sis of spectral analysis (UV, IR, ^{1}HNMR, ^{13}CNMR, DEPT, ^{1}H-^{1}H COSY, ^{1}H-^{13}C COSY), they were identified as 4-propenyl-phenyl-2-methyl butanoate(Ⅰ) and 4-methoxy-1-propenyl-phenyl-2-methyl butanoate(Ⅱ). Ⅰ is a new compound named thellungianin F. Ⅱ was obtained from this plant for the first time.

Key Words *Pimpinella thellungiana* thellungianin F

A New Indole Derivative Isolated from the Root of Tuber Fleeceflower(*Polygonum multiflorum*)

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Abstract From the root of *Polygonum multiflorum* Thunb., a new indole derivative, named Indole-3(L-α-amino-α-hydroxy propionic acid)methyl ester(Ⅺ), was isolated together with the ten known compounds, chrysophanol(Ⅰ), physcione(Ⅱ), emodin(Ⅲ), citreorosein(Ⅳ), chrysophanol 8-O-β-D-glucopyranoside(Ⅴ), physcione 8-O-β-D-glucopyranoside(Ⅵ), emodin 8-O-β-D-glucopyranoside(Ⅶ), torachrysone 8-O-β-D-glucopyranoside(Ⅷ), 2, 3, 5, 4′-tetrahydroxystilbene2-O-β-D-glucopyranoside(Ⅸ), and methylgallate(Ⅹ). Their structures were determined by spectroscopic means. These anthraquinone compounds and aloe-emodin(Ⅻ), rhein(ⅩⅢ), aloe-emodin 8-O-β-D-glucopyranoside(ⅩⅣ), chrysophanol 8-O-β-D-(6′-O-malonyl)glucopyranoside(ⅩⅤ), sennoside A (ⅩⅥ) and sennoside B(ⅩⅦ) had no inhibitory effect against recombinant HIV-1 protease at concentration of 100 μmol/L in vitro.

Key Words Root of *Polygonum multiflorum* Indole derivative Indole-3(L-α-amino-α-hydroxy propionic acid)methyl ester Recombinant HIV-1 protease

1 Introduction

The root of *Polygonum multiflorum* Thunb. (*Polygonaceae*), He shou wu, is a tonic drug, invigorate the liver and kidney, tonifying the kidney, and for treatment of yin-deficiency of liver and kidney, vertigo, insomnia, lassitude of the loins and legs in Chinese medicine[1]. Our pharmacological results showed that five-day successive *po* administration of an EtOH extract of root of *P. multiflorum* can inhibit significantly the activity of monoamine oxidase B(MAO-B), though the extract showed no activity for MAO-A in male senescence-accelerated mice[2].

As a part of our chemical investigations on the active constituents from natural sources, we report chemical investigation of the root of *P. multiflorum* which led to the isolation of eleven compounds from the EtOAc and n-BuOH soluble fractions of the ethanolic extract, and inhibitory effects of some anthraquinone compounds against recombinant(REC) HIV-1 protease in *vitro*.

2 Results and Discussion

An ethanolic extract of the root of *P. multiflorum* was fractionated into EtOAc- and n-BuOH-soluble fractions. Repeated column chromatography of these fractions led to the isolation of eleven compounds. One new indole derivitive was isolated and identified as Indole-3(L-α-amino-α-hydroxy propionic acid) methyl ester (Ⅺ). Ten known

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compounds, chrysophanol (Ⅰ), physcione (Ⅱ), emodin (Ⅲ), citreorosein (Ⅳ), chrysophanol 8-O-β-D-glucopyranoside (Ⅴ), physcione 8-O-β-D-glucopyranoside (Ⅵ), emodin 8-O-β-D-glucopyranoside (Ⅶ), torachrysone 8-O-β-D-glucopyranoside (Ⅷ), 2,3,5,4′-tetrahydroxystilbene 2-O-β-D-glucopyranoside (Ⅸ), and methylgallate (Ⅹ) were also isolated and fully characterized. Their ^{13}C NMR data were shown in Table 1, respectively.

