Supporting Information

Highly regioselective aminobromination of $\alpha,\beta$-unsaturated nitro compounds with benzyl carbamate/$N$-bromosuccinimide as nitrogen/bromine source

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1. General information

Unless otherwise stated, all reagents were purchased from commercial sources and used without further purification. Reaction progress was monitored by TLC using silica gel 60F-254 with detection by UV. Flash chromatography was performed using silica gel 60 (200-300mesh). Thin layer chromatography was carried out on silica gel 60 F-254 TLC plates of 20 cm × 20 cm. Melting points are uncorrected. IR spectra were collected on Bruker Vector 22 in KBr pellets. $^1$H and $^{13}$C NMR (TMS used as internal standard) spectra were recorded with a Bruker ARX300 spectrometer. High resolution mass spectra for all the new compounds were done by Micro mass Q-Tof instrument (ESI). The crystal structure was recorded on a X-ray diffraction spectrometer.

2. Typical procedure for aminohalogenation of $\alpha,\beta$-unsaturated nitro compounds with CbzNH$_2$/NBS

Into a vial were added $\alpha,\beta$-unsaturated nitro compounds substrates (0.5 mmol), NBS (1.5 mmol), CbzNH$_2$ (1.5 mmol), K$_3$PO$_4$ (5 mol%). Then, 3 mL of acetonitrile was added to the vial. The solution was stirred at room temperature without the protection of inert gas and monitored by TLC. When the reaction was completed, the mixture was directly purified by TLC plate (Petroleum ether/EtOAc, 4:1).

Benzyl 2,2-dibromo-2-nitro-1-phenylethylcarbamate (3a)

White solid (93% yield). mp 86–87 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.35–7.45 (m, 10H), 6.42 (d, $J = 10.2$ Hz, 1H), 5.84 (d, $J = 10.2$ Hz, 1H), 5.08–5.17 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 154.92, 135.58, 133.38, 129.8, 129.1, 128.67, 128.63, 128.52, 128.41, 93.83, 67.99, 65.15. IR (KBr): $\nu = 3280, 3064, 2965, 1690, 1573, 1531, 1251$ cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For C$_{16}$H$_{14}$N$_2$O$_4$Br$_2$Na: 480.9186; found: 480.9193.

Benzyl 2,2-dibromo-1-(2-chlorophenyl)-2-nitroethylcarbamate (3b)

White solid (55% yield). mp 98–99 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.29–7.43 (m, 10H), 6.42 (d, $J = 10.2$ Hz, 1H), 5.84 (d, $J = 10.2$ Hz, 1H), 5.08–5.17 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 154.92, 135.58, 133.38, 129.8, 129.1, 128.67, 128.63, 128.52, 128.41, 93.83, 67.99, 65.15. IR (KBr): $\nu = 3280, 3064, 2965, 1690, 1573, 1531, 1251$ cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For C$_{16}$H$_{14}$N$_2$O$_4$Br$_2$Na: 480.9186; found: 480.9193.
9H), 6.03 (d, J = 10.3 Hz, 1H), 5.79 (d, J = 10.1 Hz, 1H), 5.02–5.21 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 154.74, 135.99, 135.40, 131.90, 130.42, 128.9, 128.85, 128.71, 128.68, 128.61, 128.41, 93.10, 68.10, 64.52. IR (KBr): $\nu$ = 3339, 3035, 2949, 1693, 1576, 1536, 1352, 1286, 1260, 1028 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd. For C$_{16}$H$_{13}$N$_2$O$_4$Br$_2$ClNa: 514.8798; found 514.8802.

**Benzyl 2,2-dibromo-1-(3-chlorophenyl)-2-nitroethylcarbamate (3c)**

White solid (88% yield). mp 87–89 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.47–7.57 (m, 2H), 7.28–7.43 (m, 7H), 6.03 (d, J = 10.0 Hz, 1H), 5.94 (d, J = 10.0 Hz, 1H), 5.05–5.19 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 154.75, 135.38, 132.45, 131.85, 130.69, 128.69, 128.62, 128.41, 128.13, 124.26, 92.99, 68.10, 64.59. IR (KBr): $\nu$ = 3257, 3059, 2967, 1704, 1683, 1577, 1540, 1494, 1258 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd. For C$_{16}$H$_{13}$N$_2$O$_4$Br$_2$ClNa: 514.8797; found 514.8802.

