Supporting Information

for

Solution-phase automated synthesis of an
α-amino aldehyde as a versatile intermediate

Hisashi Masui¹, Sae Yosugi¹, Shinichiro Fuse² and Takashi Takahashi*¹

Address: ¹Yokohama University of Pharmacy, 601 Matano-cho, Totsuka-ku,
Yokohama 245-0066, Japan, and ²Laboratory for Chemistry and Life Science,
Institute of Innovative Research, Tokyo Institute of Technology, 4259
Nagatsuta-cho, Midori-ku, Yokohama 226-8503, Japan

Email: Takashi Takahashi - ttak@hamayaku.ac.jp

* Corresponding author

Synthetic procedures and ¹H NMR spectral data of compounds
2a–c, 3a–c, and 4a–c
Experimental

General

NMR spectra were recorded on a JEOL Model ECA-500 instrument. Chemical shifts are reported in parts per million (ppm) relative to the signal for the internal standard tetramethylsilane (0.0 ppm) or the solvent CDCl₃ (7.26 ppm, ¹H NMR; or 77.1 ppm, ¹³C NMR) peaks. Data for ¹H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), and integration. ¹³C NMR spectrum data are reported as follows: chemical shift (δ ppm), and where applicable, multiplicity and coupling constants. Multiplicities are reported using the following abbreviations: s, singlet; d, doublet; t, triplet; q, quartet; sp, septet; m, multiplet; br, broad; and J, coupling constants in hertz. Only the strongest and/or structurally relevant IR peaks are reported (cm⁻¹). All reactions were monitored by thin-layer chromatography performed using 0.2 mm E. Merck silica gel plate (60F-254). The reactants and products were visualized using UV light (254 nm), or by heating after treatment with p-anisaldehyde solution, ceric sulfate solution, ninhydrin solution, or 10% ethanoic phosphomolybdic acid. Column chromatography separations were performed using silica gel (Merck).

Methyl (tert-butoxycarbonyl)-L-serinate (2a)

\[
\begin{align*}
\text{HO} & \quad \text{NH₂HCl} \\
\text{C} & \quad \text{O} \\
\text{Me} & \quad \text{Me} \\
\text{NH₂HCl} & \quad \text{C} \\
\text{Boc₂O, NEt₃} & \quad \text{THF} \\
\text{r.t., 5 h, 82%} & \quad \text{NHBoc}
\end{align*}
\]
The reagents, washings and solvents were placed in each reservoirs and bottles as described below.

**RF1**  methyl L-serinate hydrochloride (2.00 g, 12.9 mmol)

**RR1**  Boc$_2$O (3.38 g, 15.5 mmol) in 11.0 mL of THF

**RR2**  THF (50.0 mL)
To a solution of methyl L-serinate hydrochloride (1, 2.00 g, 12.9 mmol, 1.00 equiv., RF1) in THF (50.0 mL, RR2) were added Et$_3$N (5.36 mL, 38.7 mmol, 3.00 equiv, RR3) and Boc$_2$O (3.38 g, 15.5 mmol, 1.20 equiv, RR1) in THF (11.0 mL, RR1) diluted with THF (5.00 mL, RR4) and transferred to RF1 at 25 °C. After being stirred at the same temperature for 5 h, the reaction mixture (RF1) was quenched with 1 M HCl aq. (RS2) and the aqueous layer was extracted with two portions of ethyl acetate (RS1). The combined organic layer was washed with 10% NaCl aq. (RS3), dried over Na$_2$SO$_4$ (DT1), filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (50% ethyl acetate in hexane) to give methyl (tert-butoxycarbonyl)-L-serinate (2.31 g, 10.5 mmol, 82%) as a colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) δ 5.43 (s, 1H), 4.39 (s, 1H), 3.98 (dd, $J = 4.0$ Hz, 11.5 Hz, 1H), 3.93 (dd, $J = 3.5$ Hz, 11.5 Hz, 1H), 3.79 (s, 3H), 1.45 (s, 9H).
3-(tert-Butyl) 4-methyl (S)-2,2-dimethyloxazolidine-3,4-dicarboxylate (3a)

![Chemical Structure]

