Volume 50 Number 3 • July 2014

Chemistry of Natural Compounds

ХИМИЯ ПРИРОДНЫХ СОЕДИНЕНИЙ
(Khimiya Prirodnykh Soedinenii)

Translated from Russian

Springer

10600 • ISSN 0009-3130
50(3) 399-588 (2014)

CONTENTS

(continued)

Engl./Russ.

Chemistry of Natural Compounds, Vol. 50, No. 3, July, 2014 [Russian original No. 3, May–June, 2014]

BIOLOGICALLY ACTIVE COMPOUNDS FROM *Climacoptera obtusifolia*

G. A. Seitimova,[1] B. K. Eskalieva,[1*] G. Sh. Burasheva,[1]
and M. Iqbal Choudhary[2]

Arid soils cover 34% of Kazakhstan. A multitude of halophytes grow on them. The chemistry of halophytes is of significant scientific and practical interest. Mainly salt-accumulating plants of the family Chenopodiaceae, which is represented in Kazakhstan by 47 genera and 218 species [1–3], exhibit halophilicity. These include plants of the genus *Climacoptera*, of which 14 species grow in Kazakhstan [4–7].

Herein we communicate results for the chemical composition of *C. obtusifolia* collected during flowering in Almaty Region. An analysis of the constituents detected flavonoids (1.4%), saponins (2.3%), amino acids (2.7%), phenolic acids (1.8%), carbohydrates (2.5%), and coumarins (0.1%).

Biologically active compounds from this plant were studied by extracting ground raw material with EtOH (70%) in a raw material–extractant ratio of 1:6–8 for 3 d at room temperature. The aqueous EtOH extract was concentrated. The concentrate was worked up successively with $CHCl_3$, EtOAc, and *n*-BuOH with subsequent condensing of each fraction. Column chromatography over silica gel of the $CHCl_3$ fraction isolated **1–4** upon elution by $CHCl_3$–hexane (9:1 and 8:2). Biologically active compounds were isolated from the *n*-BuOH extract using absorption-distributive chromatography over a nanosorbent produced by carbonization of plant raw material (rice husks) at the Institute of Combustion Problems, Al-Farabi Kazakh Nat. Univ. [7]. The eluents were H_2O and aqueous EtOH in various ratios. Thus, compound **5** was isolated pure from the *n*-BuOH fraction.

The isolated compounds were identified based on physicochemical data and comparisons with the literature as stigmasterol (**1**), $C_{29}H_{28}O$, FAB-MS *m/z* 412 [7] and dehydrodiconiferylaldehyde (**2**), $C_{20}H_{21}O_6$, EI-MS *m/z* $[M]^+$ 356, white amorphous compound, $[\alpha]_D^{25}$ +20.0° (*c* 0.1, MeOH), mp 205°C (dec.), R_f 0.5 ($CHCl_3$–MeOH, 9.3:0.7).

The IR spectrum of **2** exhibited absorption bands for CHO stretching vibrations (1670 cm^{-1}) and skeletal benzene ring vibrations (1608, 1505). ^1H NMR spectrum (300 MHz, $CDCl_3$, δ, ppm, J/Hz): 6.90 (s, H-2), 6.90 (s, H-5), 6.90 (s, H-6), 5.65 (d, J = 7.9, H-7), 3.70 (m, H-8), 3.96 (s, H-9), 7.05 (d, J = 1.3, H-2′), 7.13 (d, J = 1.3, H-6′), 7.40 (d, J = 17.5, H-7′), 6.61 (dd, J = 17.5, 8.5, H-8′), 9.65 (d, J = 8.5, H-9′), 3.88 (s, OMe), 3.94 (s, OMe), 5.70 (s, 4-OH), 9.65 (d, J = 8.5, 4-CHO) [8].

3-[2-(4-Hydroxy-3-methoxyphenyl)-3-hydroxymethyl-7-methoxy-2,3-dihydro-1-benzofuran-5-yl]propan-1-ol (3). $C_{20}H_{25}O_6$, $[M]^+$ 360, white amorphous compound, $[\alpha]_D^{25}$ +5.5° (*c* 0.18, MeOH), mp 209°C (dec.), R_f 0.5 ($CHCl_3$–MeOH, 9.3:0.7). ^1H NMR spectrum (300 MHz, $CDCl_3$, δ, ppm, J/Hz): 6.93 (d, J = 1.83, H-2), 6.86 (d, J = 8.09, H-5), 6.90 (dd, J = 8.09, 1.83, H-6), 5.54 (d, J = 7.48, H-7), 3.59 (m, H-8), 3.94 (dd, J = 10.91, 4.42, H-9a), 3.88 (dd, J = 10.91, 6.02, H-9b), 6.67 (s, H-2′), 6.65 (s, H-6′), 2.66 (t, H-7′), 1.87 (m, H-8′), 3.68 (t, J = 6.41, H-9′), 3.88 (s, OMe), 3.94 (s, OMe), 5.70 (s, 4-OH), 3.87 (s, 9-OH) [9].