**Benzyl 2,2-dibromo-1-(4-bromophenyl)-2-nitroethylcarbamate (3d)**

Colorless oil (91% yield). $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.47–7.57 (m, 2H), 7.28–7.42 (m, 7H), 6.03 (d, J = 9.89 Hz, 1H), 5.94 (d, J = 9.8 Hz, 1H), 5.03–5.19 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 154.73, 135.38, 132.45, 131.85, 130.69, 128.69, 128.62, 128.41, 124.26, 92.99, 68.10, 64.59. IR (KBr): $\nu$ = 3409, 3310, 3034, 2958, 1708, 1577, 1490, 1323, 1232 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd. For C$_{16}$H$_{13}$N$_2$O$_4$Br$_3$ClNa: 558.8284; found 558.8298.

**Benzyl 2,2-dibromo-1-(3-fluorophenyl)-2-nitroethylcarbamate (3e)**

White solid (86% yield). mp 102–104 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.29–7.42 (m, 6H), 7.07–7.25 (m, 3H), 6.04 (d, J = 10.3 Hz, 1H), 5.81 (d, J = 10.1 Hz, 1H), 5.06–5.2 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 162.43 (d, $^1$$J_{CF}$ = 248.09 Hz), 154.82, 135.73 (d, $^3$$J_{CF}$ = 6.43 Hz), 135.42, 130.23 (d, $^3$$J_{CF}$ = 7.81 Hz), 128.68, 128.6, 128.39, 125.06, 116.87 (d, $^2$$J_{CF}$ = 21.13 Hz), 116.2 (d, $^2$$J_{CF}$ = 23.12 Hz), 92.91, 68.12, 64.60. IR (KBr): $\nu$ = 3278, 3063, 2968, 1689, 1575, 1533, 1251, 1236 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd. For C$_{16}$H$_{13}$N$_2$O$_4$Br$_2$FNa: 498.9105; found 498.9099.

**Benzyl 2,2-dibromo-1-(4-fluorophenyl)-2-nitroethylcarbamate (3f)**
White solid (85% yield). mp 91–93 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.39–7.49 (m, 2H), 7.28–7.38 (m, 5H), 7.02–7.13 (m, 2H), 6.03 (d, $J = 10.1$ Hz, 1H), 5.83 (d, $J = 10.2$ Hz, 1H), 5.05–5.19 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 163.35 (d, $J_{CF} = 250.46$ Hz), 154.81, 135.46, 131.02 (d, $J_{CF} = 8.37$ Hz), 129.32, 128.68, 128.59, 128.37, 115.74 (d, $J_{CF} = 22.04$ Hz), 93.55, 68.06, 64.52. IR (KBr): $\nu = 3264, 3062, 3038, 1701, 1686, 1579, 1511, 1326, 1257, 1232$ cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd. For C$_{16}$H$_{13}$N$_2$O$_4$Br$_2$FNa: 498.9104; found 498.9099.

**Benzyl 2,2-dibromo-2-nitro-1-m-tolylethylcarbamate (3g)**

White solid (97% yield). mp 109–111 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.13–7.46 (m, 9H), 6.01 (d, $J = 10.3$ Hz, 1H), 5.87 (d, $J = 10.4$ Hz, 1H), 5.01–5.24 (m, 2H), 2.35 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 154.84, 138.44, 135.53, 133.25, 130.53, 129.77, 128.65, 128.57, 128.52, 128.43, 125.98, 93.79, 67.95, 65.11, 21.48. IR (KBr): $\nu = 3275, 3060, 3038, 1705, 1688, 1574, 1533, 1251, 1054$ cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd. For C$_{17}$H$_{16}$N$_2$O$_4$Br$_2$Na 494.9336; found 494.935.