Compound Ⅺ showed IR bands at 3458, 1626, 1104, and 750 cm^{-1} for N-H, ester, C-O, and Ar-H, respectively. Its EI-MS spectrum showed the quasi molecular ion peak at m/z 234, corresponding to $C_{12}H_{14}N_2O_3$. The elucidated formula was further confirmed by HR-MS and elementary analysis. Its ^1H and ^{13}C NMR spectra (Table 2) were quite similar to those of L-tryptophan, except for some signals in the C_3 (to shift downfield by 1.7 ppm) and side-chain. Therefore, compound Ⅺ should be a 3-substituted indolyl skeleton. The ^{13}C NMR data, as well as the DEPT spectra, indicated the presence of 4 carbons of which were O-methyl, carbonyl ester (δ 171.4), quaternary, and methylene in side-chain, respectively. ^1H and ^{13}C NMR and HMQC spectra were also consistent with the proposed structure (Fig. 1). Compound Ⅺ was found to be optically active, $[\alpha]_D$ − 48° (c, 1, MeOH). The above-mentioned spectral findings support the structure for compound Ⅺ as Indile-3-(L-α-amino-α-hydroxy propionic acid) methyl ester. This compound was isolated for the first time from the natural source.

In addition, we screened thirteen anthraquinones and one naphthalene for anti-HIV-1 protease activity. All compounds showed no inhibitory effect at concentrations of 100 μmol/L *in vitro* (Table 3).

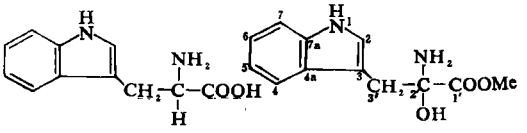

Fig 1 Structure of Compound Ⅺ

3 Experimental

3.1 Apparatus: Melting points were determined on a Yanagi motomicro-melting point apparatus and are uncorrected. IR spectra were taken on a Hitachi 260-10 and Perkin-Elmer 983 infrared spectrometer. UV were measured with a Shimadzu UV-2200 spectrophotometer (Shimadzu, Kyoto). ^1H NMR and ^{13}C NMR were measured with JEOL GX-270 (^1H, 270 MHz, ^{13}C, 67.5 MHz), Varian Gemini 300 (^1H, 300 MHz, ^{13}C, 75.5 MHz) and 400 (^1H, 400 MHz, ^{13}C, 100 MHz). Tetramethylsilane was used as an internal standard. Optical rotations were measured with JASCO DIP-4 automatic polarimeter. EI-MS, FAB-MS and HR-MS were measured with JEOL JMS-DX 300 L mass spectrometer.

3.2 Chromatography: Wako gel 100 (Wako Pure Chemical Industries Co., Osaka, Japan) was used for column chromatography. Thin-layer chromatography (TLC) plates (Merck Kieselgel 60 F_{254}; layer thickness, 0.25 mm) were purchased from E. Merck (Darmstadt, FRG).

3.3 Materials and Reagents: Roots of *P. multiflorum* were collected at Ma-er-kang county, Sichuan province of China, in August 1991. The plant was identified by professor Zhe Ming gu. Aloe-emodin (Ⅻ), rhein (ⅩⅢ), aloe-emodin 8-O-β-D-glucopy-

ranoside (XIV), chrysophanol 8-O-β-D-(6′-O-malonyl) glucopyranoside (XV), sennoside A (XVI), and sennoside B (XVII) were isolated from the rhizomes of *Rheum qinlingense* and *R. palmatum*.

The recombinant (Rec) HIV-1 protease was obtained as reported[3]. L-Tryptophan was purchased from Wako Pure Chemical Industries Ltd. (Osaka, Japan).

3.4 Extraction and fractionation: Dried and pulverised roots (2.5 kg) were refluxed with ethanol (4 L) five times for one hour each time. The ethanol layer was filtered and concentrated in vacuo to yield ethanol extract (285 g, PME, yields. 11.4%). The PME was suspended in H_2O (1 L), and extracted successively with ethyl acetate (2 L ×5) and n-butanol (2 L×5) to yield EtOAc extract (41 g, 1.64%), n-BuOH extract (185 g, 7.4%), and water extract (49 g, 19.6%), respectively.