**Procedure:**
- **START**
- **STIR-RF1**
- **RR1-RF1**
- **RR4-RR1-RF1**
- **RR3-RF1**
- **RR6-RR3-RF1**
- **WAIT(Min) (Wait 360 min)**
- **STIROFF-RF1**
- **RF1-SF**
- **SF-SF2**
- **RR2-RF1**
- **SF2-RF1**
- **STIR-RF1**
- **WAIT(Min) (Wait 5 min)**
- **STIROFF-RF1**
- **RS1-RF1**
- **SEP-RF1(UP)**
- **SF1-RF1**
- **RS1-RF1**
- **SEP-RF1(UP)**
The reagents, washings and solvents were placed in each reservoirs and bottles as described below.

RF1 methyl (tert-butoxycarbonyl)-L-serinate (2a, 1.94 g, 8.85 mmol) in 20.0 mL of CH₂Cl₂
RR1 2,2-dimethoxypropane (3.25 mL, 26.6 mmol) in 15.0 mL of CH₂Cl₂
RR2 NaOH (10% aqueous) 60.0 mL
RR3 BF₃·OEt₂ (0.0556 mL, 0.443 mmol) in 15.0 mL of CH₂Cl₂
RR4 CH₂Cl₂ 10.0 mL
RR6 CH₂Cl₂ 10.0 mL
RS1 EtOAc 160 mL
RS3 NaCl (10% aqueous) 80.0 mL
DT2 Na₂SO₄

To a solution of methyl (tert-butoxycarbonyl)-L-serinate (2a, 1.94 g, 8.85 mmol, 1.00 equiv, RF1) in CH₂Cl₂ (20.0 mL, RF1) were added 2,2-dimethoxypropane (3.25 mL, 26.6 mmol, 3.00 equiv, RR1) in CH₂Cl₂ (15.0 mL, RR1) diluted with CH₂Cl₂ (10.0 mL, RR4) and boron trifluoride-ethyl ether complex (0.0553 mL, 0.443 mmol, 0.500 equiv, RR3) in CH₂Cl₂ (15.0 mL, RR3) diluted with CH₂Cl₂ (10.0 mL, RR6) and transferred to RF1 at 25 °C. After being stirred at the same
temperature for 3 h, the reaction mixture (RF1) was quenched with saturated NaOH aq. (RR2) and the aqueous layer was extracted with two portions of ethyl acetate (RS1). The combined organic layer was washed with 10% NaCl aq. (RS3), dried over Na$_2$SO$_4$ (DT2), filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (16% ethyl acetate in hexane) to give 3-(tert-butyl) 4-methyl (S)-2,2-dimethylxazolidine-3,4-dicarboxylate (2.28 g, 8.79 mmol, 99%) as a colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) δ 4.49 (dd, $J = 2.5$ Hz, 7.0 Hz, 1H), 4.38 (dd, $J = 3.0$ Hz, 7.0 Hz, 1H), 4.16-4.12 (m, 1H), 4.04-4.02 (m, 1H), 3.75 (s, 3H), 1.67-1.41 (m, 15H).

**tert-Butyl (R)-4-(hydroxymethyl)-2,2-dimethylxazolidine-3-carboxylate (4a)**

![Chemical structure of 4a]
The reagents, washings and solvents were placed in each reservoirs and bottles as described below.

RF1 3-(tert-butyl) 4-methyl (S)-2,2-dimethyloxazolidine-3,4-dicarboxylate (1.00 g, 3.86 mmol, 1.00 equiv, RF1) in 50.0 mL of toluene

RR2 Diisobutylaluminum Hydride (7.72 mL, 7.72 mmol, 2.00 equiv)

RS1 EtOAc 160 mL

RS2 Rochelle salt (10% aqueous) 60.0 mL

RS3 NaCl (10% aqueous) 80.0 mL

DT1 Na₂SO₄

To a solution of 3-(tert-butyl) 4-methyl (S)-2,2-dimethyloxazolidine-3,4-dicarboxylate (1.00 g, 3.86 mmol, 1.00 equiv, RF1) in toluene (50.0 mL, RF1) were added DIBAL (7.72 mL, 7.72 mmol, 2.00 equiv, RR2) and transferred to RF1 at −80 °C. After being stirred at the same temperature for 4 h, the reaction mixture was quenched with saturated aqueous Rochelle salt and the aqueous
layer (RS2) at 25 °C. After being stirred at the same temperature for 1 h, the reaction mixture was extracted with two portions of ethyl acetate (RS1). The combined organic layer was washed with 10% NaCl aq. (RS3), dried over Na₂SO₄ (DT1), filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (25% ethyl acetate in hexane) to give tert-butyl (R)-4-(hydroxymethyl)-2,2-dimethyloxazolidine-3-carboxylate (0.603 g, 2.63 mmol, 71%) as a colorless oil.

