[^{15}N] Diisopropyl Amine. A nitrogen flushed 250 ml 24/40 RB flask equipped with a septum and stir bar was charged sequentially with $^{15}NH_4Cl$ (3.0 g, 55 mmol), $NaCNBH_3$ (6.9 g 110 mmol), NaOAc (6.6 g, 80.5 mmol), and powered 4 Å molecular sieves. After the flask was cooled to 0 °C, MeOH (150 ml) was added followed by HOAc (0.44 ml, 7.7 mmol) and acetone (12.2 ml, 165 mmol) with rapid stirring. The reaction was allowed to warm up gradually over 17 hours. The suspension was cooled to 0 °C and the pH was adjusted to 12 with solid NaOH pellets. The suspension was warmed to RT and the liquids were vacuumed transferred. The solution was warmed to 0 °C and transferred to a 24/40, 250 ml, RB flask. Gaseous HCl was bubbled through the solution at 0 °C until the pH reached 0 (see setup below and following description). Concentrated H_2SO_4 is slowly added with a dropping funnel to a three-neck 250 ml RB containing ~10g NaCl and ~30 ml concentrated HCl. The gas is directed out the side-arm, through a bubbler filled with concentrated H_2SO_4. In case of back suction, the assembly includes a trap. Ultimately, the gaseous HCl is passed into the amine solution through a glass pasteur pipette. Check the pH periodically and be cautious not to sample the highly acidic gas environment of the flask.

Experimental setup for gaseous HCl formation.

The solution was concentrated via roto evaporation and the slurry was recrystallized from 1:1 THF/*i*-PrOH. A second crop was attainted by concentrating the mother liquor. The combined crops were dried overnight by vacuum in a 14/20 250 ml RB flask that was attached to a 90 ° vacuum line adapter and was equipped with a stir bar. 4.92 g (65% yield) of pure material was obtained. ^{1}H NMR (D_2O) δ 3.37 (7 line m, 1 H, J = 6.4 Hz), 1.18 (dd, 6 H, $J_{H\text{-}H}$ = 6.5 Hz, $J_{N\text{-}H}$ = 4.4 Hz); 13C{^{1}H} NMR (D_2O) δ 40.0 (CH, $^{1}J_{N\text{-}C}$ < 1 Hz), 10.0 (CH_3, $^{2}J_{N\text{-}C}$ < 1 Hz).