(Supporting Information)

Chemical Constituents from the Peels of *Citrus sudachi*

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Known Compounds from the Peels of *Citrus sudachi*

Following 27 known compounds were isolated from the peels of *C. sudachi*; isoobacunoic acid (**8**),[1] limonin (**9**),[2] methyl deacetylnomilinate (**10**),[3] nomilinic acid (**11**),[4] vanillic acid (**12**),[5] 1*S*,2*S*,4*R*-limonene-1,2-diol (**13**),[6] (+)-4*S*-7-hydroxypiperitone (**14**),[7] methyl ferulate (**15**),[8] ferulic acid (**16**),[9] citrusin III (**17**),[10] citrusin IX (**18**),[11] sudachitin (**6**),[12] 3'-demethoxysudachitin (**7**),[12] 7-methylsudachitin (**19**),[13] xanthomicrol (**20**),[13] jaceosidin (**21**),[14] sudachiin B (**22**),[15] sudachiin C (**23**),[15] prunin (**24**),[16] narirutin (**25**),[17] naringin (**26**),[17] hesperidin (**27**),[17] neohesperidin (**28**),[18] eriocitrin (**29**),[19] poncirin (**30**),[20] hesperetin 7-*O*-(2'',6''-di-*O*-α-rhamnopyranosyl)-β-glucopyranoside (**31**),[17] naringenin 7-*O*-(2'',6''-di-*O*-α-rhamnopyranosyl)-β-glucopyranoside (**32**).[21] Their structure were identified by the analysis of their NMR spectral data, and then by comparison with those of literature listed below, respectively. Eleven compounds (**8**, **10**, **12** – **18**, **24**, **32**) were the first isolation from *C. sudachi*.

Isolation of Known Compounds

The EtOAc soluble fraction (54 g, the extraction and the partion were described in text) was subjected to silica gel column chromatography (1 kg, 11 x 100 cm). The column was eluted with solvents of increasing polarity (*n*-hexane-EtOAc, EtOAc, EtOAc-MeOH, MeOH) to give 15 major fractions (frs. 1-15). Fraction 1 (460 mg) was separated on GPC ($CHCl_3$), Si HPLC ($CHCl_3$-MeOH, 95:5) to give **15** (5 mg). Fraction 3 (477 mg) was separated by Si HPLC ($CHCl_3$-MeOH), GPC (MeOH) to give **12** (3 mg), **13** (15 mg), **19** (6 mg), **20** (10 mg). Fraction 4 (3.7 g) was chromatographed on a silica gel column ($CHCl_3$-MeOH) to give three fractions (frs 2.1-2.3) and **7** (300 mg). Fraction 2.3 was separated by a Toyopearl HW-40 column ($CHCl_3$-MeOH, 1:1), Si

HPLC ($CHCl_3$-MeOH), GPC (MeOH) to give **14** (7 mg), **16** (8 mg), **21** (5 mg). Fraction 5 (1.1 g) was subjected on Si HPLC ($CHCl_3$-MeOH) to give **6** (360 mg). Fraction 6 (2.4 g) was chromatographed on a Toyopearl HW-40 column ($CHCl_3$-MeOH) to give four fractions (frs 6.1-6.4). Fraction 6.3 was separated by Si HPLC ($CHCl_3$-MeOH), GPC (MeOH) to give **8** (13 mg), **10** (44 mg). Fraction 7 (19.0 g) was chromatographed on a silica gel column ($CHCl_3$-MeOH) to give seven fractions (frs 7.1-7.7). Fraction 7.3 was subjected on recrystallization (MeOH); fraction 7.4 subjected on Si HPLC (hexane-EtOAc) to give **9** (1.0 g), **11** (5 mg), respectively. Fraction 11 (3.0 g) was separated by a Sephadex LH-20 (MeOH) column, GPC (MeOH), ODS (MeOH-H_2O) to give **24** (2 mg). Fractions 12 (1.9 g) was recrystallized from MeOH to give **22** (144 mg). Fraction 13 (1.9 g) was separated by a Sephadex LH-20 (MeOH) column, recrystallization (MeOH), GPC (MeOH) to give **25** (138 mg), **29** (60 mg). Fraction 14 (8.3 g) was separated by a Sephadex LH-20 (MeOH) column to give seven fractions (frs 14.1-14.7) and a mixture of **27**, **28** (2.5 g, 3:2). Fraction 14.7 was separated by GPC (MeOH), ODS (MeOH-H_2O) to give **27** (193 mg), **30** (1 mg), a mixture of **25**, **26** (85 mg, 1:1) and that of **26**, **29** (63 mg, 2:5). Fraction 15 (1.5 g) was separated by a Sephadex LH-20 (MeOH) column, GPC (MeOH), ODS (MeOH-H_2O) to give **17** (63 mg), **18** (29 mg), **23** (13 mg), **31** (11 mg).

One percent of *n*-BuOH soluble fraction (3.8 g) was chromatographed on a Sephadex LH-20 (MeOH) column chromatography to give five fractions (frs 1-5). Fraction 2 (1.3 g) was subjected on GPC (MeOH) to give **31** (4 mg), **32** (21 mg). Fraction 3 (460 mg) was separated by GPC (MeOH), ODS (MeOH-H_2O) to give a mixture of **25**, **26** (104 mg, 1:1) and that of **27**, **28** (106 mg, 1:2). Fractions 4 (155 mg) was subjected on GPC

(MeOH) to give a mixture of **25**, **26** (36 mg, 3:2) and that of **27**, **28** (22 mg, 1:1), that of **26**, **29** (37 mg, 1:3).

References and Notes

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