**Benzyl 2,2-dibromo-2-nitro-1-p-tolylethylcarbamate (3h)**

White solid (62% yield). mp 93–95 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.27–7.43 (m, 7H), 7.14–7.22 (m, 2H), 6.01 (d, $J = 10.2$ Hz, 1H), 5.83 (d, $J = 10.2$ Hz, 1H), 5.05–5.19 (m, 2H), 2.36 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 154.89, 139.88, 135.57, 130.32, 129.34, 128.89, 128.65, 128.52, 128.40, 94.06, 67.93, 64.95, 21.23. IR (KBr): $\nu = 3411, 3312, 3033, 2957, 1712, 1575, 1513, 1324, 1233, 1049$ cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd. For C$_{17}$H$_{16}$N$_2$O$_4$Br$_2$Na: 494.9340; found 494.9350.

**Benzyl 2,2-dibromo-1-(naphthalen-1-yl)-2-nitroethylcarbamate (3i)**

Yellow solid (89% yield). mp 103–105 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 8.44 (d, $J = 8.12$ Hz, 1H), 7.45–7.98 (m, 6H), 7.27–7.43 (m, 5H), 7.1 (d, $J = 10.2$ Hz, 1H), 5.94 (d, $J = 10.1$ Hz, 1H), 5.01–5.18 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 155.01, 135.44, 133.74, 132.01, 131.16, 130.56, 129.03, 128.64, 128.52, 128.39, 127.32, 126.41, 125.24, 124.92, 123.44, 93.76, 68.02, 58.63. IR (KBr): $\nu = 3412, 3311, 3065, 3035, 1713, 1577, 1322, 1058, 910$ cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For
C$_{20}$H$_{16}$N$_2$O$_4$Br$_2$Na: 530.9350; found 530.9350.

**Benzy1 2,2-dibromo-1-(3-methoxyphenyl)-2-nitroethylcarbamate (3j)**

White solid (83% yield). mp 97–99°C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.28–7.45 (m, 6H), 6.89–7.05 (m, 3H), 6.01 (d, $J = 10.3$ Hz, 1H), 5.81 (d, $J = 10.3$ Hz, 1H), 5.05–5.19 (m, 2H), 3.81 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 159.52, 154.82, 135.50, 134.74, 129.67, 128.64, 128.52, 128.4, 121.17, 115.16, 114.92, 93.53, 67.98, 65.06, 55.38. IR (KBr): $\nu$ = 3286, 3062, 2960, 2886, 1691, 1574, 1531, 1251, 1230, 1056 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For C$_{17}$H$_{16}$N$_2$O$_5$Br$_2$Na: 510.9304; found 510.9299.

**Benzy1 2,2-dibromo-1-(4-methoxyphenyl)-2-nitroethylcarbamate (3k)**

Yellow oil (95% yield). $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.29–7.44 (m, 7H), 6.84–6.93 (m, 2H), 5.99 (d, $J = 10.4$ Hz, 1H), 5.79 (d, $J = 10.2$ Hz, 1H), 5.05–5.21 (m, 2H), 3.81 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 160.54, 154.85, 135.57, 130.28, 128.64, 128.51, 128.38, 125.17, 113.99, 94.28, 67.92, 64.73, 55.33. IR (KBr): $\nu$ = 3411, 3317, 2959, 2839, 1713, 1574, 1513, 1250, 1030 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For C$_{17}$H$_{16}$N$_2$O$_5$Br$_2$Na: 510.9299; found 510.9299.

**Benzy1 1-(4-(benzyloxy)phenyl)-2,2-dibromo-2-nitroethylcarbamate (3l)**

Colorless oil (78% yield). $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.27–7.58 (m, 12H), 6.94–7.08 (m, 2H), 6.53 (d, $J = 10.3$ Hz, 1H), 6.44 (d, $J = 10.2$ Hz, 1H), 5.03–5.21 (m, 4H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 156.79, 155.06, 136.15, 135.79, 131.01, 130.62, 128.80, 128.59, 128.37, 128.24, 127.35, 121.05, 112.91, 94.01, 70.69, 67.68, 62.11. IR (KBr): $\nu$ = 3412, 3316, 3033, 2955, 2882, 1716, 1575, 1497, 1227, 1043 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd. For C$_{23}$H$_{20}$N$_2$O$_5$Br$_2$Na: 586.9605; found 586.9612.

