

## Flavonoid Variation in Fronds of *Cyrtomium falcatum* Complex

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岩科 司\*・松本 定\*:オニヤブソテツ(広義)に含まれるフラボノイドの変異

The genus *Cyrtomium* consists of ca. 15 species and is mainly distributed from the Himalayas to Japan (Iwatsuki 1992). Ten species, *Cyrtomium hookerianum* (C. Presl) C. Chr., *C. balansae* (Christ) C. Chr., *C. caryotideum* (Wall. ex Hook. & Grev.) C. Presl, *C. falcatum* sensu lato, *C. fortunei* J. Sm., *C. macrophyllum* (Makino) Tagawa, *C. atropunctatum* Sa. Kurata, *C. microindusium* Sa. Kurata sensu S. Matsumoto in shed., *C. tukusicola* Tagawa and *C. laetevirens* (Hiyama) Nakaike, are native to Japan (Nakaike 1992). Of their species, *C. falcatum* are widely distributed from Japan, Korea, China, Vietnam, Taiwan, India to Hawaii, North America, Europe. Matsumoto (2003) divided the species into *C. falcatum* subsp. *falcatum* sensu S. Matsumoto, subsp. *australe* S. Matsumoto nom. nud. and subsp. *littorale* S. Matsumoto nom. nud. and *C. devexiscapulae* (Koidz.) Ching.

The flavonoids of the genus *Cyrtomium* have been reported from some species. A common flavonol, quercetin 3-O-glucoside was found from *C. falcatum* sensu lato, *C. fortunei*, *C. caryotideum*, *C. tukusicola* and *C. macrophyllum* (Kishimoto 1956a). Kaempferol 3-O-glucoside was also detected from their species except *C. macrophyllum*. Rare kaempferol 7-O-(6"-succinyl)-glucoside was isolated from *C. falcatum* sensu lato and *C. fortunei* (Hiraoka 1978, Hiraoka and Maeda 1979). The common C-glycosylflavones, vitexin and orientin were also found from the same taxa (Hiraoka 1978). Two rare C-methylflavanone glycosides, cyptopterin and cyrtomin, were isolated from *C. falcatum* sensu lato (Kishimoto 1956a). Their aglycones were characterized as 5,7,4'-trihydroxy-6,8-di-C-methylflavanone (farrerol) and 5,7,3',4'-tetrahydroxy-6,8-di-C-methylflavanone (cyrtominetin) (Kishimoto 1956b, 1956c).

More recently, the flavonoids in nineteen *Cyrtomium* and the related three *Cyrtogonellum* and two *Phanerophlebia* taxa were surveyed and chemotaxonically discussed (Iwashina et al. 2006). In this time, two C-methylflavanone glycosides, cyptopterin and cyrtomin were completely identified as farrerol 7-O- $\beta$ -D-glucopyranoside and cyrtominetin 7-O- $\beta$ -D-glucopyranoside, respectively. Flavonoid composition of *C. falcatum* subsp. *falcatum*, subsp. *australe* and subsp. *littorale*, and *C. devexiscapulae* was also surveyed and pointed out the qualitative difference between *C. falcatum* sensu stricto and *C. devexiscapulae*.

In this paper, flavonoid variation among many individuals and populations of *C. falcatum* subspecies, subsp. *falcatum*, subsp. *australe* and subsp. *littorale*, and *C. devexiscapulae* including *C. falcatum* sensu lato are described.

### Materials and Methods

#### Plant materials

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Fourty-five individuals of *C. devexiscapulae*, 239 of *C. falcatum* subsp. *falcatum*, 94 of *C. falcatum* subsp. *australe*, and 74 of *C. falcatum* subsp. *littorale* were used as plant materials for flavonoid survey. The collection sites, accession numbers and flavonoid composition of plant materials are shown in App. 1. Live plants are grown in Tsukuba Botanical Garden, National Science Museum and voucher specimens were deposited in National Science Museum, Japan (TNS).

#### *Extraction and isolation of flavonoids*

Fresh fronds were extracted with MeOH. The flavonoids were isolated by preparative paper chromatography using solvent systems: BAW (*n*-BuOH/HOAc/H<sub>2</sub>O = 4:1:5, upper phase), 15% HOAc and then BEW (*n*-BuOH/EtOH/H<sub>2</sub>O = 4:1:2.2). The isolated flavonoids were purified by Sephadex LH-20 column chromatography (solvent system: 70% MeOH).

#### *Flavonoid composition*

Flavonoid composition was surveyed by two-dimensional paper chromatography (2D-PC) using BAW (1st) and 15%HOAc (2nd) and HPLC using Shim-pack CLC-ODS (I.D. 6.0×150 mm, Shimadzu), at flow-rate: 1.0 ml/min, injection: 10  $\mu$ l, detection: 190–400 nm, and eluent: MeCN/H<sub>2</sub>O/H<sub>3</sub>PO<sub>4</sub> (22:78:0.2).

#### *Identification of flavonoids*

The isolated flavonoids were identified by UV, <sup>1</sup>H and <sup>13</sup>C NMR, complete and mild acid hydrolysis (in 12% HCl, 100°C, 30 min, and 1.2% HCl:MeOH = 1:1, 100°C, 5–40 min, respectively), and direct PC and HPLC comparisons with authentic specimens. UV spectral analysis were performed according to Mabry *et al.* (1970). <sup>1</sup>H and <sup>13</sup>C NMR spectra were performed in pyridine-*d*<sub>5</sub> at 500 MHz (<sup>1</sup>H NMR) and 125 MHz (<sup>13</sup>C NMR). PC, UV, and <sup>1</sup>H and <sup>13</sup>C NMR data of the isolated flavonoids were as follows.

Quercetin 3-*O*-glucoside (Isoquercitrin, 1). PC: Rf 0.58 (BAW), 0.57 (BEW), 0.32 (15%HOAc), 0.17 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - yellow. UV  $\lambda$  max (nm): MeOH 257, 265sh, 296sh, 357; +NaOMe 273, 328, 409 (inc.); +AlCl<sub>3</sub> 275, 433; +AlCl<sub>3</sub>/HCl 269, 299, 361, 399; +NaOAc 273, 326, 394; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 261, 296, 379.

Kaempferol 3-*O*-glucoside (Astragalin, 2). PC: Rf 0.72 (BAW), 0.71 (BEW), 0.40 (15%HOAc), 0.24 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark greenish yellow. UV  $\lambda$  max (nm): MeOH 266, 296sh, 349; +NaOMe 275, 325, 399 (inc.); +AlCl<sub>3</sub> 274, 304, 352, 395; +AlCl<sub>3</sub>/HCl 275, 302, 346, 395; +NaOAc 274, 310, 388; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 267, 296sh, 353.