The EtOAc extract (40 g) was subjected to column chromatography on silica gel and eluted with cyclohexane-$CHCl_3$ (1 : 1), $CHCl_3$, and $CHCl_3$-MeOH gradually increasing polarity. The elutes were collected in 100 mL portions, monitored by TLC. Fraction 5 eluted with $CHCl_3$ were subjected to repeated CC to yield compounds I (33 mg), II (32 mg), III (25 mg), and IV (18 mg). Similarly, fractions eluted with $CHCl_3$-MeOH gave compounds V (250 mg), VI (320 mg), VII (28 mg), and VIII (15 g).

The n-BuOH extract (100 g) was subjected to column chromatography on silica gel and eluted with $CHCl_3$ and $CHCl_3$-MeOH gradually increasing polarity. The elutes were collected in 100 mL portions, monitored by TLC. Fractions eluted with $CHCl_3$ gave compound IX (30 mg). Fractions eluted with $CHCl_3$-MeOH (4 : 1) gave compound V (48 mg), VI (50 mg), VII (10 mg), VIII (1 g), IX (51 mg), X (62 mg). Fractions eluted with $CHCl_3$-MeOH (3 : 2) gave compound XI (102 mg).

Compound I (chrysophanol): monoclinic (benzene), mp 196 ℃～197 ℃; IR_{max} (KBr) cm^{-1}: 3429 (OH), 1677 (free C=O), 1626 (chelated C=O), 1560, 1457; UV λ_{max} nm (log ε) EtOH: 225 (4.2), 256 (2.4), 277 (1.2), 287 (1.6); EI-MS m/z: 254 [M^+]; ^{1}HNMR (400 MHz, DMSO-d_6) δppm: 11.80 (2 H, s, $C_{1,8}$-OH), 7.66 (1 H, d, J=8.0 Hz, C_5-H) 7.56 (1 H, dd, J=1.2, 8.0 Hz, C_6-H), 7.39 (1 H, s, C_4-H), 7.26 (1 H, d, , J=8.0 HZ, C_7-H), 7.07 (1 H, s, C_2-H), 2.33 (3 H, s, Me) 3.94 (3 H, s, Me). ^{13}C NMR: see Table 1.

Compound II (physcione): monoclinic needles (benzene), mp 207 ℃～208 ℃; IR_{max} (KBr) cm^{-1}: 3429 (OH), 1684 (freeC=O), 1626 (chelated C=O), 1557, 1541, 1506; UV λ_{max} nm (log ε) EtOH: 223 (4.2), 253 (1.8), 285 (2.2); EI-MSm/z: 284 [M^+]; ^{1}HNMR (400 MHz, DMSO-d_6) δppm: 12.03 (1 H, s, C_8-OH or C_1-OH), 11.95 (1 H, s, C_1-OH or C_8-OH), 7.54 (1 H, d, J=1.2 Hz, C_4-H), 7.20 (1 H, d, J=1.2 Hz, C_5-H), 7.19 (1 H, d, J=2.4 Hz, C_2-H), 6.88 (1 H, d, J=2.4 Hz, C_7-H), 3.94 (3 H, s, OMe), 2.43 (3 H, s, Me). ^{13}C NMR: see Table 1.

Compound III (emodin): orange needles (EtOH), mp 255 ℃～257 ℃; IR_{max} (KBr) cm^{-1}: 3429 (OH), 1684 (free C=O), 1632 (chelated C=O), 1558, 1541, 1485; UV λ_{max} nm (log ε) EtOH: 221 (4.4), 253 (1.9), 266 (2.1), 290 (2.5); EI-MS m/z: 270 [M^+]; ^{1}HNMR (400 MHz, DMSO-d_6) δppm: 12.03 (1 H, s, C_8-OH or C_1-OH), 11.95 (1

H, s, C_1-OH or C_8-OH), 11.33(1 H, s, C_6-OH), 7.41(1 H, d, J=1.2 Hz, C_4-H), 7.08(1 H, d, J=1.2 Hz, C_2-H), 7.06(1 H, d, J=2.4 Hz, C_5-H), 6.54(1 H, d, J=2.4 Hz, C_7-H), 2.38(3 H, s, Me). ^{13}C NMR: see Table 1.

