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## 7-HYDROXYECHINOZOLINONE, A NEW ALKALOID FROM THE FLOWERS OF *ECHINOPS ECHINATUS*

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**ABSTRACT.**—A new minor alkaloid from the flowers of *Echinops echinatus* has been identified as 7-hydroxy-3-(2-hydroxyethyl)-(3*H*)-quinazolin-4-one and named 7-hydroxyechinozolinone **1**.

Earlier work on *Echinops echinatus* Roxb. (Compositae) resulted in the isolation of several compounds, viz., a flavone glycoside (1), alkaloids (2), triterpenoids, the antifeedant compound ethyl palmitate (3), and flavonoids (4). So far there have been no chemical investigations reported on alkaloids from the flowers of this plant. The basic fraction of the flowers led to the characterization of a new minor alkaloid, 7-hydroxy-3-(2-hydroxyethyl)-(3*H*)-quinazolin-4-one **1**, as its hydrochloride.

The flowers of *E. echinatus* were extracted with MeOH, and the CHCl<sub>3</sub> part of a pH gradient extraction, on chromatographic separation, afforded compound **1**, [M]<sup>+</sup> *m/z* 206, as an amorphous base. Compound **1** was found to be homogeneous on tlc and by ms. Its ir (KBr) absorption bands at 1658 and 1605 cm<sup>-1</sup> and the uv spectral data at λ max 232, 266, and 340 nm were similar to those of (3*H*)-quinazolin-4-one (5). The ir spectrum of **1** showed the presence of an OH group (3300 cm<sup>-1</sup> br), and the uv spectrum on addition of NaOMe was shifted to λ max 248, 292, and 384 nm, suggesting the presence of a phenolic hydroxyl group. The <sup>1</sup>H-nmr spectrum of **1** (CD<sub>3</sub>OD, 100 MHz) clearly showed the presence of a β-hydroxyethyl moiety (δ 4.15, 2H, *t*,

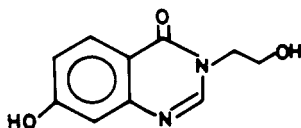
*J* = 5 Hz; δ 3.58, 2H, *t*, *J* = 5 Hz). The presence of a 7-hydroxy group was demonstrated by the appearance of a doublet at δ 7.82 (*J* = 8.5 Hz) due to the H-5 and a double doublet at δ 7.54 (*J*<sub>5,6</sub> = 8.5 Hz and *J*<sub>6,8</sub> = 2.65 Hz) due to H-6. The H-8 appeared as a doublet (*J* = 2.65 Hz) at δ 7.50 due to meta coupling with the H-6. The H-2 appeared as a sharp singlet at δ 8.86.

During the literature survey, it was found that the H-2 signal of febrifugine, a 3-substituted (3*H*)-quinazolin-4-one derivative having antimalarial activity, appeared at δ 8.91 in its hydrochloride (6). This was further confirmed by the <sup>1</sup>H-nmr study of synthetic echinozolinone hydrochloride, in which H-2 appeared as a singlet at δ 8.90 (unpublished results). This fact was well corroborated with the H-2 chemical shift value of compound **1** as its hydrochloride.

The eims ion peaks at *m/z* 135 and 107, due to retro-Diels-Alder fragmentations (2), are consistent with structure **1**. The other fragment ion peaks of compound **1** at *m/z* 189, 188, 162, and 161 were derived in the pathways similar to those of echinozolinone (2). Compound **1** is, therefore, 7-hydroxy-3-(2-hydroxyethyl)-(3*H*)-quinazolin-4-one hydrochloride, and is a new alkaloid to which the trivial name of 7-hydroxyechinozolinone has been given.

### EXPERIMENTAL

**GENERAL EXPERIMENTAL PROCEDURES.**—Ir spectra were determined on a Perkin-Elmer 299 B Ft-ir spectrometer. <sup>1</sup>H-nmr spectra were recorded at 80 MHz with TMS as internal standard on Varian FT-80A or Jeol FX-100 spec-



**1**

trometers. Eims was obtained on JMX DX-300 or Finnigan Mat 1020 GC/MS instruments at 70 eV. Si gel 60–120 mesh (BDH, India) was used for cc and Si gel plates (0.25 mm thickness) prepared from Si gel (BDH, India) were used for tlc. The tlc solvent systems used were: (A) CHCl<sub>3</sub>-MeOH (9:1); (B) CHCl<sub>3</sub>-MeOH (17:3); and (C) CHCl<sub>3</sub>-MeOH (4:1).

**PLANT MATERIAL.**—The plant material was collected from Kurukshetra, India, and identified as *E. echinatus* by our Botany Division where a voucher specimen (No. 3507) has been deposited.

**ISOLATION.**—The fresh flowers (1 kg) were shade-dried and crushed. The crushed material was then extracted with cold MeOH (7 × 10 liters) in a percolator. The crude extract was concentrated to 200 ml and then diluted with an equal volume of H<sub>2</sub>O. The solution was then acidified with aqueous HCl (3%) and kept overnight at 4°. The solution was filtered to remove suspended material, and the filtrate was extracted with CHCl<sub>3</sub> at different pH values (3–6.5). The solution was then adjusted to pH 8.5 with 25% aqueous NH<sub>3</sub> and extracted with CHCl<sub>3</sub> (3 × 400 ml). The CHCl<sub>3</sub> solutions of the acidic part showing similar spots on tlc analysis were pooled and distilled to give residue A (0.1 g). Similarly the basic CHCl<sub>3</sub> parts were pooled to give residue B (0.18 g).

**ISOLATION OF 1.**—Residue A was chromatographed over Si gel, and the column was eluted with *n*-C<sub>6</sub>H<sub>14</sub>, C<sub>6</sub>H<sub>6</sub>, C<sub>6</sub>H<sub>6</sub>-CHCl<sub>3</sub> (2:1), C<sub>6</sub>H<sub>6</sub>-CHCl<sub>3</sub> (1:1), C<sub>6</sub>H<sub>6</sub>-CHCl<sub>3</sub> (1:2), CHCl<sub>3</sub>, and CHCl<sub>3</sub>/MeOH of increasing polarities. The CHCl<sub>3</sub>-MeOH (9:1) eluents gave a pale colored solid which was further purified by preparative tlc (Si

gel) using CHCl<sub>3</sub>-MeOH (17:3) to afford **1** as an amorphous solid (0.009 g): tlc (Si gel) *R<sub>f</sub>* 0.28 [CHCl<sub>3</sub>-MeOH (17:3)]; ir  $\nu$  max (KBr) cm<sup>-1</sup> 3300, 2935, 2882, 1662, 1618, 1572, 1485, 1390, 1360, 1340, 1315, 1280, 1253, 1212, 1120, 1050, 925, 840;  $\lambda$  max (MeOH) 232 ( $\epsilon$  10109), 266 ( $\epsilon$  930), 340 ( $\epsilon$  1009) nm; eims *m/z* (rel. int.) [M]<sup>+</sup> 206 (13), 189 (89), 188 (93), 162 (25), 161 (5), 135 (47), 107 (20). Elemental analysis found C 58.11, H 4.91, N 13.50; C<sub>10</sub>H<sub>10</sub>N<sub>2</sub>O<sub>3</sub> requires C 58.25, H 4.85, N 13.59%.

Residue B on chromatography over Si gel afforded echinopsine and echinopsidine, identified by direct comparison with the authentic samples (2).

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