Kaempferol 3-*O*-glucoside-7-*O*-rhamnoside (3). PC: Rf 0.42 (BAW), 0.43 (BEW), 0.63 (15%HOAc), 0.52 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark greenish yellow. UV  $\lambda$  max (nm): MeOH 266, 342; +NaOMe 273, 387 (inc.); +AlCl<sub>3</sub> 274, 300, 351, 391; +AlCl<sub>3</sub>/HCl 276, 299, 343, 394; +NaOAc 267, 376, 394sh; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 266, 288, 351. <sup>1</sup>H NMR (500 MHz, pyridine-*d*<sub>5</sub>):  $\delta$  8.45 (2H, *d*, *J* = 9.2 Hz, H-2', 6'), 7.26 (2H, *d*, *J* = 8.9 Hz, H-3', 5'), 6.94 (1H, *d*, *J* = 2.1 Hz, H-8), 6.78 (1H, *d*, *J* = 2.1 Hz, H-6), 6.42 (1H, *d*, *J* = 8.6 Hz, glucosyl H-1), 6.26 (1H, *d*, *J* = 1.2 Hz, rhamnosyl H-1), 4.8–4.0 (*m*, sugar protons), 1.66 (3H, *d*, *J* = 5.8 Hz, rhamnosyl Me). <sup>13</sup>C NMR (125 MHz, pyridine-*d*<sub>5</sub>): (kaempferol)  $\delta$  156.9 (C-2), 134.8 (C-3), 178.9 (C-4), 162.3 (C-5), 94.8 (C-6), 162.8 (C-7), 106.9 (C-8), 157.8 (C-9), 103.6 (C-10), 123.0 (C-1'), 132.0 (C-2', C-6'), 116.2 (C-3', C-5'), 161.9 (C-4'); (glucose)  $\delta$  100.4 (C-1), 76.1 (C-2), 78.5 (C-3), 71.5 (C-4), 79.2 (C-5), 62.6 (C-6); (rhamnose)  $\delta$  100.1 (C-1), 71.6 (C-2), 72.4 (C-3), 73.6 (C-4), 71.5 (C-5), 18.7 (C-6).

Orientin (4). PC: Rf 0.24 (BAW), 0.28 (BEW), 0.14 (15%HOAc), 0.06 (5%HOAc); Color UV - dark

purple, UV/NH<sub>3</sub> - greenish yellow. UV  $\lambda$  max (nm): MeOH 257, 268, 349; +NaOMe 270, 330sh, 406 (inc.); +AlCl<sub>3</sub> 274, 424; +AlCl<sub>3</sub>/HCl 263sh, 276, 297, 358, 385sh; +NaOAc 277, 325sh, 397; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 265, 375.

Isoorientin (**5**). PC: Rf 0.42 (BAW), 0.43 (BEW), 0.29 (15%HOAc), 0.15 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - greenish yellow. UV  $\lambda$  max (nm): MeOH 257, 268, 349; +NaOMe 270, 335sh, 409 (inc.); +AlCl<sub>3</sub> 275, 425; +AlCl<sub>3</sub>/HCl 263sh, 277, 296sh, 360, 385sh; +NaOAc 274, 327, 394; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 264, 376.

Orientin 2''-O-glucoside (**6**). PC: Rf 0.20 (BAW), 0.44 (BEW), 0.58 (15%HOAc), 0.49 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark yellow. UV  $\lambda$  max (nm): MeOH 257, 269, 348; +NaOMe 273, 337sh, 409 (inc.); +AlCl<sub>3</sub> 274, 422; +AlCl<sub>3</sub>/HCl 263sh, 276, 298, 358, 387sh; +NaOAc 268sh, 279, 400; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 264, 375. <sup>1</sup>H NMR (500 MHz, pyridine-d<sub>5</sub>):  $\delta$  8.38 (2H, d, *J* = 9.5 Hz, H-2', 6'), 7.90 (1H, *d*, *J* = 7.9 Hz, H-5'), 7.14 (1H, s, H-3), 6.74 (1H, s, H-6), 6.03 (1H, *d*, *J* = 10.1 Hz, glucosyl H-1), 5.83 (1H, *d*, *J* = 10.1 Hz, glucosyl H-1), 4.8-3.9 (*m*, sugar protons). <sup>13</sup>C NMR (125 MHz, pyridine-d<sub>5</sub>): (luteolin)  $\delta$  165.1 (C-2, C-7), 105.3 (C-3), 183.3 (C-4), 162.5 (C-5), 99.2 (C-6), 106.9 (C-8), 155.5 (C-9), 103.6 (C-10), 121.0 (C-1'), 114.9 (C-2'), 147.5 (C-3'), 150.9 (C-4'), 115.6 (C-5'), 120.2 (C-6'); (*O*-glucose)  $\delta$  103.6 (C-1), 74.3 (C-2), 76.4 (C-3), 64.7 (C-4), 76.1 (C-5), 62.6 (C-6); (*C*-glucose)  $\delta$  71.6 (C-1), 83.5 (C-2), 78.6 (C-3), 71.5 (C-4), 83.4 (C-5), 62.7 (C-6).

Isoorientin 2''-O-glucoside (**7**). PC: Rf 0.25 (BAW), 0.37 (BEW), 0.61 (15%HOAc), 0.56 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark yellow. UV  $\lambda$  max (nm): MeOH 257sh, 270, 347; +NaOMe 275, 337sh, 407 (inc.); +AlCl<sub>3</sub> 276, 422; +AlCl<sub>3</sub>/HCl 260sh, 279, 296sh, 357, 385sh; +NaOAc 269sh, 279, 399; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 267, 375. <sup>1</sup>H NMR (500 MHz, pyridine-d<sub>5</sub>):  $\delta$  8.37 (2H, *d*, *J* = 8.5 Hz, H-2', 6'), 7.83 (1H, *dd*, *J* = 2.1 and 8.2 Hz, H-5'), 7.16 (1H, s, H-3), 6.74 (1H, *d*, *J* = 2.1 Hz, H-8), 6.03 (1H, *d*, *J* = 9.5 Hz, glucosyl H-1), 5.89 (1H, *d*, *J* = 9.5 Hz, glucosyl H-1), 4.7-3.9 (*m*, sugar protons). <sup>13</sup>C NMR (125 MHz, pyridine-d<sub>5</sub>): (luteolin)  $\delta$  163.8 (C-2, C-7), 104.0 (C-3), 181.8 (C-4), 156.4 (C-5), 108.7 (C-6), 93.5 (C-8), 160.5 (C-9), 102.7 (C-10), 122.6 (C-1'), 114.6 (C-2'), 146.3 (C-3'), 149.2 (C-4'), 116.8 (C-5'), 119.5 (C-6'); (*O*-glucose)  $\delta$  104.0 (C-1), 74.4 (C-2), 78.4 (C-3), 70.5 (C-4), 78.1 (C-5), 62.6 (C-6); (*C*-glucose)  $\delta$  71.5 (C-1), 83.3 (C-2), 78.5 (C-3), 70.5 (C-4), 80.7 (C-5), 62.6 (C-6).

Vitexin 2''-O-glucoside (**8**). PC: Rf 0.35 (BAW), 0.57 (BEW), 0.64 (15%HOAc), 0.54 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark greenish yellow. UV  $\lambda$  max (nm): MeOH 270, 333; +NaOMe 280, 331, 396 (inc.); +AlCl<sub>3</sub> 277, 304, 348, 382; +AlCl<sub>3</sub>/HCl 278, 303, 342, 382sh; +NaOAc 280, 314, 334, 393; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 271, 345. <sup>1</sup>H NMR (500 MHz, pyridine-d<sub>5</sub>):  $\delta$  8.37 (2H, *d*, *J* = 8.5 Hz, H-2', 6'), 7.82 (2H, *d*, *J* = 8.5 Hz, H-3', 5'), 7.17 (1H, s, H-3), 6.75 (1H, s, H-6), 6.03 (1H, *d*, *J* = 10.1 Hz, glucosyl H-1), 5.83 (1H, *d*, *J* = 9.5 Hz, glucosyl H-1), 4.8-3.9 (*m*, sugar protons). <sup>13</sup>C NMR (125 MHz, pyridine-d<sub>5</sub>): (apigenin)  $\delta$  164.7 (C-2), 103.6 (C-3), 183.3 (C-4), 162.6 (C-5), 99.3 (C-6), 164.3 (C-7), 106.9 (C-8), 157.9 (C-9), 105.4 (C-10), 122.9 (C-1'), 121.6 (C-2', C-6'), 116.8 (C-3', C-5'), 162.7 (C-4'); (*O*-glucose)  $\delta$  103.9 (C-1), 73.4 (C-2), 76.1 (C-3, C-5), 71.6 (C-4), 62.6 (C-6); (*C*-glucose)  $\delta$  71.8 (C-1), 80.7 (C-2), 78.5 (C-3), 71.6 (C-4), 80.6 (C-5), 62.6 (C-6).

