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2-Bromo-4-methoxy-5-(1-methylethoxy)benzeneacetic Acid

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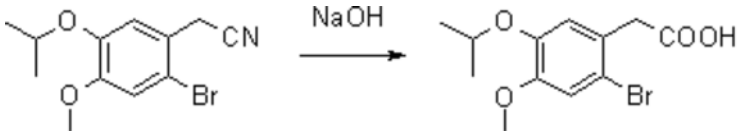

2-Bromo-4-methoxy-5-(1-methylethoxy)benzeneacetonitrile [1] (5.86 g, 20.6 mmol) was refluxed in 25% NaOH (50 mL)/EtOH (50 mL) for 12 h. The mixture was concentrated to a volume of 50 mL, the residue was washed with Et_2O (2 x 10 mL), and the pH was adjusted to 1 using conc. HCl. The aqueous layer was extracted with Et_2O (3 x 50 mL), and the combined organic layer was washed with water (5 x 100 mL) and brine (100 mL), dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was triturated with iPr_2O (2 x 50 mL). Yield: colorless crystals (5.59 g, 90%), mp. 128 - 132 °C.

TLC: petroleum ether : EtOAc = 80 : 30, R_f = 0.1.

Anal. Calcd for $C_{12}H_{15}BrO_4$: C, 47.54; H, 4.99. Found: C, 47.76; H, 4.93.

^{1}H NMR ($CDCl_3$): d 10.10 (b, 1H), 7.02 (s, 1H), 6.81 (s, 1H), 4.47 (septet, J = 6.4 Hz, 1H), 3.82 (s, 3H), 3.71 (s, 2H), 1.32 (d, J = 6.4 Hz, 6H).

^{13}C NMR ($CDCl_3$): d 176.9 (s), 150.3 (s), 146.5 (s), 125.1 (s), 118.4 (d), 116.0 (d), 115.3 (s), 71.8 (d), 56.1 (q), 40.7 (t), 21.9 (q).

References and Notes

1. Treu, M.; Mereiter, K.; Hametner, Ch.; Fröhlich, J.; Jordis, U. Carboxylic Galanthamine Analogs II: 4a,5,9,10,11,12-Hexahydro-6H-benzo[a]cyclohepta[hi]benzofuran. Submitted to *J. Heterocyclic Chem.*

Samples Availability: Available from the authors.