Chemistry of Natural Compounds, Vol. 46, No. 6, 2011

CHEMICAL CONSTITUENTS OF THE AERIAL PART OF *Atractylodes macrocephala*

Wei Peng, Ting Han, Yang Wang, Wen-Bo Xin, Cheng-Jian Zheng, and Lu-Ping Qin*

UDC 547.972

Atractylodes macrocephala Koidz., belonging to the Compositae family, is widely distributed in China. It has been used as an important crude drug against stomach diseases, digestive disorders, and anorexia. Previous phytochemical investigations of *A. macrocephala* showed the presence of polyacetylenes, sesquiterpenoids, and sesquiterpene glycosides [1–3]. Most previous study on *A. macrocephala* focused on the rhizomes but not other parts because of traditional customs in the use of this plant. To the best of our knowledge, there have been no reports on the aerial part of *A. macrocephala* extracts and their chemical components. In this studies of the chemical constituents of the aerial part of *A. macrocephala*, nine compounds were isolated and identified. All compounds were obtained from this plant for the first time.

The dried and powdered aerial part of *A. macrocephala* were extracted five times (each extraction period lasted 4 days) with 75% aqueous ethanol solution repeatedly by maceration. The solution was concentrated and partitioned with solvents, starting with petroleum ether, ethyl acetate, and *n*-butanol. Part of the ethyl acetate fraction was subjected to a series of chromatographic techniques, such as silica gel column (200–300 mash), Sephadex LH-20, and PTLC, yielding compounds **1–8**.

The compounds were identified using mass and NMR spectra, and all these data were in good agreement with the literature data.

7-Hydroxycoumarin (1). $C_9H_6O_3$, colorless crystal. ^{1}H NMR (600 MHz, CD_3OD, δ, ppm, J/Hz): 6.15 (1H, d, J = 9.4, H-3), 7.81 (1H, d, J = 9.4, H-4), 6.41 (1H, d, J = 8.5, H-5), 6.77 (1H, dd, J = 2.3, J = 8.5, H-6), 6.68 (1H, d, J = 2.3, H-8). ^{13}C NMR (CD_3OD, 150 MHz, δ): 162.28 (C-2), 113.11 (C-3), 144.61 (C-4), 129.22 (C-5), 110.89 (C-6), 161.76 (C-7), 102.00 (C-8), 111.71 (C-4a), 155.82 (C-8a) [4].

2,6-Dimethoxyquinone (2). $C_8H_8O_4$, yellow powder. ESI-MS *m/z*: 169.32 $[M + H]^+$. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 3.82 (6H, s, 2 × OCH_3), 5.86 (2H, s, H-3,5). ^{13}C NMR (150 MHz, $CDCl_3$, δ): 56.46 (2 × OCH_3), 107.40 (C-3, C-5), 157.34 (C-2, 6), 176.65 (C-1), 186.89 (C-4) [5].

Apigenin (3). $C_{15}H_{10}O_5$, yellow powder. ESI-MS *m/z*: 271.15 $[M + H]^+$. ^{1}H NMR (600 MHz, DMSO-d_6, δ, ppm, J/Hz): 6.16 (1H, d, J = 2.1, H-6), 6.45 (1H, d, J = 2.1, H-8), 6.74 (1H, s, H-3), 6.89 (2H, dd, J = 2.0, J = 8.8, H-3′ and H-5′), 7.89 (2H, dd, J = 2.0, J = 8.8, H-2′ and H-6′). ^{13}C NMR (150 MHz, DMSO-d_6, δ): 164.18 (C-2), 104.14 (C-3), 182.18 (C-4), 161.90 (C-5), 99.28 (C-6), 164.59 (C-7), 94.40 (C-8), 161.61 (C-9), 103.28 (C-10), 121.62 (C-1′), 157.75 (C-4′), 128.90 (C-2′ and C-6′), 116.40 (C-3′ and C-5′) [6].

Luteolin (4). $C_{15}H_{10}O_6$, yellow powder. ESI-MS *m/z*: 287.05 $[M + H]^+$. ^{1}H NMR (600 MHz, DMSO-d_6, δ, ppm, J/Hz): 6.16 (1H, d, J = 2.0, H-6), 6.42 (1H, d, J = 2.0, H-8), 6.64 (1H, s, H-3), 6.86 (1H, dd, J = 8.3, H-5′), 7.36 (1H, dd, J = 2.1, H-2′), 7.39 (1H, dd, J = 2.2, J = 8.3, H-6′). ^{13}C NMR (150 MHz, DMSO-d_6, δ): 164.34 (C-2), 103.30 (C-3), 182.09 (C-4), 161.90 (C-5), 99.27 (C-6), 164.57 (C-7), 94.29 (C-8), 157.73 (C-9), 104.13 (C-10), 121.94 (C-1′), 150.14 (C-4′), 113.78 (C-2′), 146.172 (C-3′), 116.46 (C-5′), 119.42 (C-6′) [7].