**Benzy1 2,2-dibromo-2-nitro-1-(4-(trifluoromethyl)phenyl)ethylcarbamate (3m)**

Colorless oil (83% yield). $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.66 (d, $J = 8.52$ Hz, 2H), 7.59 (d, $J = 8.52$ Hz, 2H), 7.29–7.43 (m, 5H), 6.12 (d, $J = 10.4$ Hz, 1H), 5.85 (d, $J =$
10.2 Hz, 1H), 5.05–5.19 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): δ 154.69, 137.36, 135.32, 131.85 (q, $^2$J$_{CF}$ = 32.91 Hz), 129.64, 128.68, 128.4, 125.59 (q, $^3$J$_{CF}$ = 3.27 Hz), 123.65 (q, $^1$J$_{CF}$ = 273.29 Hz), 92.46, 68.18, 64.64. IR (KBr): ν = 3415, 3311, 3035, 2959, 1712, 1578, 1326, 1131, 1071, 1018 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For C$_{17}$H$_3$N$_2$O$_4$Br$_2$F$_3$Na: 548.9075; found 548.9067.

**Benzyl 2,2-dibromo-1-(furan-2-yl)-2-nitroethylcarbamate (3n)**

Brown oil (68% yield). $^1$H NMR (300 MHz, CDCl$_3$): δ 7.42 (dd, $J$ = 0.75, 1.78 Hz, 1H), 7.31–7.41 (m, 5H), 6.44 (d, $J$ = 3.32 Hz, 1H), 6.38 (dd, $J$ = 1.9, 3.35 Hz, 1H), 6.24 (d, $J$ = 10.44 Hz, 1H), 5.84 (d, $J$ = 10.23 Hz, 1H), 5.11–5.26 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): δ 155.07, 146.03, 143.74, 135.49, 128.68, 128.58, 128.39, 111.54, 110.95, 91.18, 68.11, 60.45. IR (KBr): ν = 3254, 3034, 2956, 1694, 1572, 1538, 1323, 1257, 1016 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For C$_{14}$H$_{12}$N$_2$O$_5$Br$_2$Na: 470.8971; found 470.8986.

**Benzyl 2,2-dibromo-2-nitro-1-(thiophen-2-yl)ethylcarbamate (3o)**

Yellow solid (72% yield). mp 98–100 °C. $^1$H NMR (300 MHz, CDCl$_3$): δ 7.29–7.42 (m, 6H), 7.18 (d, $J$ = 3.72 Hz, 1H), 7.01 (dd, $J$ = 3.62, 5.13 Hz, 1H), 6.38 (d, $J$ = 10.44 Hz, 1H), 5.7 (d, $J$ = 10.39 Hz, 1H), 5.07–5.24 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): δ 154.72, 135.42, 129.58, 128.66, 128.57, 128.38, 127.37, 126.9, 126.83, 92.62, 68.11, 62.18. IR (KBr): ν = 3269, 3033, 2955, 1693, 1577, 1522, 1323, 1249 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For C$_{14}$H$_{12}$N$_2$O$_4$Br$_2$Na: 486.8753; found 486.8757.

**Benzyl 1,1-dibromo-1-nitrononan-2-ylcarbamate (3p)**

Colorless oil (82% yield). $^1$H NMR (300 MHz, CDCl$_3$): δ 7.31–7.42 (m, 5H), 5.08–5.25 (m, 2H), 4.98 (d, $J$ = 10.47 Hz, 1H), 4.65–4.88 (m, 1H), 1.76–1.97 (m, 1H), 1.07–1.55 (m, 11H), 0.88 (t, $J$ = 6.97Hz, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): δ 155.57, 135.83, 128.62, 128.44, 128.21, 93.84, 67.66, 62.43, 32.27, 31.65, 29.00, 28.92, 25.81, 22.60, 14.09. IR (KBr): ν = 3404, 3309, 2955, 2928, 2857, 1709, 1575, 1326, 1247, 1052 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For C$_{17}$H$_{24}$N$_2$O$_4$Br$_2$Na: 502.9978;
found 502.9976.

