.8 Hz, H-28) $^{13}\text{C-NMR}$ (MeOD, 75.4MHz): δ 129.3 (C-1), 129.4(C-2), 129.6 (C-3), 129.8 (C-4), 132.5 (C-5), 129.4 (C-6), 132.3 (C-7), 35.3 (C-8), 48.7 (C-9), 132.5 (C-10), 26.4 (C-11), 30.1 (C-12), 132.0 (C-13), 132.4 (C-14), 30.4 (C-15), 30.6 (C-16), 136.8 (C-17), 14.0 (C-18), 25.5 (C-19), 130.3 (C-20), 20.1 (C-21), 26.2 (C-22), 68.5 (C-23), 35.1(C-24), 70.1 (C-25), 169.3 (C-26), 66.6 (C-27), 11.4 (C-28) EI-MS m/z (rel.int) 57.1, 91.1, 206.1, 293.1, 418. HR-EI-MS m/z : 418.2 $[\text{M}+\text{H}]^+$ Calculated for $\text{C}_{28}\text{H}_{34}\text{O}_3$: 418).

Table 1: Results of the extraction of the plants *Zaleya pentandra*

Plant Name	Part Used	Solvent	Weight of Extract (g)	Abbreviation for the extracts
<i>Zaleya pentandra</i>	Aerial parts (1000 g)	Methanol	35	ZPM

Table 2: ^{13}C (75.4 MHz) and $^1\text{H-NMR}$ (300 MHz) Spectral Data of Compound "Pentandraone"

Carbon No.	Multiplicity DEPT	^{13}C - NMR (δ)	H- NMR (δ)	J. Value
C-1	CH	129.3	7.59 d	(J = 2.7, Hz, H-1)
C-2	CH	129.4	7.60 dd	(J = 3.0, 6.0 Hz, H-2)
C-3	CH	129.6	7.62 dd	(J = 1.2, 3.1, Hz, H-3)
C-4	CH	129.8	7.61 d	(J = 3.3, Hz, H-4)
C-5	C	132.5	-	-
C-6	C	129.4	-	-
C-7	CH	132.3	5.31 s	J = (1H, s, H-7)
C-8	C	35.3	-	-
C-9	CH	48.7	2.27 t	(J = 7.2, Hz, H-9)
C-10	C	132.5	-	-
C-11	CH ₂	26.4	1.55 dt	(J = 9.0, 8.1, Hz, H-11)
C-12	CH ₂	30.1	1.58 t	(J = 8.2, Hz, H-12)
C-13	C	132.0	-	-
C-14	C	132.4	-	-
C-15	CH ₂	30.4	1.60 dt	(J = 6.9 Hz, 8.3, H-15)
C-16	CH ₂	30.6	1.63 dt	(J = 6.8, 4.2 Hz, H-16)
C-17	C	136.8	-	-
C-18	CH ₃	14.0	0.89 s	-
C-19	CH ₃	25.5	0.90s	-
C-20	C	130.3	-	-
C-21	CH ₂	20.1	2.32 q	(J = 7.5 Hz, H-21)
C-22	CH ₃	26.2	0.97 t	(J = 0.9, Hz, H-22)
C-23	CH	68.5	4.20 dt	(J = 5.4, 6.6 Hz, H-23)
C-24	CH ₂	35.1	2.25 dd	(J = 5.9, 6.2 Hz, H-24)
C-25	CH	70.1	4.16 q	(J = 12.0 Hz, H-25)
C-26	C	169.3	-	-
C-27	CH ₂	66.6	3.89 q	(J = 1.5, Hz, H-27)
C-28	CH ₃	11.4	0.86 t	(J = 2.8, Hz, H-28)

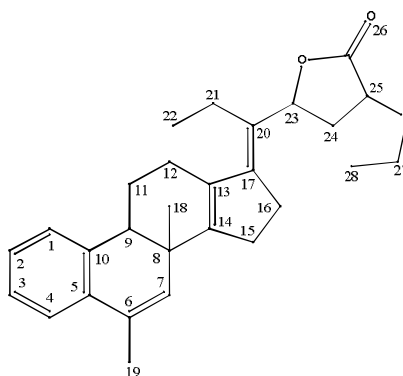


Figure 1: Structure of compound (Pentandraone: $\text{C}_{28}\text{H}_{34}\text{O}_3$: 418)

