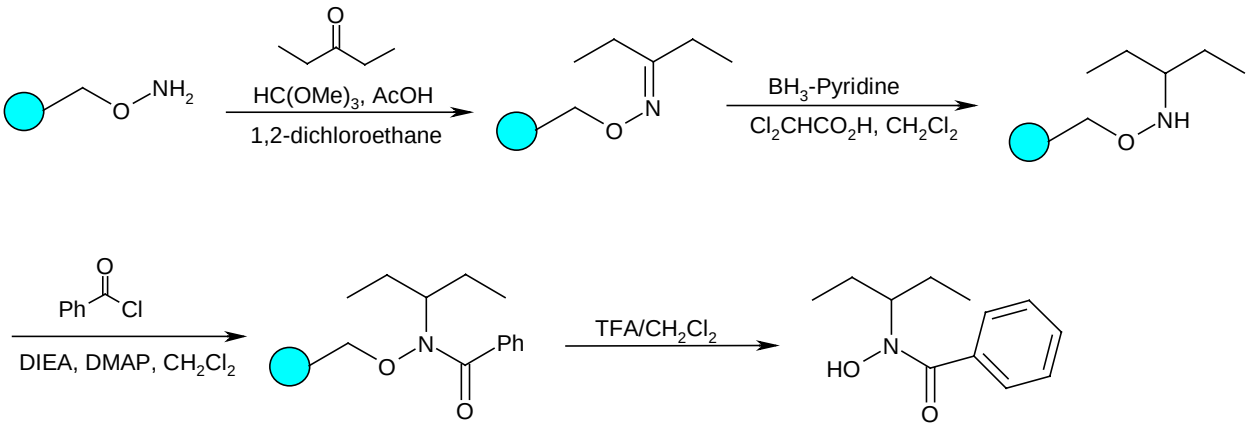
Supporting information for Robinson and Holladay, "Convenient Preparation of *O*-Linked Polymer-Bound *N*-Substituted Hydroxylamines, Intermediates for Synthesis of *N*-Substituted Hydroxamic Acids"

Preparation of N-(3-pentyl)-benzohydroxamic acid



Abbreviations: DCE, 1,2-dichloroethane; DCM, dichloromethane; others in accordance with *J. Org. Chem.*, **1996**, 61, 22A.

A 50-mL fritted polypropylene syringe was charged with O-linked resin-bound hydroxylamine (1.02 g, ~1.4 mmol) prepared from commercial[1] Wang resin as described by Floyd, et al.[2] A solution of 3-pentanone (1.5 mL, 14 mmol), acetic acid (0.28 mL, 4.9 mmol) and trimethyl orthoformate (12 mL) in DCE (16 mL) was introduced, and the mixture was agitated for 18 h. The solution was drained, and the procedure was repeated for an additional 8 h. After the solution was drained, the resin was washed with DCE (2X), DMF (5X) and DCM (4X).

The resin from above was transferred to a 200 mL polypropylene container, and treated with BH_3-pyridine (2.6 mL of 8 M solution, 21.2 mmol) and DCM (30 mL). The mixture was cooled to 0 ^{o}C and dichloroacetic acid (2.6 mL, 31 mmol) was added dropwise. The vessel was capped and the mixture was shaken for 18 h, with occasional venting during the first hour. The resin was then transferred to a 50-mL fritted syringe and washed with DCM (2X), MeOH (1X), shaken with MeOH for 30min followed by draining (1X), MeOH (1X), DMF (4X) and DCM (5X).

The above resin in a 50-mL fritted syringe was treated with a solution of benzoyl chloride (2.4 mL, 14 mmol), DIEA (2.7 mL, 15.5 mmol) and DMAP (85 mg, 0.7 mmol) in DCM (28 mL). The mixture was shaken for 18 h, then the resin was drained, washed with DCM (4X), DMF (4X) and DCM (4X) and dried in a vacuum desiccator overnight.

To half of the above resin in a 20 mL glass vial was added a 1:1 solution of TFA/ CH_2Cl_2 (15 mL). The mixture was agitated for 1.5 h, then the solvent was evaporated under a stream of nitrogen, and the resin was extracted twice with CH_2Cl_2. Evaporation of the solvent afforded 118 mg of a light orange syrup (83% pure by HPLC, monitored at 215 nm). A 41 mg sample was chromatographed on silica gel (hexane/EtOAc/HOAc, 50:25:4) to afford 30 mg (60% yield) of a light orange oil, which crystallized on standing.

^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, 6H), 1.53 (m, 2H), 1.87 (m, 2H), 3.65 (m, 1H), 7.43-7.51 (m, 5H). High resolution MS (FAB^+): Calcd. for $C_{12}H_{18}NO_2$ (M + H), 208.1338; found, 208.1337.

1) Wang resin with nominal loading of 1.4 mmol/g was purchased from Advanced Chemtech.
2) Floyd, C. D.; Lewis, C. N.; Patel, S. R.; Whittaker, M. *Tetrahedron Lett*. **1996**, *37*, 8045-8048.