**Benzyl 2,2-dibromo-1-(3-bromo-4-methoxyphenyl)-2-nitroethylcarbamate (3q)**

Colorless oil (87% yield). $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.65 (d, $J = 2.36$ Hz, 1H), 7.28–7.42 (m, 6H), 6.86 (d, $J = 8.57$ Hz, 1H), 5.99 (d, $J = 10.42$ Hz, 1H), 5.84 (d, $J = 10.26$ Hz, 1H), 5.04–5.18 (m, 2H), 3.9 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 156.88, 154.67, 135.4, 133.62, 129.65, 128.67, 128.57, 128.39, 126.69, 111.79, 111.48, 93.51, 68.07, 64.12, 56.34. IR (KBr): $\nu$ = 3409, 3309, 2209, 2958, 2841, 1712, 1576, 1498, 1294, 1056 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd For C$_{17}$H$_{15}$N$_2$O$_5$Br$_3$Na: 588.8406; found 588.8404.

**Benzyl 2,2-dibromo-1-(4-cyanophenyl)-2-nitroethylcarbamate (3r)**

Colorless oil (81% yield). $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.68 (d, $J = 8.35$ Hz, 2H), 7.59 (d, $J = 8.35$ Hz, 2H), 7.28–7.41 (m, 5H), 6.11 (d, $J = 10.51$ Hz, 1H), 5.92 (d, $J = 10.41$ Hz, 1H), 5.06–5.18 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 154.59, 138.48, 135.19, 132.30, 129.95, 128.69, 128.41, 117.89, 113.83, 91.88, 68.27, 64.62. IR (KBr): $\nu$ = 3312, 2957, 2232, 1731, 1715, 1576, 1506, 1232, 1050 cm$^{-1}$. HRMS (ESI/[M+Na]$^+$) Calcd. For C$_{17}$H$_{13}$N$_3$O$_4$Br$_2$Na: 505.9150; found 505.9146.
3. X-ray crystal structure of compound 3a

Figure 1 X-ray crystal structure of compound 3a (CCDC number 859296)
4. Removal of *N*-carbobenzoxy protecting group

To a flask containing 3a (2 mmol), a solution of HBr in AcOH (5mL, 33% w/w) was added. The mixture was stirred at room temperature for 2 h. When evolution of bubbles stopped, excess HBr and HOAc were filtered, giving white powder 4 with 85% yield.

*2,2-dibromo-2-nitro-1-phenylethanaminium bromide (4)* White solid (85% yield).

$^1$H NMR (300 MHz, D$_2$O): $\delta$ 7.48–7.53 (m, 5H), 5.33 (s, 1H). $^{13}$C NMR (75 MHz, D$_2$O): $\delta$ 131.66, 130.6, 129.7, 128.42, 127.67, 59.26. IR (KBr): $\nu$ = 3264, 3013, 2908, 1959, 1630, 1573, 1499, 1399, 997cm$^{-1}$. 

Electronic Supplementary Material (ESI) for RSC Advances
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5. $^1$H and $^{13}$C NMR spectra for compound 3 and 4

$^1$H and $^{13}$C NMR spectra of 3a
$^1$H and $^{13}$C NMR spectra of 3b
$^1$H and $^{13}$C NMR spectra of 3c
$^1$H and $^{13}$C NMR spectra of 3d
$^1$H and $^{13}$C NMR spectra of 3e
$^1$H and $^{13}$C NMR spectra of 3f
$^1$H and $^{13}$C NMR spectra of 3g
\(^1\)H and \(^{13}\)C NMR spectra of 3h
$^1$H and $^{13}$C NMR spectra of 3i
$^1$H and $^{13}$C NMR spectra of 3j
$^1$H and $^{13}$C NMR spectra of 3k
$^1$H and $^{13}$C NMR spectra of 3l
$^1$H and $^{13}$C NMR spectra of 3m
$^1$H and $^{13}$C NMR spectra of 3n
$^1$H and $^{13}$C NMR spectra of 3o

Electronic Supplementary Material (ESI) for RSC Advances
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$^1$H and $^{13}$C NMR spectra of 3p
$^1$H and $^{13}$C NMR spectra of 3q
\[ ^1H \text{ and } ^{13}C \text{ NMR spectra of } 3\alpha \]
$^1$H and $^{13}$C NMR spectra of Deprotection Product of 4