School of Pharmacy, Second Military Medical University, 325 Guohe Rd., Shanghai 200433, China, fax : +86 21 81871300, e-mail: qinsmmu@126.com. Published in Khimiya Prirodnykh Soedinenii, No. 6, pp. 816–817, November–December, 2010. Original article submitted June 22, 2009.

0009-3130/11/4606-0959

5,7,4′-Trihydroxy-3′,5′-dimethoxyflavone (5). $C_{17}H_{l4}O_7$, yellow needles. ^{1}H NMR (600 MHz, DMSO-d_6, δ, ppm, J/Hz): 3.86 (6H, s , 2 × OCH_3), 6.17 (1H, d, J = 2.1, H-6), 6.53 (1H, d, J = 2.1, H-8), 6.95 (1H, s, H-3), 7.30 (2H, s, H-2′ and H-6′). ^{13}C NMR (150 MHz, DMSO-d_6, δ): 164.10 (C-2), 104.17 (C-3), 182.24 (C-4), 161.85 (C-5), 99.27 (C-6), 164.59 (C-7), 94.64 (C-8), 157.77 (C-9), 104.04 (C-10), 120.85 (C-1′), 140.32 (C-4′), 104.85 (C-2′ and C-6′), 148.64 (C-3′ and C-5′) [8].

Ethyl 3-(4-Hydroxyphenyl)acrylate (6). $C_{11}H_{12}O_3$, colorless crystal. ^{1}H NMR (600 MHz, CD_3OD, δ, ppm, J/Hz): 1.29 (3H, t, CH_3), 4.19 (2H, q, OCH_2), 6.28 (1H, d, J = 15.6, H-8), 6.79 (2H, dd, J = 8.6, J = 1.8, H-5 and H-3), 7.42 (2H, dd, J = 8.6, J = 1.8, H-6 and H-2), 7.43 (1H, d, J = 15.6, H-7). ^{13}C NMR (150 MHz, CD_3OD, δ): 14.62 (CH_3), 61.39 (OCH_2), 115.35 (C-8), 116.80 (C-3 and C-5), 127.17 (C-1), 131.09 (C-2 and C-6), 146.31 (C-7), 161.19 (C-4), 169.29 (C-9) [9].

Ethyl 3,4-Dihydroxycinnamate (7). $C_{11}H_{12}O_4$, colorless crystal. ESI-MS *m/z*: 207.14 $[M - H]^-$. ^{1}H NMR (600 MHz, CD_3OD, δ, ppm, J/Hz): 1.29 (3H, t, CH_3), 4.19 (2H, q, OCH_2), 6.22 (1H, d, J = 16.2, H-8), 6.76 (1H, d, J = 7.8, H-5), 6.91 (1H, dd, J = 7.8, J = 1.8, H-6), 7.02 (1H, d, J = 1.8, H-2), 7.52 (1H, d, J = 16.2, H-7). ^{13}C NMR (150 MHz, CD_3OD, δ): 14.61 (CH_3), 61.40 (OCH_2), 115.11 (C-2), 115.28 (C-5), 116.49 (C-8), 122.87 (C-6), 127.74 (C-1), 146.70 (C-7), 146.77 (C-4), 149.49 (C-3), 169.32 (C-9).

Lupeol (8). $C_{30}H_{50}O$, white needles. ESI-MS *m/z*: 427.39 $[M + H]^+$. Identification of compound **8** was performed by ^{1}H NMR and ^{13}C NMR data compared with those reported in [10].

All these compounds were isolated from *A. macrocephala* for the first time.

Phytochemical studies of the aerial part of *A. macrocephala* are continuing.

REFERENCES

1. Z. L. Chen, W. Y. Cao, G. Zhou, and M. Wichtl, *Phytochemistry*, **45**, 765 (1997).
2. J. Kitajima, A. Kamoshita, and T. Ishikawa, *Chem. Pharm. Bull*., **51**, 1106 (2003).
3. B. S. Huang, *Acta Bot. Sin.,* **31**, 614 (1992).
4. H. T. Chang and X. Li, *J. Shenyang Pharm. Univ.,* **16**, 103 (1999).
5. X. J. Hu, H. Z. Jin, W. Zhang, S. K. Yan, W. Z. Xu, R. H. Liu, Y. H. Shen, and W. D. Zhang, *Chem. Nat. Comp.,* **45**, 126 (2009)
6. H. Ltokawa, K. Sueo, and K. Takeya, *Chem. Pharm. Bull.*, **29**, 254 (1981).
7. Y. Diaa and W. F. August, *Planta Med.,* **61**, 570 (l995).
8. D. Q. Yu and J. S. Yang, *Handbook of Analytical Chemistry* (seventh faseieule), 2nd Ed., Beijing: Chemical Industry Press, 1999, pp. 818–821.
9. P. Zhang and Xi. W. Yang, *China J. Chin. Mater. Med.,* **33**, 2918 (2008).
10. Y. M. Luo, A. H. Liu, B. W. Yu, L. J. Kang, and L. Q. Huang, *Chin. Pharm. J.*, **40**, 1296 (2005).