Isovitrifolin 2''-O-glucoside (**9**). PC: Rf 0.35 (BAW), 0.67 (BEW), 0.72 (15%HOAc), 0.64 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark greenish yellow. UV  $\lambda$  max (nm): MeOH 270, 333; +NaOMe 280, 331, 396 (inc.); +AlCl<sub>3</sub> 277, 304, 348, 382; +AlCl<sub>3</sub>/HCl 278, 303, 342, 382sh; +NaOAc 280, 314, 334, 393; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 271, 345. <sup>1</sup>H NMR (500 MHz, pyridine-d<sub>5</sub>):  $\delta$  8.37 (2H, *d*, *J* = 8.5 Hz, H-2', 6'), 7.82 (2H, *d*, *J* = 8.5 Hz, H-3', 5'), 7.17 (1H, s, H-3), 6.93 (1H, s, H-8), 6.03 (1H, *d*, *J* = 10.1 Hz, glucosyl H-1), 5.83 (1H, *d*, *J* = 9.5 Hz, glucosyl H-1), 4.8-3.9 (*m*, sugar protons). <sup>13</sup>C NMR (125 MHz, pyridine-d<sub>5</sub>):

(apigenin)  $\delta$  164.7 (C-2, C-7), 103.6 (C-3), 183.3 (C-4), 162.6 (C-5), 111.7 (C-6), 94.5 (C-8), 157.9 (C-9), 105.4 (C-10), 122.9 (C-1'), 129.6 (C-2', C-6'), 116.9 (C-3', C-5'), 162.7 (C-4'); (*O*-glucose)  $\delta$  103.9 (C-1), 71.8 (C-2, C-5), 73.4 (C-3), 71.6 (C-4), 62.6 (C-6); (C-glucose)  $\delta$  71.8 (C-1), 83.1 (C-2), 78.5 (C-3), 71.6 (C-4), 80.7 (C-5), 62.6 (C-6).

**Vicenin-2 (10).** PC: Rf 0.23 (BAW), 0.25 (BEW), 0.52 (15%HOAc), 0.37 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark yellow. UV  $\lambda$  max (nm): MeOH 273, 332; +NaOMe 283, 334, 400 (inc.); +AlCl<sub>3</sub> 281, 305, 351, 386sh; +AlCl<sub>3</sub>/HCl 280, 304, 347, 384sh; +NaOAc 282, 315sh, 336, 397; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 275sh, 285, 321, 348sh, 414sh.

**Farrerol 7-*O*-glucoside (11).** PC: Rf 0.75 (BAW), 0.83 (BEW), 0.54 (15%HOAc), 0.29 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - greenish dark purple. UV  $\lambda$  max (nm): MeOH 283, 361; +NaOMe 244sh, 287, 392 (inc.); +AlCl<sub>3</sub> 287, 312sh, 362; +AlCl<sub>3</sub>/HCl 285, 311sh, 363; +NaOAc 284, 363; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 285, 363. <sup>1</sup>H NMR (500 MHz, pyridine-*d*<sub>5</sub>):  $\delta$  12.59 (1H, *s*, 5-OH), 7.55 (2H, *d*, *J* = 7.9 Hz, H-2', 6'), 7.09 (2H, *dd*, *J* = 1.8 and 9.8 Hz, H-3', 5'), 5.3-5.5 (Obsc. with H-2 and glucosyl H-1), 4.6-4.0 (*m*, sugar protons), 3.25 (1H, *dd*, *J* = 10.4 and 17.1 Hz, H-3<sub>ax</sub>), 2.91 (1H, *dd*, *J* = 3.4 and 6.7 Hz, H-3<sub>eq</sub>), 2.65 (3H, *s*, 6 or 8-Me), 2.55 (3H, *s*, 6 or 8-Me). <sup>13</sup>C NMR (125 MHz, pyridine-*d*<sub>5</sub>): (farrerol)  $\delta$  79.1 (C-2), 43.6 (C-3), 198.6 (C-4), 159.5 (C-5, C-9), 112.1 (C-6), 162.7 (C-7), 111.2 (C-8), 105.9 (C-10), 130.9 (C-1'), 128.6 (C-2', C-6'), 116.5 (C-3', C-5'), 158.4 (C-4'), 10.0 (6 or 8-Me), 9.5 (6 or 8-Me); (glucose)  $\delta$  105.8 (C-1), 75.8 (C-2), 78.5 (C-3), 71.6 (C-4), 78.8 (C-5), 62.7 (C-6).

**Cyrtominetin 7-*O*-glucoside (Cyrtomin, 12).** PC: Rf 0.66 (BAW), 0.72 (BEW), 0.45 (15%HOAc), 0.25 (5%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark purple. UV  $\lambda$  max (nm): MeOH 285, 350; +NaOMe 244sh, 289, 390 (inc.); +AlCl<sub>3</sub> 289, 310sh, 357; +AlCl<sub>3</sub>/HCl 288, 307sh, 360; +NaOAc 286, 350; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 286, 351. <sup>1</sup>H NMR (500 MHz, pyridine-*d*<sub>5</sub>):  $\delta$  12.59 (1H, *s*, 5-OH), 7.30 (2H, *d*, *J* = 8.2 Hz, H-2', 6'), 7.06 (1H, *d*, *J* = 1.8 Hz, H-5'), 5.3-5.5 (Obsc. with H-2 and glucosyl H-1), 4.6-4.0 (*m*, sugar protons), 3.25 (1H, *dd*, *J* = 10.4 and 17.1 Hz, H-3<sub>ax</sub>), 2.91 (1H, *dd*, *J* = 3.4 and 6.7 Hz, H-3<sub>eq</sub>), 2.64 (3H, *s*, 6 or 8-Me), 2.52 (3H, *s*, 6 or 8-Me). <sup>13</sup>C NMR (125 MHz, pyridine-*d*<sub>5</sub>): (cyrtominetin)  $\delta$  79.3 (C-2), 43.6 (C-3), 198.7 (C-4), 159.5 (C-5, C-9), 112.1 (C-6), 162.8 (C-7), 111.2 (C-8), 105.9 (C-10), 130.9 (C-1'), 115.1 (C-2'), 147.6 (C-3'), 147.9 (C-4'), 116.6 (C-5'), 118.3 (C-6'), 10.1 (6 or 8-Me), 9.4 (6 or 8-Me); (glucose)  $\delta$  105.8 (C-1), 75.8 (C-2), 78.5 (C-3), 71.6 (C-4), 78.8 (C-5), 62.7 (C-6).