Compound Ⅳ (citreorosein): orange needles(MeOH), mp 277 ℃～279 ℃; IR_{max} (KBr)cm^{-1}:3442(OH), 1673(free C=O), 1627(chelated C=O), 1561, 1432; EIMS m/z:286[M^+]; ^{1}HNMR(270 MHz, DMSO-d_6)δppm: 12.09(1 H, s, C_8-OH or C_1-OH), 12.06(1 H, s, C_1-OH or C_8-OH), 7.63(1 H, s, C_5-H), 7.24(1 H, s, C_7-H), 7.12(1 H, d, J=2.1 Hz, C_4-H), 6.59(1 H, d, J=2.1 Hz, C_2-H), 4.60(2 H,brs,C_3-CH_2OH). ^{13}C NMR:see Table 1.

Compound Ⅴ (chrysophanol 8-O-β-D-glucopyranoside): yellow powder; IR_{max} (KBr)cm^{-1}:3387(OH), 1672(free C=O), 1632(chelated C=O), 1594, 1445, 1076 (sugar C-O); UVλ$_{max}$nm(log ε)EtOH: 221 (4.4), 258(3.0), 283(2.2); EI-MS:m/z 416[M^+], 254[M^+ − glc]; FAB-MS:m/z 416[M^+]; ^{1}HNMR(300 MHz, DMSO-d_6) δppm:12.85(1 H, s, C_1-OH), 7.88(1 H, dd, J=1.5, 6.3 Hz, C_5-H), 7.85(1 H, dd, J=7.7, 8.0 Hz, C_6-H), 7.71(1 H, dd, J=1.5, 7.9 Hz, C_7-H), 7.51(1 H, d, J=1.2 Hz, C_4-H), 7.20(1 H, d, J=1.2 Hz, C_2-H), 5.17(1 H, d, J=7.8 Hz, aromeric H), 3.71(1 H, dd, J=5.6, 10.3 Hz, G-C_6-Ha), 3.50(1 H, dd, J=5.9, 11.9 Hz, G-C_6-Hb), 3.46(1 H, dd, J=3.2, 8.5 Hz, G-C_2-H), 3.43(1 H, dd, J=3.4, 8.1 Hz, G-C_5-H), 3.31(1 H, dd, J=5.3, 9.0 Hz, G-C_3-H), 3.24(1 H, dd, J=5.1, 9.2 Hz, G-C_4-H). ^{13}C NMR:see Table 1.

Compound Ⅵ (physcione 8-O-β-D-glucopyranoside):yellow needles(MeOH), mp 244 ℃～245 ℃; IR_{max} (KBr) cm^{-1}:3400(OH), 1670(free C=O), 1630 (chelated C=O), 1595, 1076(sugar C-O); UVλ$_{max}$nm(log ε)MeOH:221(4.3), 245(3.4), 269(2.2), 277(1.3); FAB-MS:m/z 446[M^+]; ^{1}HNMR(300 MHz, DMSO-d_6) δppm :12.81(1 H, s, C_1-OH), 7.59(1 H, d, J=1.2 Hz, C_4-H), 7.43(1 H, d, J=1.2 Hz, C_5-H), 7.25(1 H, d, J=1.2 Hz, C_2-H), 7.04(1 H, d, J=1.2 Hz, C_7-H), 5.51 (1 H, d, J=7.8 Hz, aromeric H), 3.96(3 H, s, OMe), 3.71(1 H, dd, J=5.6, 10.3 Hz, G-C_6-Ha), 3.50(1 H, dd, J=5.9, 11.9 Hz, G-C_6-Hb), 3.46(1 H, dd, J=3.2, 8.5 Hz, G-C_2-H), 3.43(1 H, dd, J=3.4, 8.1 Hz, G-C_5-H), 3.31(1 H, dd, J=5.3, 9.0 Hz, G-C_3-H), 3.24(1 H, dd, J=5.13, 9.2 Hz, G-C_4-H), 2.43(3 H, s, Me). ^{13}C NMR:see Table 1.