¹H NMR (500 MHz, CDCl₃) δ 9.61-9.54 (m, 1H), 4.35-4.18 (m, 1H), 4.12-4.02 (m, 2H), 1.65-1.41 (m, 15H).

Methyl (((9H-fluoren-9-yl)methoxy)carbonyl)-L-serinate (2b)

![Chemical structure of methyl (((9H-fluoren-9-yl)methoxy)carbonyl)-L-serinate (2b)]

START

RR2-RF1

STIR-RF1

RR3-RF1

RR1-RF1

RR4-RR1-RF1

WAIT(Min) (Wait 300 min)

RS2-RF1

WAIT(Min) (Wait 3 min)

STIROFF-RF1
RS1-RF1
SEP-RF1 (UP)
SF1-RF1
RS1-RF1
SEP-RF1(UP)
SF2-SF
RS3-RF1
SEP-RF1(UP)
SF2-DT2-CF1
END

The reagents, washings and solvents were placed in each reservoirs and bottles as described below.

RF1 methyl L-serinate hydrochloride (1.00 g, 6.31 mmol)
RR1 Fmoc-OSu (2.55 g, 7.57 mmol) in 15.0 mL of dioxane
RR2 dioxane (5.00 mL)
RR3 NaHCO₃ (1.59 g, 18.9 mmol) in 25.2 mL of H₂O
RR4 dioxane 4.20 mL
RS1 EtOAc 120 mL
RS2 HCl (1 M aqueous) 60.0 mL
RS3 NaCl (10% aqueous) 80.0 mL
DT2 Na₂SO₄

To a solution of methyl L-serinate hydrochloride (1.00 g, 6.31 mmol, 1.00 equiv., RF1) in dioxane (5.00 mL, RR2) were added NaHCO₃ (1.59 g, 18.9 mmol, 3.00
equiv, RR3) in H₂O (25.2 mL, RR3) and Fmoc-OSu (2.55 g, 7.57 mmol, 1.20 equiv, RR1) in dioxane (15.0 mL, RR1) diluted with dioxane (4.20 mL, RR4) and transferred to RF1 at 25 °C. After being stirred at the same temperature for 5 h, the reaction mixture (RF1) was quenched with 1 M HCl aq. (RS2) and the aqueous layer was extracted with two portions of ethyl acetate (RS1). The combined organic layer was washed with 10% NaCl aq. (RS3), dried over Na₂SO₄ (DT2), filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (50% ethyl acetate in hexane) to give methyl (((9H-fluoren-9-yl)methoxy)carbonyl)-L-serinate (2.08 g, 6.09 mmol, 97%) as a colorless oil.

¹H NMR (500 MHz, CDCl₃) δ 7.78-7.61 (m, 4H), 7.43-7.31 (m, 4H), 5.67 (d, J = 7.0 Hz, 1H), 4.44-4.43 (m, 3H), 4.24 (t, J = 6.5 Hz, 1H), 4.02-3.94 (m, 2H), 3.80 (s, 3H), 2.05 (t, J = 6.0 Hz, 1H).

3-((9H-Fluoren-9-yl)methyl) 4-methyl (S)-2,2-dimethyloxazolidine-3,4-dicarboxylate (3b)

3-((9H-Fluoren-9-yl)methyl) 4-methyl (S)-2,2-dimethyloxazolidine-3,4-dicarboxylate (3b)

![Chemical structure](image)