**Farrerol (13).** PC: Rf 0.86 (BAW), 0.88 (BEW), 0.90 (Forestal: HOAc:HCl/H<sub>2</sub>O = 30:3:10), 0.14 (15%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark purple. UV  $\lambda$  max (nm): MeOH 294, 348; +NaOMe 243sh, 338 (inc.); +AlCl<sub>3</sub> 316, 361, 407sh; +AlCl<sub>3</sub>/HCl 315, 364sh, 400; +NaOAc 290, 338; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 296, 348. <sup>1</sup>H NMR (500 MHz, pyridine-*d*<sub>5</sub>):  $\delta$  13.06 (1H, *s*, 5-OH), 7.60 (1H, *d*, *J* = 1.8 Hz, H-6'), 7.58 (1H, *d*, *J* = 2.1 Hz, H-2'), 7.28 (1H, *d*, *J* = 1.8 Hz, H-5'), 7.25 (1H, *d*, *J* = 2.1 Hz, H-3'), 5.52 (1H, *dd*, *J* = 3.1 and 12.5 Hz, H-2), 3.29 (1H, *dd*, *J* = 12.5 and 17.1 Hz, H-3<sub>ax</sub>), 2.97 (1H, *dd*, *J* = 3.2 and 16.9 Hz, H-3<sub>eq</sub>), 2.44 (3H, *s*, 6 or 8-Me), 2.34 (3H, *s*, 6 or 8-Me). <sup>13</sup>C NMR (125 MHz, pyridine-*d*<sub>5</sub>):  $\delta$  79.1 (C-2), 43.4 (C-3), 197.2 (C-4), 159.4 (C-5), 104.6 (C-6), 160.1 (C-7), 103.6 (C-8), 164.3 (C-9), 102.9 (C-10), 130.3 (C-1'), 128.6 (C-2', C-6'), 116.5 (C-3', C-5'), 158.5 (C-4'), 9.0 (6 or 8-Me), 8.5 (6 or 8-Me).

**Cyrtominetin (14).** PC: Rf 0.86 (BAW), 0.88 (BEW), 0.85 (Forestal), 0.14 (15%HOAc); Color UV - dark purple, UV/NH<sub>3</sub> - dark purple. UV  $\lambda$  max (nm): MeOH 294, 345; +NaOMe 243sh, 337 (inc.); +AlCl<sub>3</sub> 315, 360, 405sh; +AlCl<sub>3</sub>/HCl 315, 370, 405sh; +NaOAc 290, 338; +NaOAc/H<sub>3</sub>BO<sub>3</sub> 295, 346. <sup>1</sup>H NMR (500 MHz, pyridine-*d*<sub>5</sub>):  $\delta$  13.01 (1H, *s*, 5-OH), 7.33 (1H, *d*, *J* = 1.2 Hz, H-6'), 7.30 (1H, *d*, *J* = 1.2 Hz, H-2'), 7.10 (1H, *d*, *J* = 1.5 Hz, H-5'), 5.52 (1H, *dd*, *J* = 2.8 and 12.2 Hz, H-2), 3.29 (1H, *dd*, *J* = 12.4 and 16.9 Hz, H-3<sub>ax</sub>), 3.02 (1H, *dd*, *J* = 3.1 and 10.1 Hz, H-3<sub>eq</sub>), 2.42 (3H, *s*, 6 or 8-Me), 2.30 (3H, *s*, 6 or 8-Me). <sup>13</sup>C

NMR (125 MHz, pyridine-*d*<sub>5</sub>): δ 79.1 (C-2), 43.5 (C-3), 197.2 (C-4), 159.4 (C-5), 104.4 (C-6), 160.0 (C-7), 103.7 (C-8), 164.2 (C-9), 102.9 (C-10), 131.2 (C-1'), 115.1 (C-2'), 147.6 (C-3'), 147.8 (C-4'), 116.6 (C-5'), 118.3 (C-6'). 9.0 (6 or 8-Me), 8.4 (6 or 8-Me).

## Results and Discussion

Three flavonols (**1** - **3**), seven *C*-glycosylflavones (**4** - **10**) and four *C*-methylflavanones (**11** - **14**) were isolated from *C. falcatum* subsp. *falcatum*, subsp. *australe* and subsp. *littorale*, and *C. devexiscapulae*. Of their flavonoids, characterization of four *C*-methylflavanones have already been described by Iwashina *et al.* (2006). Two flavonols, quercetin 3-*O*-glucoside (**1**) and kaempferol 3-*O*-glucoside (**2**), and three *C*-glycosylflavones, orientin (**4**), isoorientin (**5**) and vicenin-2 (**10**) were identified by UV spectral properties and direct PC and HPLC comparisons with authentic specimens. Another flavonol, kaempferol 3-*O*-glucoside-7-*O*-rhamnoside (**3**), and four *C*-glycosylflavone *O*-glycosides, orientin 2''-*O*-glucoside (**6**), isoorientin 2''-*O*-glucoside (**7**), vitexin 2''-*O*-glucoside (**8**) and isovitexin 2''-*O*-glucoside (**9**) were identified based on UV, <sup>1</sup>H and <sup>13</sup>C NMR spectral data (see Materials and Methods).

Among three *C. falcatum* subspecies and *C. devexiscapulae*, four *C*-methylflavanones, farrerol and cyrtominetin, and their 7-*O*-glucosides were only detected from *C. devexiscapulae* (Table 1), showing that the presence of flavanones is a chemical character of *C. devexiscapulae* but not *C. falcatum* *sensu lato*. On the other hand, other flavonols and *C*-glycosylflavones were not detectable except a few individuals, e.g., 417: Oura, Shimoda, Shizuoka Pref.; 437: Fukue Is., Nagasaki Pref. (presence of flavonoid **3**), 444-446: Hou-ji temple, Putuo-shan, Zhoushan Qundao Isls., Zhejiang Prov. China (presence of flavonoid **10**) and so on (App. 1).

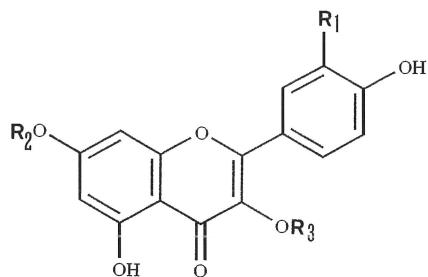
Flavonols and *C*-glycosylflavones can commonly detect in three *C. falcatum* subspecies. The appearance of *C*-glycosylflavones was apparently the same among three subspecies, i.e., major 2''-*O*-

Table 1. Distribution of the flavonoids in *Cyrtomium falcatum* subsp. *falcatum*, subsp. *australe* and subsp. *littorale*, and *C. devexiscapulae* including *C. falcatum* *sensu lato*

Taxa	Flavonols			<i>C</i> -Glycosylflavones							Flavanones			
	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>	<b>10</b>	<b>11</b>	<b>12</b>	<b>13</b>	<b>14</b>
<i>C. falcatum</i> subsp. <i>falcatum</i> (239)	36 15%	36 15%	168 70%	8 3%	6 3%	153 64%	105 44%	119 50%	118 49%	17 7%	0 0	0 0	0 0	0 0
<i>C. falcatum</i> subsp. <i>australe</i> (94)	85 90%	85 90%	78 83%	11 12%	8 9%	64 68%	62 66%	52 55%	51 54%	23 24%	0 0	0 0	0 0	0 0
<i>C. falcatum</i> subsp. <i>littorale</i> (74)	74 100%	74 100%	0	7 9%	6 8%	37 50%	38 51%	17 23%	17 23%	0	0 0	0 0	0 0	0 0
<i>C. devexiscapulae</i> (45)	0 4%	0 0	2 0	0 0	0 0	0 0	0 0	0 0	0 0	6 13%	45 100%	45 100%	38 90%	38 90%

( ) = No. of individuals examined.