Compound Ⅶ (emodin8-O-β-D-glucopyranoside):yellow powder; IR_{max}(KBr)cm^{-1}: 3409(OH), 1732(free C=O), 1628(chelated C=O), 1596, 1508, 1477, 1073(sugar C-O); UVλ$_{max}$nm(log ε)EtOH:221(4.1), 258 (2.2), FAB-MS: m/z 432 [M^+]; ^{1}HNMR(300 MHz, DMSO-d_6)δppm :13.17 (1 H, s, C_1-OH), 11.26(1 H, s, C_6-H), 7.46(1 H, d, J=1.2 Hz, C_4-H), 7.29(1 H, d, J=1.2, Hz, C_5-H), 7.15(1 H, d, J=1.2 Hz, C_2-H), 6.70(1 H, d, J=1.2 Hz, C_7-H), 5.07(1 H, d, J=7.2 Hz, anomeric H), 3.71(1 H, dd, J=5.6, 10.3 Hz, G-C_6-Ha), 3.50(1 H, dd, J=5.9, 11.9 Hz, G-C_6-Hb), 3.46(1 H, dd, J=3.2, 8.5 Hz, G-C_2-H), 3.43(1 H, dd, J=3.4, 8.1 Hz, G-C_5-H), 3.31(1 H, dd, J=5.3, 9.0 Hz, G-C_3-H), 3.24(1 H, dd, J=5.13, 9.2 Hz, G-C_4-H), 2.40(3 H, s, Me). ^{13}C NMR:see Table 1.

Compound Ⅷ (torachrysone 8-O-β-D-

glucopyranoside): yellow needles, (MeOH), mp 151 ℃～153 ℃; IR_{max}(KBr)cm^{-1}: 3387 (OH), 1620(free C=O), 1585; $UV\lambda_{max}$ nm (log ε) MeOH: 235(4.55), 240(4.66), 260 (4.55), 271(4.22), 312(3.32), 325(3.81), 340 (4.1); FAB-MS (positive) m/z: 409 [M^+]; ^{1}HNMR (270 MHz, CD_3OD) δppm: 7.03(1 H, s, C_4-H), 7.01(1 H, d, J=2.2 Hz, C_5-H), 6.82(1 H, d, J=2.2 Hz, C_7-H), 5.09(1 H, d, J=7.3 Hz, G-C_1-H), 3.95 (1 H, d, J=2.0, 12.2 Hz, G-C_6-Ha), 3.86 (3 H, s, OMe), 3.75(1 H, dd, J=5.1, 12.1 Hz, G-C_6-Hb), 3.40～3.70(4 H, m, G-$C_{2,5}$-H), 2.58(3 H, s, COMe), 2.29(3 H, s, C_3-Me); ^{13}CNMR (67.5 MHz, CD_3OD) δppm: 208.9 s(CO), 161.2 s(C_8), 157.9 s (C_6), 154.5 s(C_1), 139.9 s(C_3), 136.2 s (C_2), 124.8 s(C_{8a}), 121.1 d(C_4), 111.1 s (C_{4a}), 105.2 d(C_5 or C_7), 105.1 d(C_7 or C_5), 103.3 d(G-C_1), 79.6 d(G-C_3), 78.9 d (G-C_5), 75.7 d(G-C_2), 72.1 d(G-C_4), 63.2 t(G-C_6), 56.8 q(OMe), 33.4 q(COMe), 21.0 q(C_3-Me).