START

STIR-RF1

RR1-RF1

RR4-RR1-RF1

RR3-RF1
RR6-RR3-RF1
WAIT(Min) (Wait 360 min)
STIROFF-RF1
RF1-SF
SF-SF2
RR2-RF1
SF2-RF1
STIR-RF1
WAIT(Min) (Wait 5 min)
STIROFF-RF1
RS1-RF1
SEP-RF1(UP)
SF1-RF1
RS1-RF1
SEP-RF1(UP)
SF2-SF
RS3-RF1
SEP-RF1(UP)
SF2-DT2-CF1
END
The reagents, washings and solvents were placed in each reservoirs and bottles as described below.
RF1  methyl (((9H-fluoren-9-yl)methoxy)carbonyl)-L-serinate (1.00 g, 2.93 mmol) in 15.6 mL of CH₂Cl₂
To a solution of methyl (((9H-fluoren-9-yl)methoxy)carbonyl)-L-serinate (2b, 1.00 g, 2.93 mmol, 1.00 equiv, RF1) in CH$_2$Cl$_2$ (15.6 mL, RF1) were added 2,2-dimethoxypropane (1.08 mL, 8.79 mmol, 3.00 equiv, RR1) in CH$_2$Cl$_2$ (5.00 mL, RR1) diluted with CH$_2$Cl$_2$ (5.00 mL, RR4) and boron trifluoride·ethyl ether complex (0.0368 mL, 0.293 mmol, 0.100 equiv., RR3) in CH$_2$Cl$_2$ (15.0 mL, RR3) diluted with CH$_2$Cl$_2$ (5.00 mL, RR6) and transferred to RF1 at 25 °C. After being stirred at the same temperature for 3 h, the reaction mixture (RF1) was quenched with saturated NaOH aq. (RR2) and the aqueous layer was extracted with two portions of ethyl acetate (RS1). The combined organic layer was washed with 10% NaCl aq. (RS3), dried over Na$_2$SO$_4$ (DT2), filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (25% ethyl acetate in hexane) to give 3-((tert-butyl) 3-((9H-fluoren-9-yl)methyl) 4-methyl (S)-2,2-dimethoxazolidine-3,4-dicarboxylate (0.977 g, 2.56 mmol, 87%) as a colorless oil.
$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.74-7.53 (m, 4H), 7.39-7.30 (m, 4H), 4.72 (t, $J$ = 4.5 Hz, 1H), 4.47-4.07 (m, 5H), 3.65 (s, 3H), 1.73 (s, 3H), 1.53 (s, 3H).

(9H-Fuoren-9-yl)methyl (S)-4-formyl-2,2-dimethyloxazolidine-3-carboxylate (4b)

![Diagram](image)

START
STIR-RF1
RR2-RF1
WAIT(Min) (Wait 240 min)
RS2-RF1
RS1-RF1
WAIT(Min) (Wait 60 min)
STIROFF-RF1
SEP-RF1(UP)
SF1-RF1
RS1-RF1
SEP-RF1(UP)
SF2-SF
RS3-RF1
SEP-RF1(UP)
SF2-DT1-CF1
The reagents, washings and solvents were placed in each reservoirs and bottles as described below.

**RF1** 3-((9H-fluoren-9-yl)methyl) 4-methyl (S)-2,2-dimethylazolidine-3,4-dicarboxylate (1.00 g, 2.62 mmol, 1.00 equiv., RF1) in 52.4 mL of toluene

**RR2** Diisobutylaluminum hydride (5.24 mL, 5.24 mmol, 2.00 equiv.)

**RS1** EtOAc 160 mL

**RS2** Rochelle salt (10% aqueous) 60.0 mL

**RS3** NaCl (10% aqueous) 80.0 mL

**DT1** Na$_2$SO$_4$

To a solution of 3-((9H-fluoren-9-yl)methyl) 4-methyl (S)-2,2-dimethylazolidine-3,4-dicarboxylate (3b, 1.00 g, 2.62 mmol, 1.00 equiv, RF1) in toluene (52.4 mL, RF1) were added DIBAL (5.24 mL, 5.24 mmol, 2.00 equiv, RR2) and transferred to RF1 at −80 °C. After being stirred at the same temperature for 4 h, the reaction mixture was quenched with saturated aqueous Rochelle salt and the aqueous layer (RS2) at 25 °C. After being stirred at the same temperature for 1 h, the reaction mixture was extracted with two portions of ethyl acetate (RS1). The combined organic layer was washed with 10% NaCl aq. (RS3), dried over Na$_2$SO$_4$ (DT1), filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (25% ethyl acetate in hexane) to give (9H-fluoren-9-yl)methyl (S)-4-formyl-2,2-dimethylazolidine-3-carboxylate (0.206 g, 0.586 mmol, 22%) as a colorless oil.
$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 9.14 (s, 1H) 7.71-7.51 (m, 4H), 7.41-7.31 (m, 4H), 4.80 (m, 1H), 4.62-3.89 (m, 5H), 1.60 (s, 3H), 1.49 (s, 3H).

