Supporting information for

A Total Synthesis of Hydroxylysine in Protected Form and Investigation f the Reductive Opening of *p*-Methoxybenzylidene Acetals

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- S2: General Methods and Materials
- S3: ¹H NMR for compound **5**
- S4: ¹³C NMR for compound **5**
- S5: ¹H NMR for compound **6**
- S6: ¹³C NMR for compound **6**
- S7: ¹H NMR for compound **7b**
- S8: ¹³C NMR for compound **7b**
- S9: ¹H NMR for compound **10**
- S10: ¹³C NMR for compound **10**
- S11: ¹H NMR for compound **11**
- S12: ¹H NMR for compound **12**
- S13, S14: ¹⁵N NMR for compound **6**
- S15, S16: ¹⁵N NMR for compound **6** + TBDMSC1
- S17: HPLC chromatograms for diastereomeric separations for compound 11
- S18: HPLC chromatograms for compound 13

General Methods and Materials. All reactions were carried out under an inert atmosphere with dry solvents under anhydrous conditions, unless otherwise stated. CH_2Cl_2 was distilled from calcium hydride, whereas THF and toluene were distilled from potassium benzophenone and sodium, respectively. Methanol was dried over 3 Å molecular sieves. TLC was performed on Silica gel 60 F_{254} with detection by UV light and staining with a solution of ethanolic phosphomolybdic acid. Flash column chromatography (eluents given in brackets) was performed on silica gel.

¹H and ¹³C NMR spectra for compounds **5-7b** and **10** were recorded at 400 and 100 MHz, respectively, for solutions in CDCl₃ [residual CHCl₃ (δ_{H} 7.26 ppm) or CDCl₃ (δ_{C} 77.0 ppm), as internal standard] at 298 K. ¹H NMR spectra for compounds **11** and **12** were recorded at 400 MHz for solutions in DMSO-d₆ [residual DMSO-d₅ (δ_{H} 2.50ppm) as internal standard] at 368 K (To equilibrate morpholine conformers). Due to decomposition (cleavage of the TBDMS group) upon prolonged heating at 268 K, ¹³C NMR spectra could not be recorded for compounds **11** and **12**. ¹⁵N NMR spectra were recorded at 40.49 MHz for solutions in CD₃CN (the most downfield signal was arbitrarily set to 0 ppm). First-order chemical shifts and coupling constants were obtained from one-dimensional spectra; carbon and proton resonances were assigned from COSY and HETCOR experiments. Optical rotations were measured at 20 °C. Determinations of diastereomeric excesses were performed with HPLC on a reverse-phase column (water:acetonitrile gradient) and by using ¹H NMR spectroscopy.



























¹⁵N NMR



¹⁵N NMR (one of two sets of signals shown)



¹⁵N NMR



¹⁵N NMR (one of two sets of signals shown)





S18