Compound Ⅸ (2,3,5,4′-tetrahydroxystilbene2-O-β-D-glucopyranoside): yellowish needles (EtOH), mp 184 ℃～186 ℃; IR_{max}(KBr)cm^{-1}: 3275(OH), 1604(free C=O), 1601(sugar C-O); $UV\lambda_{max}$ nm (log ε) MeOH: 216(4.22), 308(4.40), 319(4.32); FAB-MS (positive) m/z: 407 [M^+ + 1]; ^{1}HNMR (4000 MHz, CD_3OD) δppm: 7.70(1 H, d, J=16.5 Hz, β-H), 7.44(2 H, d, J=8.6 Hz, $C_{2',6'}$-H), 6.92(1 H, d, J=16.5 Hz, α-H), 6.76(2 H, d, J=8.6 Hz, $C_{3',5'}$-H), 6.62(1 H, d, J=2.7 Hz, C_6-H), 6.25 (1 H, d, J=2.7 Hz, C_4-H), 4.51(1 H, d, J=7.6 Hz, G-C_1-H), 3.82(1 H, dd, J=2.4, 12.0 Hz, G-C_6-Ha), 3.76(1 H, dd, J=4.0, 12.0 Hz, G-C_6-Hb), 3.52～3.60(2 H, m, G-$C_{3,4}$-H), 3.41～3.46(1 H, m, G-C_2-H), 3.25～3.29(1 H, m, G-C_5-H); ^{13}CNMR (100 MHz, CD_3OD) δppm: 159.1 s(C_4'), 156.7 s(C_5), 152.8 s(C_3), 138.7 s(C_2), 134.5 s(C_1), 131.7 s(C_1'), 130.9 d($C_β$), 130.0 d($C_{3',5'}$), 122.6 d($C_α$), 117.2 d ($C_{2',6'}$), 109.0 d(C_6), 104.4 d(C_4), 103.6 d (G-C_1), 79.0 d(G-C_3), 78.7 d(G-C_5), 76.3 d(G-C_2), 71.6 d(G-C_4), 63.0 t(G-C_6).

Compound Ⅹ (methylgallate): colorless needles (EtOH-MeOH), mp 197 ℃～199 ℃; IR_{max}(KBr)cm^{-1}: 3382(OH), 1696, 1622, 1532; $UV\lambda_{max}$ nm (log ε) MeOH: 221 (4.5), 278(4.0); EI-MS m/z: 184[M^+]; ^{1}HNMR (300 MHz, DMSO-d_6) δppm: 3.78 (3 H, s, OMe), 7.00(2 H, s, $C_{2,6}$-H); ^{13}CNMR (75 MHz, DMSO-d_6) δppm: 166.3 s (-COOH), 145.5 s($C_{3,5}$), 138.3 s(C_4), 119.4 s (C_1), 108.5 d($C_{2,6}$), 51.5 q(OMe).

Compound Ⅺ [Indole-3 (L-α-amino-α-hydroxy-propionic acid) methyl ester]: yellowish powder; $[\alpha]_D$ −48°(c, 1, MeOH); IR (KBr)cm^{-1}: 3458 (N-H), 1626 (ester C=O), 1104(C-O), 750(Ar-H); EI-MS m/z: 234[M^+], 219[M^+−O+H], 201[M^+−O+H−H_2O], 187 [201 − N], 142 [M^+ − $C_2H_6O_3N$], 130[M^+ − $C_3H_6O_3N$], 116[M^+ − $C_4H_8O_3N$]. Anal. (%) Calcd. for $C_{12}H_{14}O_3N_2$: C 61.52, H 6.02, O 20.49, N 11.96; Found: C 61.94, H 6.04, O 20.31, N 11.81; HR-MS: Calcd for $C_{12}H_{14}O_3N_2$ [M^+]: 234.1880, Found: 234.1886; ^{1}HNMR (270 MHz, CD_3OD) δppm: 7.63(1 H, d, J=7.8 Hz, C_{11}-H), 7.35(1 H, d, J=8.1 Hz, C_8-H), 7.19(1 H, s, C_2-H), 7.11(1 H, dd, J=7.0 Hz, C_9-H), 7.03(1 H, dd, J=7.0, 7.0 Hz, C_{10}-H), 3.37(2 H, s, -CH_2-), 3.26(3 H, s, COMe); ^{13}CNMR: see Table 2.

3.5 Rec HIV-protease Assay: Thirty four μL of 50 μmol/L acetate buffer (pH 5.0) containing 2μg of a substrate (His-Lys-Ala-