**Methyl (benzyloxy)carbonyl-L-serinate (2c)**

![Chemical Structure](image)

START
RR2-RF1
STIR-RF1
RR3-RF1
RR1-RF1
RR4-RR1-RF1
WAIT(Min) (Wait 300 min)
RS2-RF1
WAIT(Min) (Wait 3 min)
STIROFF-RF1
RS1-RF1
SEP-RF1 (UP)
SF1-RF1
RS1-RF1
SEP-RF1(UP)
SF2-SF
RS3-RF1
The reagents, washings and solvents were placed in each reservoirs and bottles as described below.

RF1  methyl L-serinate hydrochloride (1.00 g, 6.43 mmol)
RR1  Cbz-Cl (1.10 mL, 7.72 mmol) in 15.0 mL of dioxane
RR2  dioxane (5.00 mL)
RR3  NaHCO$_3$ (1.62 g, 19.3 mmol) in 25.7 mL of H$_2$O
RR4  dioxane 4.20 mL
RS1  EtOAc 120 mL
RS2  HCl (1 M aqueous) 60.0 mL
RS3  NaCl (10% aqueous) 80.0 mL
DT2  Na$_2$SO$_4$

To a solution of methyl L-serinate hydrochloride (1, 1.00 g, 6.43 mmol, 1.00 equiv, RF1) in dioxane (5.00 mL, RR2) were added NaHCO$_3$ (1.62 g, 19.3 mmol, 3.00 equiv, RR3) in H$_2$O (25.7 mL, RR3) and Cbz-Cl (1.10 mL, 7.72 mmol, 1.20 equiv, RR1) in dioxane (15.0 mL, RR1) diluted with dioxane (4.20 mL, RR4) and transferred to RF1 at 25 °C. After being stirred at the same temperature for 5 h, the reaction mixture (RF1) was quenched with 1 M HCl aq. (RS2) and the aqueous layer was extracted with two portions of ethyl acetate (RS1). The combined organic layer was washed with 10% NaCl aq. (RS3), dried over Na$_2$SO$_4$ (DT2), filtered, and concentrated in vacuo. The residue was
purified by column chromatography on silica gel (50% ethyl acetate in hexane) to give methyl ((benzyl oxy)carbonyl)-L-serinate (1.50 g, 5.92 mmol, 92%) as a colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) δ 7.37-7.31 (m, 5H), 5.74 (s, 1H), 5.12 (s, 2H), 4.45 (t, $J = 7.5$ Hz, 1H), 4.01-3.91 (m, 2H), 3.78 (s, 3H), 2.34 (s, 1H).

3-benzyl 4-methyl (S)-2,2-dimethyloxazolidine-3,4-dicarboxylate (3c)

START
STIR-RF1
RR1-RF1
RR4-RR1-RF1
RR3-RF1
RR6-RR3-RF1
WAIT(Min) (Wait 360 min)
STIROFF-RF1
RF1-SF
SF-SF2
RR2-RF1
SF2-RF1
STIR-RF1
WAIT(Min) (Wait 5 min)
The reagents, washings and solvents were placed in each reservoirs and bottles as described below.

