

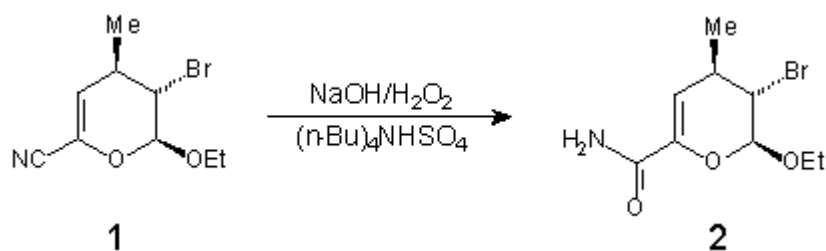
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## Synthesis of 3-Bromo-2-ethoxy-4-methyl-3,4-dihydro-2H-pyran-6-amide

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### Scheme

The amide **2** [1] was prepared by hydrolysis of the nitrile **1** according to the reported procedure [2].

To a cooled (0[infinity]C) solution of **1** (0.68 g, 2.76 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (2 ml) was added successively hydrogen peroxide (30%, 1.29 ml), tetra-(n-butyl)ammonium hydrogen sulfate (187 mg) and an aqueous solution of sodium hydroxide (20%, 1.03 ml). The reaction mixture was allowed to warm to room temperature. Methylenechloride (30 ml) was added after 1 h. The organic layer was separated, washed with brine, dried (Na<sub>2</sub>SO<sub>4</sub>) and concentrated in vacuo. The residue (0.25 g) was flash chromatographed (AcOEt/Hexane = 3:1) to afford 370 mg (70%) of **2**.

R<sub>f</sub> (AcOEt): 0.34.

M. p. 130-131.5 deg.C.

IR (KBr): 3400vs, 3180s, 1668s, 1630vs, 1598s, 1415s, 963s, 912m, 820m.

<sup>1</sup>H-NMR (CDCl<sub>3</sub>): 6.38 (br, 1H, NH); 5.89 (dd, J = 3.0, 0.5, 1H, H-5); 5.69 (br, 1H, NH); 5.05 (dd, J = 1.3, 1.0, 1H, H-2), 4.28 (ddd, J = 5.0, 1.3, 0.5, 1H, H-3); 3.98 and 3.71 (2x dq, J = 9.7, 7.0, OCH<sub>2</sub>Me), 2.89 (qddd, J = 7.0, 5.0, 3.0, 1.0, 1H, H-4); 1.31 (t, J = 7.0, 3H, OCH<sub>2</sub>CH<sub>3</sub>); 1.27 (d, J = 7.0, 3H, Me).

<sup>13</sup>C-NMR (CDCl<sub>3</sub>): 164.0 (CONH<sub>2</sub>), 141.7 (C-6), 110.7 (C-5), 99.5 (C-2), 65.5 (OCH<sub>2</sub>Me), 52.1 (C-3), 32.2 (C-4), 17.8 (Me), 14.9 (OCH<sub>2</sub>CH<sub>3</sub>).

CI-MS: 283/281 (M+NH<sub>4</sub><sup>+</sup>, 69/76), 267/265 (M+2, 12/13), 266/264 (M+H<sup>+</sup>, 100/100), 184 (M-Br, 68), 152/150 (24/23), 138 (100), 124 (12), 122 (9), 111 (189, 99 (19), 88 (26), 83 (15), 77 (27).

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### References

1. Zhuo, J.-C.; Wyler, H. *Helv. Chim. Acta* **1995**, *78*, 151.
2. Cacchi, S.; Misiti, D.; La Torre, F. *Synthesis* **1980**, 243.

*Sample Availability:* Available from MDPI, 0.1g, MDPI 12540.

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