Arg-Val-Leu-(pNO_2-Phe)-Glu-Ala-Nle-Ser-NH_2) was mixed with 4.0 μL of a compound solution (using H_2O-DMSO = 1 : 1 as solvent), THEN 2 μL of recHIV-1 protease (1/100) was added into this mixture. The reaction mixture was incubated at 37 ℃ for one and a half hour and then terminated by heating at 90 ℃ for one min. The hydrolysate (pNO_2-Phe-Glu-Ala-Nle-Ser-NH_2) and remained substrate were quantitatively analyzed by reversed-phase HPLC (LiChrospher 100 RP-18 column (250 mm × 4 mm, Merck, Darmstadt, Germany) with a gradient of acetonitrile (18%～34%) in 0.1% trifluoroacetic acid (TFA) at a flow rate of 1.0 mL/min. The elution profile was monitored at 280 nm. The substrate and the hydrolysate were eluted at 9.6 and 3.9 min, respectively. The peak areas were calculated with an integrator C-R1A Chromatopac (Shimadzu). The inhibitory activity of a compound on HIV-protease was calculated as follows: Inhibition (%) = $(A_{control} - A_{sample} \times 100/A_{control}$ (where A is a relative peak area of the hydrolysate). Acetylpepstatin was used as a positive control.

Table 1 ^{13}CNMR spectral data of compounds I ～ Ⅶ

No.	I	Ⅱ	Ⅲ	Ⅳ	Ⅴ	Ⅵ	Ⅶ
1a	116.3 s	113.1 s	113.1 s	114.0 s	114.9 s	114.4 s	114.4 s
1	161.5 s	161.3 s	161.3 s	161.4 s	161.8 s	160.7 s	161.0 s
2	119.5 d	119.5 d	120.3 d	117.0 d	119.5 d	124.2 d	124.1 d
3	149.3 s	149.2 s	148.0 s	152.7 s	147.8 s	147.1 s	146.8 s
4	124.6 d	120.8 d	123.8 d	120.7 d	122.7 d	119.4 d	119.2 d
4a	133.5 s	132.5 s	132.5 s	132.8 s	132.1 s	132.0 s	132.0 s
5a	133.2 s	134.8 s	134.8 s	135.1 s	134.9 s	136.3 s	136.4 s
5	120.7 d	108.7 d	108.7 d	108.8 d	124.2 d	107.4 d	108.4 d
6	137.5 d	165.5 s	165.5 s	165.5 s	136.1 d	164.7 s	164.1 s
7	124.0 d	107.8 d	107.8 d	107.8 d	121.5 d	106.5 d	108.4 d
8	161.7 s	162.1 s	164.3 s	164.4 s	158.4 s	161.7 s	161.7 s
8a	113.5 s	113.7 s	108.7 s	108.8 s	118.5 s	113.4 s	113.4 s
9	191.8 s	189.4 s	189.4 s	189.6 s	187.9 s	186.4 s	186.4 s
10	181.7 s	180.9 s	180.9 s	181.2 s	182.3 s	182.0 s	182.0 s
3-Me	21.8 q		21.4 q		21.7 q	21.4 q	21.4 q
CH_2OH				62.0 t			
OMe		55.6 q				56.1 q	
G-1					100.7 d	100.7 d	100.9 d
G-2					73.5 d	73.3 d	73.3 d
G-3					76.7 d	76.6 d	76.3 d
G-4					69.7 d	69.8 d	69.5 d
G-5					77.5 d	77.5 d	77.3 d
G-6					60.8 t	60.8 t	60.6 t

Table 2 ^{13}C NMR spectral data of L-Tryptophan and compound Ⅺ

C 位	Tryptophan	Compound Ⅺ
2	126.1 d	125.9 d
3	108.8 s	110.5 s
4a	137.8 s	138.5 s
4	123.4 d	123.4 d
5	119.6 d	120.3 d
6	120.8 d	120.8 d
7	113.3 d	113.3 d
7a	127.8 s	129.3 s
1′	175.5 s	171.4 s
2′	56.4 d	81.3 s
3′	27.6 t	25.4 t
OMe		53.6 q

Table 3 Inhibitory Effect of Compounds of Anthrapuinones and Naphthalene to Recombinant HIV-1 Protease

Compounds	Inhibition (%)	Compounds	Inhibition (%)
I	2.8	Ⅷ	2.1
Ⅱ	5.3	Ⅻ	1.5
Ⅲ	5.1	XIII	6.7
Ⅳ	3.0	XIV	4.0
Ⅴ	1.4	XV	1.0
Ⅵ	1.9	XVI	0
Ⅶ	3.4	XVII	2.8

