

## PHYTOCHEMICAL REPORTS

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### PHLOROGLUCINOL DERIVATIVES OF *DRYOPTERIS SIEBOLDII*

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(Received 11 April 1973 Accepted 24 April 1973)

**Key Word Index**—*Dryopteris sieboldii*, Aspidiaceae, fern, acylphloroglucinol, filixic acid-*ABB*

In previous paper,<sup>1</sup> we reported the isolations of filixic acid-*PBP*, flavaspidic acid-*AB* and -*PB* from the rhizomes of *Dryopteris sieboldii*. From the same material we have also isolated another acylphloroglucinol, filixic acid-*ABB*.

The Et<sub>2</sub>O solution of crude filicin after removal of flavaspidic acid-*AB* was chromatographed on silica and the elution of cyclohexane-CHCl<sub>3</sub> (1:1) gave filixic acid-*ABB*.

Filixic acid-*ABB* C<sub>30</sub>H<sub>40</sub>O<sub>12</sub>, m.p. 175–177° (needle cryst. from acetone) IR  $\nu_{\max}^{\text{KBr}}$  cm<sup>-1</sup>: 3150 (OH), 2950 (methylene), 1640–1610 (enolic 1,3-diketo system or 2-hydroxyarylketone), 1430, 1193. UV  $\lambda_{\max}^{\text{EtOH}}$  nm (log  $\epsilon$ ): 224 (4.43), 299 (4.28), 345 (4.28). UV  $\lambda_{\max}^{\text{EtOH} + \text{NaOH}}$  nm (log  $\epsilon$ ): 241 (4.37), 314 (4.33). *R<sub>f</sub>* 0.62 on TLC in CHCl<sub>3</sub>-MeOH-H<sub>2</sub>O (7:3:1, lower) spot color gave orange yellow with diazotized benzidine. The NMR spectrum (100 MHz, CDCl<sub>3</sub>) gave bands at ( $\delta$ ): 1.00 (3H, *t*-COCH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>, *J* 7.5 Hz), 1.17 (3H, *t*-COCH<sub>2</sub>-CH<sub>2</sub>CH<sub>3</sub>, *J* 7.5 Hz), 1.43, 1.54 (12H, each *s* two *gem*-dimethyl groups), *ca* 1.76 (4H, *m* two -COCH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 2.72 (3H, *s*-COCH<sub>3</sub>), 3.20 (4H, overlapping *t* two -COCH<sub>2</sub>CH<sub>2</sub>-CH<sub>3</sub>), 4.02, 4.06 (4H, each *s* two  $\geq\text{C}-\text{CH}_2-\text{C}\leq$  groups). The signals at 10.16 (4H, *br*), 13.05 (1H, *br*) and 16.24 (2H, *br*) are all due to hydrogen bonded OH-groups, and quenched by addition of D<sub>2</sub>O. The MS shows the parent peak at *m/e* 640. Other important peaks at *m/e* 432, 404, 236, 193, 181, 165.

<sup>1</sup> HISADA, S., INOUE, O. and INAGAKI, I. (1973) *Phytochemistry* 12, 1493.

**Acknowledgements**—The authors thank Professor H. Itokawa, Tokyo College of Pharmacy for measurement of NMR and MS. We are also indebted to Analytical Center of our University for elemental analysis.