5,4′-Dihydroxy-6,7,3′-trimethoxyflavone (4). $C_{18}H_{16}O_7$, mp 201–203°C, $[M]^+$ 344. ^1H NMR spectrum (400 MHz, CD_3OD, δ, ppm, J/Hz): 7.60 (1H, dd, J = 8.2, 1.8, H-6′), 7.9 (1H, d, J = 1.8, H-2′), 6.8 (1H, d, J = 8.2, H-5′), 6.3 (1H, s, H-3), 6.43 (1H, s, H-8), 3.7–3.9 (9H, s, OCH_3), 12.60 (1H, s, 5-OH) [10].

Hyperin (5). $C_{22}H_{22}O_{12}$, mp 232–235°C, $[\alpha]_D^{20}$ –71.5° (*c* 0.5, EtOH). UV spectrum (EtOH, λ_{max}, nm): 361, 298 (sh.), 259; (+NaAc) 370, 270; (+$AlCl_3$) 420, 280; (+$AlCl_3$ + HCl) 380, 280; (+NaAc + H_3BO_3) 380, 290; (+$NaOC_2H_5$) 410, 280. IR spectrum (mineral oil, ν, cm^{-1}): 3400–3200 (OH), 1670, 1620, 1615 (C=O), 1520, 1470 (C=C). ^1H NMR spectrum (400 MHz, CD_3OD, δ, ppm, J/Hz): 5.4 (d, J = 7, H-1″), 6.12 (d, J = 2, H-6), 6.43 (d, J = 2, H-8), 6.85 (d, J = 8.5, H-5′), 7.2 (d, J = 2.5, H-3′), 7.53 (d, J = 2.5, H-2′), 7.85 (dd, J = 8.5, 2, H-6′) [11].

Compounds **2–4** were isolated for the first time from plants of the genus *Climacoptera*.

1) Al-Farabi Kazakh National University, Faculty of Chemistry and Chemical Engineering, 050040, Almaty, Kazakhstan, fax: (+7727) 377 33 44, e-mail: balakyz@mail.ru; 2) Chemistry Research Institute, International Center for Chemical and Biological Sciences, University, 75720, Karachi, Pakistan. Translated from *Khimiya Prirodnykh Soedinenii*, No. 3, May–June, 2014, pp. 463–464. Original article submitted November 28, 2013.

0009-3130/14/5003-0537

REFERENCES

1. N. V. Pavlova, *Flora of Kazakhstan* [in Russian], Vol. 3, Akad. Nauk KazSSR, Alma-Ata, 1960, pp. 274–281.
2. V. L. Komarov (ed.), *Flora of the USSR* [in Russian], Vol. 6, Izd. Akad. Nauk SSSR, Moscow, Leningrad, 1955, pp. 2–6.
3. L. D. Sokolov, *Plant Resources of the USSR. Flowering Plants, Their Chemical Composition and Use* [in Russian], Nauka, Leningrad, 1936, 237 pp.
4. B. Yeskaliyeva, M. A. Mesaik, A. Abbaskhan, A. Kulsoom, G. Sh. Burasheva, Zh. A. Abilov, M. I. Choudhary, and Atta-ur-Rahman, *Phytochemistry*, **67** (21), 2392 (2006).
5. B. K. Eskalieva, A. Akhmed, G. Sh. Burasheva, Zh. A. Abilov, and V. U. Akhmad, *Chem. Nat. Compd.*, **40**, 87 (2004).
6. A. K. Kipchakbaeva, B. K. Eskalieva, G. Sh. Burasheva, and H. A. Aisa, *Chem. Nat. Compd.*, **48**, 1076 (2012).
7. A. Mansurov, N. K. Zhylybaeva, P. S. Ualieva, and R. M. Mansurova, *Khim. Interesakh Ustoich. Razvit.*, No. 10, 339 (2002).
8. H. Kasahara, Y. Jiao, D. L. Bedgar, S. J. Kim, A. M. Patten, Z. Q. Xia, L. B. Davin, and N. G. Lewis, *Phytochemistry*, **67**, 1765 (2006).
9. L. Pieters, S. V. Dyck, M. Gao, R. Bai, E. Hamel, A. Vlientick, and G. Lemiere, *J. Med. Chem.*, **42**, 5475 (1999).
10. E. A. Kul′magambetova, L. N. Pribytkova, and S. M. Adekenov, *Chem. Nat. Compd.*, **36**, 95 (2000).
11. K. R. Markham, *Flavones, Flavonols and Their Glycosides*, in: *Methods in Plant Biochemistry*, J. B. Harborne and P. M. Dey (eds.), Vol. 1, Academic Press, London, 1989, pp. 203–210.