RESULTS AND DISCUSSION

The isolated compound was obtained as amorphous solid from the methanolic extract of *Zaleya Pentandra*. The IR spectrum of compound, the absorption bands at 1726 cm^{-1} is due to carbonyl group function. The stretching was observed at 2855 cm^{-1} due to $\text{Sp}^3\text{ C-H}$ and at 2926 cm^{-1} indicated the presence of $\text{Sp}^2\text{ C-H}$. From the study of mass spectrum, the molecular formula was calculated as $\text{C}_{28}\text{H}_{34}\text{O}_3$ through HREIMS showing molecular ion peak $[\text{M} + \text{H}]^+$ at m/z 418 (calculated for $\text{C}_{28}\text{H}_{34}\text{O}_3$, 418). The UV spectrum in MeOH displayed λ_{max} at 275 which showed the presence of conjugation and unsaturation system in molecule. The $^1\text{H-NMR}$ spectrum of compound has displayed a peak for aromatic proton at δ 7.59 (1H, d, $J = 2.7\text{ Hz}$), 7.60 (1H, dd, $J = 3.0, 6.0\text{ Hz}$), 7.62 (1H, dd, $J = 1.2, 3.1\text{ Hz}$), 7.61 (1H, d, $J = 3.3\text{ Hz}$), hydrogen belonging to lactone appeared at δ 4.20 (1H, dt, $J = 5.4, 6.6\text{ Hz}$), δ 4.16 (1H, q, $J = 12.0\text{ Hz}$), a singlet of alkene proton appeared at δ 5.31 (1H, s), aliphatic proton also appeared at δ 1.18. The methyl protons gave signal at δ 0.86 (3H, t, $J = 2.8\text{ Hz}$), δ 0.89 (3H, s), 0.90 (3H, s) δ 0.97 (3H, t, $J = 0.9\text{ Hz}$). The $^{13}\text{C-NMR}$ spectrum of the compound revealed twenty eight carbon atoms consisting of four methyl, eight methines, seven methylenes, 9 quaternary carbon items. The downfield signal at δ 129.4-132.5 showed the presence of aromatic carbon. The downfield signals found at δ 169.3 clearly indicated the presence of $\text{C}=\text{O}$ carbon. The presence of the lacton carbons was indicated at δ 70.1. The values of $^{13}\text{C-NMR}$ (75.4 MHz) and $^1\text{H-NMR}$ (300 MHz) spectral data of compound "Pentandraone" are shown as follows. (Table 2) On the basis of spectral data, the structure of compound was established as [3-ethoxy - dihydro-5-((1E)-1-(11, 12, 15, 16 -tetrahydro-6, 8-dimethyl-8H-cyclopenta (a) phenanthren-17 (9H) -ylidene) propyl) furan-2(3H)-one] and it was found to be a novel natural product. It was named on the basis of the species as pentandraone. (Figure 1)

ACKNOWLEDGEMENT

The project was supported by B. Z. University Multan, Pakistan. We also wish to acknowledge the technical support of H.E.J. Research Institute of Chemistry, International Center for Chemical and Biological Sciences (ICCBS), University of Karachi-75270, Karachi, Pakistan.

REFERENCES

- Nasir E and Ali SI. Flora of Pakistan. Shamim Printing Press Karachi, Pakistan; 1986. p. 1, 88, 99, 101.
- Singaravel Sengottuvelu, Duraisamy Srinivasan, Rasilingam Duraisami, Jothivel Nandhakumar, Mani Vasudevan, Thangavel Sivakumar. Hepatoprotective activity of *Trianthema decandra* on carbon tetrachloride induced hepatotoxicity in rats. International Journal of Green Pharmacy 2008; 122-125.
- Meenakshi Periasamy, Bhuvaneshwari Rajendran, Ahalliya RM, Thirumoorthi Lakshmanan, Chinna GD, Janardhanan Jiji, Kannianappan GV. Antidiabetic activity of ethanolic extract of *Zaleya decandra* in alloxan-induced diabetic rats. Applied biochemistry and biotechnology