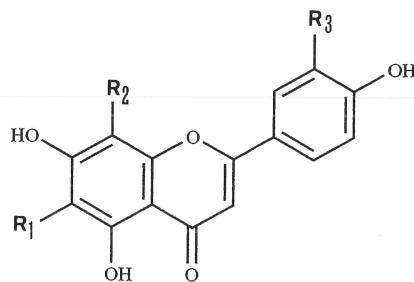
**1** = Quercetin 3-*O*-glucoside, **2** = Kaempferol 3-*O*-glucoside, **3** = Kaempferol 3-*O*-glucoside-7-*O*-rhamnoside, **4** = Orientin, **5** = Isoorientin, **6** = Orientin 2''-*O*-glucoside, **7** = Isoorientin 2''-*O*-glucoside, **8** = Vitexin 2''-*O*-glucoside, **9** = Isovitexin 2''-*O*-glucoside, **10** = Vicenin-2, **11** = Farrerol 7-*O*-glucoside, **12** = Cyrtominetin 7-*O*-glucoside, **13** = Farrerol and **14** = Cyrtominetin.



R<sub>1</sub> = OH, R<sub>2</sub> = H, R<sub>3</sub> = glucosyl: Quercetin 3-O-glucoside (Isoquercitrin, **1**)

R<sub>1</sub> = R<sub>2</sub> = H, R<sub>3</sub> = glucosyl: Kaempferol 3-O-glucoside (Astragalin, **2**)

R<sub>1</sub> = H, R<sub>2</sub> = rhamnosyl, R<sub>3</sub> = glucosyl: Kaempferol 3-O-glucoside-7-O-rhamnoside (**3**)



R<sub>1</sub> = H, R<sub>2</sub> = C-glucosyl, R<sub>3</sub> = OH: Orientin (**4**)

R<sub>1</sub> = C-glucosyl, R<sub>2</sub> = H, R<sub>3</sub> = OH: Isoorientin (**5**)

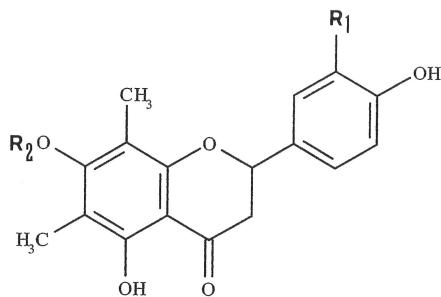
R<sub>1</sub> = H, R<sub>2</sub> = 2'-O-gluco-C-glucosyl, R<sub>3</sub> = OH: Orientin 2'-O-glucoside (**6**)

R<sub>1</sub> = 2'-O-gluco-C-glucosyl, R<sub>2</sub> = H, R<sub>3</sub> = OH: Isoorientin 2'-O-glucoside (**7**)

R<sub>1</sub> = R<sub>3</sub> = H, R<sub>2</sub> = 2'-O-gluco-C-glucosyl: Vitexin 2'-O-glucoside (**8**)

R<sub>1</sub> = 2'-O-gluco-C-glucosyl, R<sub>2</sub> = R<sub>3</sub> = H: Isovitexin 2'-O-glucoside (**9**)

R<sub>1</sub> = R<sub>2</sub> = C-glucosyl, R<sub>3</sub> = H: Vicenin-2 (**10**)



R<sub>1</sub> = H, R<sub>2</sub> = glucosyl: Farrerol 7-O-glucoside (**11**)

R<sub>1</sub> = OH, R<sub>2</sub> = glucosyl: Cyrtominetin 7-O-glucoside (**12**)

R<sub>1</sub> = R<sub>2</sub> = H: Farrerol (**13**)

R<sub>1</sub> = OH, R<sub>2</sub> = H: Cyrtominetin (**14**)

Fig. 1. Chemical structures of flavonoids isolated from *Cyrtomium falcatum* complex.

glucosides (**6**, **7**, **8** and **9**) and minor orientin (**4**), isoorientin (**5**) and vicenin-2 (**10**). However, major flavonols were different among three subspecies. Major flavonol of subsp. *falcatum* is kaempferol 3-*O*-glucoside-7-*O*-rhamnoside (**3**), but those of subsp. *littorale* are quercetin 3-*O*-glucoside (**1**) and kaempferol 3-*O*-glucoside (**2**). Three flavonols appear in subsp. *australe* as major compounds.

Of the individuals of *C. falcatum sensu lato* used as plant materials in this experiment, flavonols and *C*-glycosylflavones were not found or small amount from some individuals, e.g., 29: Ohse, Minami-Izu, Shizuoka Pref.; 52: Tajiri-hama, Hitachi, Ibaraki Pref.; 128-130: Senkaku-wan, Aikawa, (Sado Is.), Niigata Pref.; 132-133: Nagasaki-hana, Yamakawa, Kagoshima Pref. (App. 1). As the reason, since many individuals are cultivated in the greenhouse, the flavonoid biosynthesis may be controlled. On the other hand, it is noteworthy that the *C*-methylflavanones were abundantly synthesized with or without the cultivation of the plants in the greenhouse.

### Acknowledgements

Authors thank to Dr. Toshiyuki Nakaike for some Japanese plant materials, Prof. Wu Su-Kunming, Academic Sinica, for Chinese materials, Mr. Shing-Ginn Lee and Mr. Jinn-Fen Chen, Taitung District Agricultural Improvement Station, Taiwan, for Taiwanese material collection, and also Dr. Junichi Kitajima, Laboratory of Pharmacognosy, Showa Pharmaceutical University, for NMR measurement. This study was financially supported by Monbu-Kagaku-sho Scientific Research Program (No. 11640703) from the Ministry of Education, Culture, Sports, Science and Technology, Japan.

### Summary

Fourty-two individuals of *Cyrtomium devescapulae*, 239 of *C. falcatum* subsp. *falcatum*, 97 of *C. falcatum* subsp. *australe*, 74 of *C. falcatum* subsp. *littorale*, which are included in *C. falcatum sensu lato*, were analysed for flavonoid compounds. Three flavonols, quercetin 3-*O*-glucoside (**1**), kaempferol 3-*O*-glucoside (**2**) and kaempferol 3-*O*-glucoside-7-*O*-rhamnoside (**3**), seven *C*-glycosylflavones, orientin (**4**), isoorientin (**5**), orientin 2''-*O*-glucoside (**6**), isoorientin 2''-*O*-glucoside (**7**), vitexin 2''-*O*-glucoside (**8**), isovitexin 2''-*O*-glucoside (**9**) and vicenin-2 (**10**), and four *C*-methylflavanones, farrerol 7-*O*-glucoside (**11**), cyrtominetin 7-*O*-glucoside (**12**), farrerol (**13**) and cyrtominetin (**14**), were isolated from the fronds. Of their flavonoids, occurrence of four flavanones was restricted to *C. devescapulae*, showing that the presence of them is a chemical character of *C. devescapulae* alone. The appearance of *C*-glycosylflavones was apparently the same among three *C. falcatum* subspecies. However, occurrence of the flavonols were quantitatively different among three *C. falcatum* subspecies, i.e., major compound of subsp. *falcatum* is **3**, but those of subsp. *littorale* are **1** and **2**. In subsp. *australe*, all three flavonols **1**, **2** and **3** were present as major compounds.

