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Isolation of pentonic acid-3-deoxy-4-lactone and peganine-N-oxide from *Adhatoda vasica*

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Present paper deals with the isolation of two compounds namely pentonic acid-3-deoxy-4-lactone and peganine-N-oxide alongwith vasicine from the methanol extract of aerial parts of *Adhatoda vasica*. Pentonic acid-3-deoxy-4-lactone has been isolated for the first time from nature and peganine-N-oxide is reported first time from this plant. The structures of compounds were elucidated by spectroscopic methods.

Keywords: *Adhatoda vasica, Justica adhtoda,* Adosa, pentonic acid-3-deoxy-4-lactone, peganine-N-oxide.

Introduction

Adhatoda vasica or *Justica adhtoda,* is commonly known as 'Vasaka' or 'Adosa'. It is a small, evergreen shrub growing many regions of India. It is widely used for the treatment of cough, asthma, phlegm, bleeding hemorrhoids, for both adults and youth[1].

Results and discussion

Chromatographic separation of the methanol extract of dried and powdered aerial part of *Adhatoda vasica* yielded pentonic acid-3-deoxy-4-lactone, aziridine-type base peganine-N-oxide along with vasicine. Pentonic acid-3-deoxy-4-lactone and peganine-N-oxide have been isolated first time from this plant. Earlier peganine-N-oxide (**3**) was isolated from epigeal part of *Nitraria komarovii*[2]. All the compounds were identified with the help of modern spectroscopic techniques.

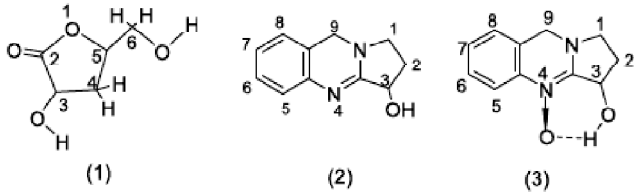

Fig. 1. Structures of compounds **1**-**3**.

Experimental

The NMR spectra were recorded in CD_3OD on JEOL AL-300 MHz FTNMR instrument. Mass spectra (ESI MS) were recorded on Water Xevo G2S Q-TOF spectrometer using MeOH solvent. IR spectra were obtained by Bruker FT-IR spectrophotometer. Melting points were uncorrected.

Extraction and isolation:

The aerial parts of *Adhatoda vasica* was air-dried for 14 days and then crushed to obtain plant material (2 kg). Then above material was soaked in MeOH for 2 days. After that filtrate was concentrated using rotary evaporator at 45°C *in vacuo* to obtain 90 g crude extract. The crude extract was dissolved in distilled H_2O and first extracted with hexane and then with EtOAc. Then ethylacetate extract (50 g) was loaded on silica gel eluted with hexane-ethylacetate mixtures. Many fractions were collected, from which alike fractions were mixed. This fraction was again chromatographed on silica gel, using $CHCl_3$ and MeOH as eluent. Fraction eluted with $CHCl_3$-MeOH (90:10) was purified by preparative TLC to afforded pentonic acid-3-deoxy-4-lactone (**1**), vasicine (**2**) and peganine-N-oxide (**3**).

*Pentonic acid-3-deoxy-4-lactone (**1**):* 20 mg, colourless oil, *m/z* 132.11; HR-ESI-MS *m/z* 133.0518 [M+H]+ (calcd. for $C_5H_9O_4$ 133.04); IR ν_{max} (KBr): 3500 (OH), 2800 (CH_2), 1740, 1200 cm^{-1}; ^1H NMR (300 MHz, CD_3OD): δ 2.32 (1H, H-4, dd, *J* 18, 2.6 Hz), 2.89 (1H, H-4, dd, *J* 18, 6.9 Hz), 3.65–3.78 (2H, H-6, m), 4.35 (1H, dd, H-5, *J* 6.9, 3.3 Hz), 4.43 (1H, H-3, m); ^{13}C NMR (CD_3OD, 75.45 MHz); δ 39.16 (CH_2–), 62.51, 69.70, 90.19, 178.68 (C=O). Further it was identified as 3-hydroxy-5-(hydroxymethyl)-dihydro-furan-2(3*H*)-one or pentonic acid-3-deoxy-γ-lactone.

*Vasicine (**2**):* 20 mg, white crystals, m.p. 212–213°C (Lit.[3,4] 213–214°C); HR-ESI-MS *m/z* 189.038 $[M+H]^+$ (calcd. for $C_{11}H_{13}N_2O$ 189.05); UV (EtOH) λ_{max} 213, 218, 289 nm; ^1H NMR (300 MHz, CD_3OD): δ 1.98 (1H, m, H-2), 2.42 (1H, m, H-2), 3.30 (1H, m, H-1), 3.45 (1H, m, H-1), 4.67 (3H, m, H-9, 3), 6.96–7.04 (3H, m, H-6, 7, 8), 7.17 (1H, m, H-5); ^{13}C NMR (CD_3OD, 75.45 MHz): δ 30.54, 47.70, 50.02, 72.41, 120.39, 123.38, 126.02, 127.38, 129.48, 141.73, 164.50.

*Peganine-N-oxide (**3**):* 15 mg, white crystals, m.p. 205–207°C (Lit.[2] 207–208°C); MS *m/z* 204 $(M)^+$ $C_{11}H_{12}N_2O_2$; IR (KBr): 770 (*o*-disubstituted benzene ring), 1230, 1265, 1464, 1511, 1586, 1631 (C-C, C-N), 2862, 2942, 3150 (OH) cm^{-1}; ^1H NMR (300 MHz, CD_3OD): δ 2.12 (1H, m), 2.62 (1H, m), 3.60–3.70 (2H, m), 4.74 (2H, t), 5.04 (1H, m), 7.09–7.29 (4H, m, H-5,6,7,8); ^{13}C NMR (CD_3OD, 75.45 MHz): δ 30.17 (CH_2-), 47.53, 51.55. 72.50, 118.58, 119.16, 127.97, 128.12, 130.31, 133.85, 164.73.

Conclusions

We have isolated pentonic acid-3-deoxy-4-lactone and peganine-N-oxide from the methanolic extract of aerial parts of *A. vasica*. It is first report of isolation of these compounds from this plant.

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Supporting Information

Files are attached.

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