Reference

1 Dictionary of Chinese Materia Medica. Publishing House of Science and Technology of Shanghai, Shanghai, 1975. 1135

2 Yang XW. China Journal of Chinese Materia Medica, 1996, 21(1):48

3 Kusumoto IT *et al*. Shoyakugaku Zasshi, 1992, 46:190

摘 要 从何首乌(*Polygonum multiflorum* Thunb.)的根中分离出11个化合物,根据光谱学分析分别鉴定为:大黄酚(chrysophanol, Ⅰ)、大黄素甲醚(physcione, Ⅱ)、大黄素(emodin, Ⅲ)、ω-羟基大黄素(citreorosein, Ⅳ)、大黄酚8-O-β-D-吡喃葡萄糖苷(chrysophanol 8-O-β-D-glucopyranoside, Ⅴ)、大黄素甲醚8-O-β-D-吡喃葡萄糖苷(physcione 8-O-β-D-glucopyranoside, Ⅵ)、大黄素8-O-β-D-吡喃葡萄糖苷(emodin 8-O-β-D-glucopyranoside, Ⅶ)、决明酮8-O-β-D-吡喃葡萄糖苷(torachrysone 8-O-β-D-glucopyranoside, Ⅷ)、2,3,5,4′-四羟基芪2-O-β-D-吡喃葡萄糖苷(2,3,5,4′-tetrahydroxystilbene 2-O-β-D-glucopyranoside, Ⅸ)、没食子酸甲基酯(methylgallate, Ⅹ)和吲哚-3-(L-α-氨基-α-羟基丙酸)甲酯〔Indole-3-(L-α-amino-α-hydroxy propionic acid)methyl ester〕。其中,化合物Ⅺ为新的天然产物。上述蒽醌和萘类化合物以及芦荟大黄素(aloe-emodin, Ⅻ)、大黄酸(rhein, ⅩⅢ)、芦荟大黄素8-O-β-D-吡喃葡萄糖苷(aloe-emodin 8-O-β-D-glucopyranoside, ⅩⅣ)、大黄酚8-O-β-D-(6′-O-丙酰基)吡喃葡萄糖苷(chrysophanol 8-O-β-D-(6′-O-malonyl)glucopyranoside, ⅩⅤ)、番泻叶苷A(sennoside A, ⅩⅥ)、和番泻叶苷B(sennoside B, ⅩⅦ)在100μmol/L离体条件下,对重组HIV-1(Recombinant HIV-1)蛋白酶活性无抑制作用。

关键词 何首乌 吲哚衍生物 吲哚-3-(L-α-氨基-α-羟基丙酸)甲酯 重组HIV-1蛋白酶

(1997-05-12收稿)

黑参中的酚甙

兰州医学院药学系(730000) 李 冲* 张承忠 刘 美**

摘 要 从黑参中提取分离出3种酚甙化合物,经化学试验和光谱鉴定它们的结构为β-(3′,4′-dihydroxyphenyl)ethyl-O-α-L-rhamnopyranosyl(1→3)-β-D-glucopyranoside(Ⅰ), acteoside isomer(Ⅱ), plantainoside C(Ⅲ)。

关键词 黑参 酚甙 苯丙素甙

黑参 *Pedicularis decora* France. 为玄参科马先蒿属植物,药用根茎,又名太白参、太白洋参。有"补虚、健脾胃、消炎止痛、滋阴补肾、补中益气之功效,治疗身体虚弱、肾虚骨蒸、潮热关节疼痛,不思饮食"等疾病[2]。分布于陕西、甘肃、四川和湖北等省,资源丰富。本文报道3个酚甙的分离和结构鉴定。

3个化合物均与三氯化铁试剂和Molish试剂呈阳性反应,示为酚甙类化合物。根据MS,^{1}H和^{13}CNMR以及DEPT脉冲试验分析,化合物Ⅰ为酚甙,Ⅱ、Ⅲ为苯丙素甙。

化合物Ⅰ为白色粉末,FAB-MS m/z: 463[M+1]$^+$,综合分析MS、^{1}H及^{13}CNMR推定分子式$C_{20}H_{30}O_{12}$。^{1}HNMR示有鼠李糖甲基δ1.12(3H, d, J=6.3 Hz),苯乙基7位

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