### 摘要

広義のオニヤブソテツに属するオニヤブソテツ(狭義)239個体、その亜種であるヒメオニヤブソテツ74個体とムニンオニヤブソテツ94個体、さらに別種とされるナガバヤブソテツ45個体のフラボノイド組成がHPLCなどによって解析された。3種類のフラボノール、Quercetin 3-*O*-glucoside (**1**), Kaempferol 3-*O*-glucoside (**2**)およびKaempferol 3-*O*-glucoside-7-*O*-rhamnoside (**3**)、7種

類のC-グリコシルフラボン、Orientin (4), Isoorientin (5), Orientin 2''-O-glucoside (6), Isoorientin 2''-O-glucoside (7), Vitexin 2''-O-glucoside (8), Isovitetexin 2''-O-glucoside (9)およびVicenin-2 (10)、そして4種類のC-メチルフラバノン、Farrerol 7-O-glucoside (11), Cyrtominetin 7-O-glucoside (12), Farrerol (13)およびCyrtominetin (14)が分離同定された。これらのフラボノイドのうち、広義のオニヤブソテツに存在するとされていたC-メチルフラバノンはナガバヤブソテツからのみ検出され、オニヤブソテツ(広義)の化学的特徴ではなく、ナガバヤブソテツのみの特徴であることが判明した。一方、オニヤブソテツ(狭義)とその2亜種のフラボノイドはフラボノールとC-グリコシルフラボンであった。これらのうち、C-グリコシルフラボンの出現頻度は3亜種でほとんど変わらなかったが、フラボノールについてはオニヤブソテツ(狭義)でKaempferol 3-O-glucoside-7-O-rhamnoside (3)が主要成分であるのに対して、ヒメオニヤブソテツではQuercetin 3-O-glucoside (1)とKaempferol 3-O-glucoside (2)が主要成分であった。さらにムニンオニヤブソテツでは、そのいずれもが主要フラボノイドとして存在した。なお、ナガバヤブソテツからはフラボノールとC-グリコシルフラボンはほとんど検出されなかった。

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App. 1 . Collection sites, acc. no. and flavonoid composition ( ) of *Cyrtomium falcatum* subsp. *falcatum*, subsp. *australe* and subsp. *littorale*, and *C. devexiscapulae*

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*Cyrtomium falcatum* subsp. *falcatum*

- 1: Funaura, Taketomi (Iriomote Is.), Okinawa Pref. (沖縄県竹富町船浦) (竹富島) ; TBG54814 (1,2,3,6,8,9), 2, 3: Sumiyoshi, Taketomi (Iriomote Is.), Okinawa Pref. (沖縄県竹富町住吉) (竹富島) ; SM880330-5, SM880330-6 (3), 4, 5: Gyokusen-do, Gushikami, Okinawa Pref. (沖縄県具志頭村玉泉洞) ; TBG54774, TBG54773 (3,6,8,9), 6-8: White-beach, Katsuren, Okinawa Pref. (沖縄県勝連町ホワイトビーチ) ; TBG54777, TBG54775, TBG54776 (1,2,3,6,7,8,9), 9: Syurei-no-mon, Naha, Okinawa Pref. (沖縄県那覇市守礼門) ; SM880330-1 (3,6,8,9), 10-12: Yaku (Yaku Is.), Kagoshima Pref. (鹿児島県屋久町) (屋久島) ; SM861011-28, SM861011-29, SM861011-32 (3,6,7,8,9), 13-15: Kawachi, Kumamoto Pref. (熊本県川内町) ; TBG27913, TBG27914, TBG27916 (3,6,8,9), 16: Shikimi, Nagasaki, Nagasaki Pref. (長崎県長崎市式見町) (3,6,8,9), 17: Hiwasa, Tokushima Pref. (徳島県日和佐町) ; TBG53945 (1,2,3,6,7,8,9), 18: Kawano, Aki, Kochi Pref. (高知県安芸市河野) ; TBG53949 (3), 19: Arisaki, Tosa-shimizu, Kochi Pref. (高知県土佐清水市蟻岬) ; TBG53962 (3), 20: Onigasaki, Kumano, Mie Pref. (三重県熊野市鬼ヶ崎) ; SM840210-1 (6,7,8,9,10), 21-23: Mikura Is., Tokyo (東京都御藏島) ; SM810707, SM810707-12, SM810709-8 (5,6,7), 24: Kaeshi-hama, Kozu Is., Tokyo (東京都神津島返浜) ; SM810711-27 (6,8,9), 25-28: Usuki, Miyake Is., Tokyo (東京都三宅島薄木) ; SM801010-6, SM801010-7, SM801010-8, SM801010-9 (1,2,3,6,7,8,9), 29, 30: Ohse, Minami-Izu, Shizuoka Pref. (静岡県南伊豆町大瀬) ; SM760516-26, 760516-27 (none), 35-37: Sotoura, Shimoda, Shizuoka Pref. (静岡県下田市外浦) ; SM800211-5, SM800211-7, SM800515-8 (1,2,3,6,7,8,9,10), 38-40: Jogasaki, Ito, Shizuoka Pref. (静岡県伊東市城ヶ崎) ; SM800615-3, SM800615-4, SM800615-22 (1,2,3,6,7,8,9), 41, 42: Oura-kaigan, Shimoda, Shizuoka Pref. (静岡県下田市大浦海岸) ; SM811004-14, SM811004-15 (3,6,7,8,9), 43: Sotoura-iriguchi, Shimoda, Shizuoka Pref. (静岡県下田市外浦入口) ; SM811009-7 (none), 44: Suzaki, Shimoda, Shizuoka Pref. (静岡県下田市須崎) ; SM811011-20 (3,6,7,8,9), 45-50: Kisami, Shimoda, Shizuoka Pref. (静岡県下田市吉佐美) ; SM820315-10, SM820315-11, SM820315-12, SM820315-13, SM820315-16, SM820315-22 (1,2,3,6,7,8,9,10), 51: Mochimune, Shizuoka, Shizuoka Pref. (静岡県静岡市用宗) ; SM730902-4 (3,6,7,8,9), 52: Tajirihama, Hitachi, Ibaraki Pref. (茨城県日立市田尻浜) ; SM8301120-10 (none), 53-57: Ajigaura, Nakaminato, Ibaraki Pref. (茨城県那珂湊市阿字浦) ; SM820509-1, SM820509-2, SM820509-3, SM820509-4, SM820509-5 (3,4,5,6,7,8,9,10), 58: Umiyama, Mikata, Fukui Pref. (福井県三方町海山) ; SM870328-1 (1,2,3,6,7,8,9,10), 59: Sugao, Fukui, Fukui Pref. (福井県福井市菅生) ; TBG33273 (3,6,7,8,9), 31-34, 60: Nanaura-kaigan, Monzen, Ishikawa Pref. (石川県門前町七浦海岸) ; SM770502-30, SM770502-31-1, SM770502-31-2, SM770502-31, SM770502-23 (1,2,3,4,5,6,7,8,9,10), 61-63: Kinkazan Is., Oshika, Miyagi Pref. (宮城県牡鹿町金華山島) ; SM730923-1, SM730923-8, SM730923-9 (none), 64, 65: Onoaida, Yaku (Yaku Is.), Kagoshima Pref. (鹿児島県屋久町屋之間) (屋久島) ; SM861011-30, SM861011-33 (3), 66-70, 72-74: Daio-saki, Daio, Mie Pref. (三重県大王町大王崎) ; SM870401-2, SM870401-3, SM870401-4, SM870401-7, SM870401-8, SM870401-3, SM870401-10, SM870401-12 (3,6,7,8,9), 71: Kakizaki, Shimoda, Shizuoka Pref. (静岡県下田市柿崎) (3,6,7), 75, 76: Borawazawa-kaigan, Hachijo (Hachijo Is.), Tokyo (東京都八丈町洞輪沢海岸) (八丈島) ; SM890127-3, SM890127-19 (1,2,6), 77: Borawazawa, Hachijo (Hachijo Is.), Tokyo (東京都八丈町洞輪沢) (八丈島) ; SM890127-20 (1,2,6), 78: Sueyoshi, Hachijo (Hachijo Is.), Tokyo (東京都八丈町末吉) (八丈島) ; SM890127-17 (1,2,6), 79, 81: Nakanogo, Hachijo (Hachijo Is.), Tokyo (東京都八丈町中之郷) (八丈

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## App. I.

