Diastereoselective Synthesis of α -Methyl and α -Hydroxy- β -Amino Acids via 4-Substituted-1,3-Oxazinan-6-ones

Brad E. Sleebs^{a,b} and Andrew B. Hughes^{*a}

^aDepartment of Chemistry, La Trobe University, Bundoora, Victoria 3086, Australia.

a.hughes@latrobe.edu.au

^bCurrent Address: The Walter and Eliza Hall Institute of Medical Research, Structural Biology Division, Medicinal Chemistry Group, 4 Research Avenue, La Trobe University Research and Development Park, Victoria 3086, Australia.

Supporting Information

Contents

General Experimental S2

Compound Number	Page	¹ H NMR Spectrum	¹³ C NMR Spectrum
8	S3	√	√
10	S4	√	√
11	S5	√	√
12	S6	√	√
13	S7	√	√
14	S8	√	√
15	S9	√	√
16	S10	√	√
17	S11	√	√
18	S12	√	√
19	S13	√	√
20	S14	√	√
25	S15	√	√
27	S16	√	√
28	S17	√	√
30	S18	√	√
31	S19	√	√
32	S20	√	√
33	S21	√	√
34	S22	√	√
37	S23	√	√
38	S24	√	√
39	S25	√	√
40	S26	√	√
41	S27	√	√
42	S28	√	√
44	S29	√	√
49	S30	√	√

General Methods

Infrared spectra were recorded on a Fourier transform IR spectrometer. NMR was performed in (D)-chloroform at 300 K unless otherwise stated on a 300 MHz spectrometer. Low and high resolution mass spectra (LSIMS) were also measured at at 70 eV; all spectra were acquired using water/methanol/acetic acid (0:99:1 or 50:50:1) mixtures as the mobile phase. Flash column chromatography was carried out using silica gel 60 particle size 0.040-0.063 μm (230-400 mesh ASTM). Amide bases were titrated before use. Ethyl acetate and hexane used for chromatography were distilled prior to use. TLC was performed on kieselgel 60 F₂₅₄ plates and visualised with a UV lamp. For dry solvents, procedures from Perrin and Armarego* were followed. All other reagents and solvents were purified or dried as described by Perrin and Armarego.*

*Perrin, D. D.; Armarego, W. L. F., Purification Of Laboratory Chemicals; 3rd ed.; Pergamon: Oxford, 1988.

(R)-N-benzyl-4-methyl-1,3-oxazinan-2-one 8

(R)-N-benzyl-4-methyl-1,3-oxazinan-2-one 8

(4S,5S)-N-Benzylsuccinimide-4,5-dimethyl-1,3-oxazinan-6-one 10

(4R,5R)-N-Benzylsuccinimide-4,5-dimethyl-1,3-oxazinan-6-one 10

(4S)-N-benzoyloxycarbonyl-4,5,5-trimethyl-1,3-oxazolin-6-one 12

(4S)-N-benzoyloxycarbonyl-4,5,5-trimethyl-1,3-oxazolin-6-one 12

(4R,5R)-4-Benzyloxycarbonyl-4,5-dimethyl-1,3-oxazinan-6-one 20

7.327

5.843
5.808

5.162
5.133
5.105
5.000

3.773
3.754

2.524
2.502
2.480
2.467
2.446
2.424

1.358
1.337
1.275
1.253

(4R,5R)-4-Benzyloxycarbonyl-4,5-dimethyl-1,3-oxazinan-6-one 20

172.251

153.951

135.681

128.561

127.881

126.051

70.837

68.025

53.246

41.403

19.369

13.216

(3S)-[1-[(benzyloxy)carbonyl]-3-amino-butanoate 4f

(2S)-[1-[(benzyloxy)carbonyl]-3-amino-butanoate 4g