RF1  methyl ((benzyloxy)carbonyl)-L-serinate (7.00 g, 27.6 mmol) in 30.0 mL of CH₂Cl₂

RR1  2,2-dimethoxypropane (10.1 mL, 82.8 mmol) in 10.0 mL of CH₂Cl₂

RR2  NaOH (10% aqueous) 40.0 mL

RR3  BF₃·OEt₂ (0.347 mL, 2.76 mmol) in 5.00 mL of CH₂Cl₂

RR4  CH₂Cl₂ 5.00 mL

RR6  CH₂Cl₂ 5.00 mL

RS1  EtOAc 160 mL

RS3  NaCl (10% aqueous) 80.0 mL

DT2  Na₂SO₄
To a solution of methyl ((benzyloxy)carbonyl)-L-serinate (7.00 g, 27.6 mmol, 1.00 equiv, RF1) in CH$_2$Cl$_2$ (30.0 mL, RF1) were added 2,2-dimethoxypropane (10.1 mL, 82.8 mmol, 3.00 equiv, RR1) in CH$_2$Cl$_2$ (10.0 mL, RR1) diluted with CH$_2$Cl$_2$ (5.00 mL, RR4) and boron trifluoride-ethyl ether complex (0.347 mL, 2.76 mmol, 0.100 equiv, RR3) in CH$_2$Cl$_2$ (5.00 mL, RR3) diluted with CH$_2$Cl$_2$ (5.00 mL, RR6) and transferred to RF1 at 25 °C. After being stirred at the same temperature for 3 h, the reaction mixture (RF1) was quenched with saturated NaOH aq. (RR2) and the aqueous layer was extracted with two portions of ethyl acetate (RS1). The combined organic layer was washed with 10% NaCl aq. (RS3), dried over Na$_2$SO$_4$ (DT2), filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (16% ethyl acetate in hexane) to give 3-benzyl 4-methyl (S)-2,2-dimethyloxazolidine-3,4-dicarboxylate (6.44 g, 22.0 mmol, 80%) as a colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) δ 7.37-7.30 (m, 5H), 5.23-5.03 (m, 2H), 4.55-4.47 (m, 2H), 4.18-4.08 (m, 1H), 3.64 (s, 3H), 1.71 (s, 3H), 1.56 (s, 3H).

**Benzyl (S)-4-formyl-2,2-dimethyloxazolidine-3-carboxylate (4c)**

![Chemical structure]  

DIBAL, toluene  
-80 °C, 4 h, 31%  

**START**  
**STIR-RF1**  
**RR2-RF1**  
**WAIT(Min)** (Wait 240 min)
RS2-RF1
RS1-RF1
WAIT(Min) (Wait 60 min)
STIROFF-RF1
SEP-RF1(UP)
SF1-RF1
RS1-RF1
SEP-RF1(UP)
SF2-SF
RS3-RF1
SEP-RF1(UP)
SF2-DT1-CF1
END

The reagents, washings and solvents were placed in each reservoirs and bottles as described below.

RF1  3-benzyl 4-methyl (S)-2,2-dimethyloxazolidine-3,4-dicarboxylate (1.00 g, 3.41 mmol, 1.00 equiv., RF1) in 40.0 mL of toluene

RR2  Diisobutylaluminum Hydride (6.82 mL, 6.82 mmol, 2.00 equiv.)

RS1  EtOAc 160 mL

RS2  Rochelle salt (10% aqueous) 60.0 mL

RS3  NaCl (10% aqueous) 80.0 mL

DT1  Na₂SO₄
To a solution of 3-benzyl 4-methyl (S)-2,2-dimethyloxazolidine-3,4-dicarboxylate (3c, 1.00 g, 3.41 mmol, 1.00 equiv, RF1) in toluene (40.0 mL, RF1) were added DIBAL (6.82 mL, 6.82 mmol, 2.00 equiv, RR2) and transferred to RF1 at −80 °C. After being stirred at the same temperature for 4 h, the reaction mixture was quenched with saturated aqueous Rochelle salt and the aqueous layer (RS2) at 25 °C. After being stirred at the same temperature for 1 h, the reaction mixture was extracted with two portions of ethyl acetate (RS1). The combined organic layer was washed with 10% NaCl aq. (RS3), dried over Na₂SO₄ (DT1), filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (25% ethyl acetate in hexane) to give benzyl (S)-4-formyl-2,2-dimethyloxazolidine-3-carboxylate (0.276 g, 1.05 mmol, 31%) as a colorless oil.

¹H NMR (500 MHz, CDCl₃) δ 9.53 (s, 1H) 7.36-7.28 (m, 5H), 5.22-5.01 (m, 2H), 4.41-4.31 (m, 2H), 4.12-4.02 (m, 1H), 1.66 (s, 3H), 1.56 (s, 3H).