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*Cyrtomium falcatum* subsp. *australe*

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(東京都小笠原村母島石門山東側) (小笠原諸島) (1,2,3), 296-298: Omoto-hama, Haha-jima Is., Ogasawara Isls., Tokyo (東京都小笠原村母島万年青浜) (小笠原諸島) (1,2,3), 299: Omoto-hama - Horai-kaigan, Haha-jima Is., Ogasawara Isls., Tokyo (東京都小笠原村母島万年青浜一蓬萊海岸) (小笠原諸島) (1,2,3), 300: East side of Ten-no-ura-one, Chichi-jima Is., Ogasawara Isls., Tokyo (東京都小笠原村父島天ノ浦尾根東側) (小笠原諸島) (1,2,3), 301, 302: Near Nishi-kaigan-one, Chichi-jima Is., Ogasawara Isls., Tokyo (東京都小笠原村父島西海岸尾根付近) (小笠原諸島) (1,2,3), 303: Gusuku, Yoron (Yoron Is.), Kagoshima Pref. (鹿児島県与論町城) (与論島); SM940224-1-2 (1,2,3,6,7,8,9,10), 304-306: Imae, Yoron (Yoron Is.), Kagoshima Pref.; (鹿児島県与論町伊前) (与論島); SM940224-2, SM940224-3, SM940224-5 (1,2,3,6,7,8,9,10), 307: Tamina-saki, Chi-na (Okinoerabu Is.), Kagoshima Pref. (鹿児島県知名町田皆崎) (沖永良部島); SM940228-5 (3,6,7,8,9,10), 308, 313-315, 317, 318: Sakiyama-hana, Fukue (Fukue Is.), Nagasaki Pref. (長崎県福江市崎山鼻) (福江島); SM940323-24-3, SM940323-18-1, SM940323-19-1, SM940323-19-2, SM940323-24-1, SM940323-24-2 (1,2,3,4,5,6,7,8,9,10), 309, 321: Usudake - Shiotsu, Fukue (Fukue Is.), Nagasaki Pref. (長崎県福江市臼岳一塙津) (福江島); SM940323-28-2, SM940323-28-1 (1,2,3,6,7,8,9,10), 310, 311: Kindahana, Yonaguni (Yonaguni Is.), Okinawa Pref. (沖縄県与那国町キンダハナ) (与那国島); SM940322-1, SM940322-2 (1,2,3,6,7), 312: Kukurabari, Yonaguni (Yonaguni Is.), Okinawa Pref. (沖縄県与那国町ククラバリ) (与那国島); SM940322-3 (3,6,7,8,9), 316: Mukai-machi, Fukue (Fukue Is.), Nagasaki Pref. (長崎県福江市向町) (福江島); SM940323-23 (1,2,6,7,8,9,10), 319, 320: Minodake - Usudake, Fukue (Fukue Is.), Nagasaki Pref. (長崎県福江市箕岳一臼岳) (福江島); SM940323-27-1, SM940323-27-2 (1,2,3,6,7,8,9), 322: Abunze, Fukue (Fukue Is.), Nagasaki Pref. (長崎県福江市鐘瀬) (福江島); SM940323-30 (1,2,3,6,7,8,9), 323: Nagamine, Tomie (Fukue Is.), Nagasaki Pref. (長崎県富江町長峰) (福江島); SM940323-37 (1,2,3,6,7,8,9), 324-326: Ose-zaki, Tamanoura (Fukue Is.), Nagasaki Pref. (長崎県玉ノ浦町大瀨崎) (福江島); SM940323-38, SM940323-39, SM940323-40 (1,2,3,6,7,8,9), 327: Arakawa, Tamanoura (Fukue Is.), Nagasaki Pref. (長崎県玉ノ浦町荒川) (福江島); SM940323-41 (1,2,3,6,7), 328-330: Awa, Nago, Okinawa Pref. (沖縄県名護市安波) ; SM911126-1, SM911126-2, SM911126-3 (1,2,3,4,5,6,7), 331, 332: Sirahama-ura, Fukue (Fukue Is.), Nagasaki Pref. (長崎県福江市白浜浦) (福江島); SM941207-51, SM941207-52 (1,2,3,6,8,9), 336: Zhongai bridge, Lanyu Isl., Taiwan (蘭嶼島・忠愛橋, 台湾); Matsumoto121 (none), 337: Lanyu Isl., Taiwan (蘭嶼島, 台湾); Matsumoto122 (none).

*Cyrtomium falcatum* subsp. *littorale*

338-341: Murasato, Mikura Is., Tokyo (東京都御藏島村里); SM810709-13, SM810709-7, SM810709-10, SM810709-12 (1,2,6,7), 342: Kaeshi-hama, Kozu Is., Tokyo (東京都神津島返浜); SM810711-30 (1,2), 343-345: Usuki, Miyake (Miyake Is.), Tokyo (東京都三宅村薄木) (三宅島); TBG13479, TBG13480, TBG13481 (1,2,6,7,8,9), 346, 347, 365: Suzuki-Tsumekisaki, Shimoda, Shizuoka Pref. (静岡県下田市須崎爪木崎); SM730527-31, SM730708-2, SM730527-15 (1,2,3,6,7,8,9), 348: Jogasaki, Ito, Shizuoka Pref. (静岡県伊東市城ヶ崎); SM760515-43 (1,6,7,8,9), 349: Nanaura-kaigan, Monzen, Ishikawa Pref. (石川県門前町七浦海岸); SM770502-25 (1,2,6,7), 350: Oshima-ga-ne, Jogasaki, Ito, Shizuoka Pref. (静岡県伊東市城ヶ崎大島ヶ根); SM800615-2 (1,2,6,7), 351, 352: Kobai, Jogasaki, Ito, Shizuoka Pref. (静岡県伊東市城ヶ崎コバイ); SM800615-13, SM800615-14 (1,2,4,5,6,7,8,9), 353: Mizugasaki, Jogasaki, Ito, Shizuoka Pref. (静岡県伊東市城ヶ崎水ヶ崎); SM811003-1 (1,2,6,7), 354-359: Nichiren-saki, Jogasaki, Ito, Shizuoka Pref. (静岡県伊東市城ヶ崎日蓮崎); SM811003-3, M811003-9, SM811003-12, SM811003-13, SM811003-14, SM811003-19 (1,2,6,7), 360-362: Futo-ko,