- 2010; 162(4): 1153-1159. <http://dx.doi.org/10.1007/s12010-009-8871-x> PMID:19957208
- Balamurugan G, Muthusamy P. Observation of the hepatoprotective and antioxidant activities of *Trianthema decandra* Linn. (Vallai Sharunnai) roots on carbon tetrachloride-treated rats. Bangladesh Journal of Pharmacology 2008; 3: 83-89. <http://dx.doi.org/10.3329/bjp.v3i2.890>
- Radfar M, Sudarshana M, Kavitha U, Satish S, Niranjan MH. Evaluation of antibacterial and antifungal activity of root and root callus extracts of *Zaleya decandra* L. African Journal of Biotechnology 2012; 11(2): 510-515.
- Jaswanth A, Jagannathan K, Robert SH, Loganathan V, Manimaran S, Ruckmani K. Antibacterial Activity of root extract of *Trianthema decandra*. Ancient science of life 2002; 21: 158-159.
- Geethalakshmi R, Sarada DVL, Ramasamy K. *Trianthema decandra* L.: A review on its phytochemical and pharmacological profile. International journal of engineering, science and technology 2010; 2(5): 976-979.
- Banu GS, Kumar G, Murugesan AG. Ethanolic leaves extract of *Trianthema portulacastrum* L. Ameliorates aflatoxin b1 induced hepatic damage in rats. Indian J Clin Biochem 2009; 24: 250-6. <http://dx.doi.org/10.1007/s12291-009-0047-5> PMID:23105844 PMID:PMC3453304
- Bhattacharya S, Chatterjee M. Protective role of *Trianthema portulacastrum* against diethylnitrosamine induced experimental hepatocarcinogenesis. Cancer Lett 1998; 129: 7-13. [http://dx.doi.org/10.1016/S0304-3835\(98\)00085-8](http://dx.doi.org/10.1016/S0304-3835(98)00085-8)
- Anreddy RN, Porika M, Yellu NR, Devsarakonda RK. Hypoglycemic and hypolipidemic activities of *Trianthema portulacastrum* Linn. Plant in normal and alloxan induced diabetic rats. Int J Pharmacol 2010; 6(2): 129-33. <http://dx.doi.org/10.3923/ijp.2010.129.133>
- Mahmood Adeel, Mahmood Aqeel, Shaheen Hamayun, Qureshi RA, Sangi Yasmin, and Gilani SA. Ethno medicinal survey of plants from district Bhimber Azad Jammu and Kashmir, Pakistan. Journal of Medicinal Plants Research 2011; 5: 2348-2360.
- Karim M, Ashraf N, Kalam A, Jahan N, Jafri MA and Ahmad G. Effects of Bishkappa (*Trianthema portulacastrum* Linn.) leaves extract in adriamycin induced nephrotic syndrome. International Journal of Green Pharmacy 2011; 5(4): 329. <http://dx.doi.org/10.4103/0973-8258.94357>
- Shivhare MK, Singour PK, Chaurasiya PK and Pawar RS. *Trianthema portulacastrum* Linn. (Bishkappa). Pharmacogn Rev 2012; 6(12): 132-140. PMID:23055639 PMID:PMC3459455
- Banerji A, Chintalwar GJ, Joshi NK, Chadha MS. Isolation of ecdysterone from Indian plants. Phytochemistry 1971; 10: 2225-6. [http://dx.doi.org/10.1016/S0031-9422\(00\)97227-3](http://dx.doi.org/10.1016/S0031-9422(00)97227-3)
- Kokpol U, Wannachet Isara N, Tip Pyang S, Chavasiri W, Veerachato G, Simpson J, and Weavers RT. A C methylflavone from *Trianthema portulacastrum*. Phytochemistry 1997; 44(4): 719-722. [http://dx.doi.org/10.1016/S0031-9422\(96\)00560-2](http://dx.doi.org/10.1016/S0031-9422(96)00560-2)
- Ahmad J, Farooqui AH and Ahmad S. *Trianthema portulacastrum* L., an Herbal Drug for the Cure of Edema. Journal of Herbs, Spices and Medicinal Plants 2000; 7(2): 65-70. http://dx.doi.org/10.1300/J044v07n02_07
- Seifu T. Ethno botanical and ethno pharmaceutical studies on medicinal plants of Chifra District, Afar Region, North Eastern Ethiopia, Doctoral dissertation, Addis Ababa University; 2004.
- Hameed M, Ashraf M, Al Quriyani F, Nawaz T, Ahmad MSA, Younis A and Naz N. Medicinal flora of the Cholistan desert: a review. Pakistan journal of Botany 2011; 43: 39-50.
- Qasim M, Gulzar S and Khan MA. Halophytes as medicinal plants. In NAM Meeting in Denizli, Turkey; 2011.

Cite this article as:

Samina Afzal, Bashir Ahmad Chaudhary, Muhammad Uzair, Khurram Afzal, Tasveer Zahra Bokhari. Isolation of pentandraone from methanolic extract of aerial part of *Zaleya pentandra*. Int. Res. J. Pharm. 2013; 4(9):21-23 <http://dx.doi.org/10.7897/2230-8407.041006>

Source of support: Nil, Conflict of interest: None Declared