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Jogasaki, Ito, Shizuoka Pref. (静岡県伊東市城ヶ崎富戸港) ; SM820316-28, SM820316-29, SM820316-36 (**1,2,4,5,6,7,8,9**), 363: Tajiri-hama, Hitachi, Ibaraki Pref. (茨城県日立市田尻浜) ; SM831120-8 (**1,2**), 364: Daio-saki, Daio, Mie Pref. (三重県大王町大王崎) ; SM870401-5 (**1,2**), 366, 367, 372: Near Senkaku-wan, Aikawa (Sado Is.), Niigata Pref. (新潟県相川町尖閣湾周辺) (佐渡) ; SM890327-1, SM890327-2, SM890327-5 (**1,2,6,7**), 368-370: Itsuura-kaigan, Kita-ibaraki, Ibaraki Pref. (茨城県北茨城市五浦海岸) ; SM890425-1, SM890425-3, SM890425-5 (**1,2,6,7,8,9**), 371: Ashizuri-misaki, Tosa-shimizu, Kochi Pref. (高知県土佐清水市足摺岬) ; SM891121-1 (**1,2,6,7**), 373-376: Sandan-beki, Shirahama, Wakayama Pref. (和歌山県白浜町三段壁) ; SM920919-12, SM920919-13, SM920919-14, SM920919-15 (**1,2,6,7**), 377, 378: Shiono-misaki, Kushimoto, Wakayama Pref. (和歌山県串本町潮岬) ; SM920920-4, SM920920-9 (**1,2**), 379-382: Kantori-saki, Taiji, Wakayama Pref. (和歌山県太地町樋取崎) ; SM920920-19, SM920920-20, SM920920-21, SM920920-24 (**1,2**), 383-385: Choshi-zaki, Todo-hokke, Oshima, Hokkaido (北海道渡島支庁樺法華村跳子崎) ; SM921022-1, SM92102-2, SM921022-3 (**1,2,6,7**), 386: Motomura, Todo-hokke, Oshima, Hokkaido (北海道渡島支庁樺法華村元村) , SM921022-6 (**1,2**), 387-389: Esan-misaki, Todo-hokke, Oshima, Hokkaido (北海道渡島支庁樺法華村恵山岬) ; SM921022-14, SM921022-19, SM921022-21 (**1,2**), 390, 391, 393: Tachimachi-saki, Hakodate, Hokkaido (北海道函館市立待岬) ; SM921023-4, SM921023-5, SM94082 (**1,2,6,7**), 392: Hoyaishi, Okushiri Is., Hokkaido (北海道奥尻島ホヤ石) ; SM921025-1 (**1,2,6,7**), 394, 395: Senda-gawa, Choshi, Chiba Pref. (千葉県跳子市千駄ヶ岩) ; SM951020-1, SM951020-2 (**1,2**), 396-400: Fukaura, Aomori Pref. (青森県深浦町) ; SM991008-1, SM991008-2, SM991008-3, SM991008-4, SM991008-5 (**1,2,6,7**), 401-404, 407, 408, 410: Borawazawa-kaigan, Hachijo (Hachijo Is.), Tokyo (東京都八丈町洞輪沢海岸) (八丈島) ; SM890127-1, SM890127-4, SM890127-5, SM890127-18, SM890127-7, SM890127-10, SM890127-18 (**1,2,4,5,6,7,8,9**), 405: Nakanogo, Hachijo (Hachijo Is.), Tokyo (東京都八丈町中之郷) (八丈島) ; SM890128-16 (**1,2,6,7,8,9**), 406, 409: Kashitate, Hachijo (Hachijo Is.), Tokyo (東京都八丈町樺立) (八丈島) ; SM890129-3, SM890127-16 (**1,2,6,7,8,9**), 411: Kashitate, Hachijo (Hachijo Is.), Tokyo (東京都八丈町樺立) (八丈島) ; SM890129-2 (**1,2,4,5,6,7,8,9**).

### *Cyrtomium devexiscapulae*

333: Wulu, Taitung Prov., Taiwan (南部横貫公路霧鹿, 台湾) ; Matsumoto84 (**11,12**), 334, 335: Xhiaoguihu, Taitung Prov., Taiwan (小鬼湖・台東, 台湾) ; Matsumoto127, Matsumoto129 (**11,12**), 412, 440, 441: Cheju Is., Korea (濟州島, 大韓民国) ; TBG123105, TBG123105, TBG123106 (**10,11,12,13,14**), 413: Nagate, Fukue (Fukue Is.), Nagasaki Pref. (長崎県福江市長手) (福江島) ; SM940323-14-3 (**11,12,13,14**), 414: Sotoura, Shimoda, Shizuoka Pref. (静岡県下田市外浦) ; SM810425 (**11,12**), 415, 416: Kisami, Shimoda, Shizuoka Pref. (静岡県下田市吉佐美) ; SM820315-27 (**11,12,13,14**), 417-422: Oura, Shimoda, Shizuoka Pref. (静岡県下田市大浦) ; SM820314-12, SM820314-13, SM820314-15, SM820314-18, SM820314-19, SM820314-25 (**3,10,11,12,13,14**), 423: Onabe, Kawazu, Shizuoka Pref. (静岡県河津町大鍋) ; SM820317-4 (**11,12,13,14**), 424: Shikine, Shimoda, Shizuoka Pref. (静岡県下田市敷根) (**11,12,13,14**), 425: Kakizaki, Shimoda, Shizuoka Pref. (静岡県下田市柿崎) (**11,12,13,14**), 426, 443: Ikeshiro - Mt. Tyokuro, Matsuzaki, Shizuoka Pref. (静岡県松崎町池代一長九郎山) ; TBG74657, SM990419-15 (**11,12,13,14**), 427: Kosedo, Nagasaki, Nagasaki Pref. (長崎県長崎市小瀬戸) (**11,12,13,14**), 428: Kabashima, Nomosaki, Nagasaki Pref (長崎県野母崎町樺島) (**11,12,13,14**), 429: Sekibe, Shizuoka, Shizuoka Pref. (静岡県静岡市石部) ; SM730902-2 (**11,12,13,14**), 430: Ujina Is., Hiroshima, Hiroshima Pref. (広島県広島市宇品島) ; SM900107 (**11,12,13,14**), 431, 432: Ueno park, Taito-ku, Tokyo (東京都台東区上野公園) ;

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SM921001-2, SM921001-3 (**11,12,13,14**), 433, 434: Onagara limestone cave, Honjo, Oita Pref. (大分県本匠村小半鍾乳洞) ; SM940215-23, SM940215-25 (**11,12,13,14**), 435, 436: Kami-osawaguchi, Shimoda, Shizuoka Pref. (静岡県下田市上大沢口) ; SM950604-1, SM950604-15 (**11,12,13,14**), 437: Fukue Is., Nagasaki Pref. (長崎県福江島) ; SM941207-114 (**3,10,11,12,13,14**), 438: Chikushi-yabakei, Nakagawa, Fukuoka Pref. (福岡県那珂川町筑紫耶馬溪) ; SM961013-13 (**11,12,13,14**), 439: Jochu, Fukuoka, Fukuoka Pref. (福岡県福岡市城中) ; SM961013-15 (**11,12,13,14**), 442: Jinggang-shan, Jiangxi Prov., China (江西省井岡山, 中華人民共和国) ; SM1101 (**11,12,13,14**), 444-446: Hou-ji temple, Putuo-shan, Zhoushan Qundao Isls., Zhejiang Prov., China (浙江省舟山群島普陀山法雨寺一仏頂山, 中華人民共和国) ; S. Matsumoto & J.-H. Gu3, S. Matsumoto & J.-H. Gu12, S. Matsumoto & J.-H. Gu15 (**10,11,12,13,14**), 447: Xiaogui-hu, Taitung Prov., Taiwan (蘭嶼・台東, 台湾) ; Matsumoto 00-129 (**11,12,13,14**), 448-451: Seto, Kanazawa-ku, Yokohama, Kanagawa Pref. (神奈川県横浜市金沢区瀬戸) ; SM0005-01, SM0005-02, SM0005-11, SM0005-17 (**11,12,13,14**), 452, 453: Kawayo, Choyo, Kumamoto Pref. (熊本県長陽村河陽) ; SM01-6, SM01-8 (**11,12,13,14**).

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\* Plant materials, of which the country names are not recorded, were collected in Japan.