Supporting Information

A new route to bicyclo[1.1.1]pentan-1-amine from 1-azido-3-iodobicyclo[1.1.1]pentane

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General Information

Materials. All chemicals were purchased from Sigma-Aldrich, Alfa Aesar, Merck and TCI and were used as received. Dried solvents (DCM, THF and diethyl ether) were drawn from GlassContour Solvent Dispensing System. Benzene was taken from Sure/Seal™ bottle (Sigma-Aldrich). All reactions requiring anhydrous conditions were carried out under argon atmosphere using oven-dried glassware. Reaction progress was monitored using Merck 60 F254, 0.25 μm silica gel plates (TLC plates) and spots were visualized by UV and/or phosphomolybdic acid, potassium permanganate, or ceric ammonium molybdate stain. Flash column chromatography was carried out using Merck 60 F254, 0.040-0.063 μm silica gel. Preparative TLC chromatography was carried out using Merck 60 F254, 0.25 μm silica gel plates. Names of structures were generated using ChemBioDraw Ultra 13.0.2.

Instrumentation. Proton nuclear magnetic resonance (1H NMR) spectra and carbon nuclear magnetic resonance (13C NMR) spectra were recorded on Bruker 400 MHz with CryoProbe or on Bruker 600 MHz. Chemical shifts for protons were reported in parts per million (ppm) that are referenced to residual protium in the NMR solvent (CDCl₃: 7.26 ppm; CD₃OD: 3.31 ppm; DMSO-d₆: 2.50 ppm; D₂O: 4.79 ppm). Chemical shifts for carbon were reported in ppm referenced to the carbon resonances of the NMR solvent (CDCl₃: 77.16 ppm; CD₃OD: 49.0 ppm; DMSO-d₆: 39.52 ppm). NMR spectra were processed using MestReNova 8.1.1. Data is presented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), integration and coupling constants (J) in Hertz (Hz). Electron impact mass spectra (EIMS) were measured using a Finnigan MAT95XP double-focusing mass spectrometer. High resolution electrospray ionization (HRMS ESI) mass spectra were recorded using Agilent 6210 Time-of-Flight LC/MS. Infrared (IR) spectra were measured on a PerkinElmer Spectrum100 FT-IR spectrophotometer. UV reactions were carried out using a Rayonet Reactor, RMR-600, at 254 nm. Differential scanning calorimetry (DSC) measurements were performed using Mettler Toledo DSC 1. DSC spectra were processed using STAR® SW 10.00 software.
Experimental Procedures

Procedure for key 1-azido-3-iodobicyclo[1.1.1]pentane (2) intermediate

1,3-diiodobicyclo[1.1.1]pentane (5).

A modified combination of procedures reported by Sh tarev et al.¹ and Wiberg and Wadell² groups was employed. To a stirred and cooled mixture (~ 40 °C, acetone/dry ice bath) of 1,1-dibromo-2,2-bis(chloromethyl)cyclopropane (3) (63 g, 212.3 mmol) in diethyl ether (100 mL) under argon atmosphere in a 3-neck RBF was added MeLi·LiBr complex (297.2 mL, 445.7 mmol, 1.5 M in diethyl ether) in a dropwise fashion through an addition funnel over a period of 1 h while maintaining the temperature between -30 and – 40 °C. After the addition was complete, the acetone/dry ice bath was replaced with an ice bath and stirred at 0 °C for 3 h, where propellane (4) was formed. Sublimed iodine crystals (56.6 g, 222.9 mmol) were slowly added to this reaction mixture over a period of 1 h with the aid of a solid addition funnel while maintaining the temperature at 0 °C. After the addition was complete, ice bath was allowed to expire and stirring was continued overnight. The reaction mixture was then diluted with ethyl acetate, quenched with saturated sodium thiosulphate, washed with brine and dried over sodium sulphate. This organic extract was filtered through a pad of silica gel and concentrated \textit{in vacuo} to give a yellow residue. This residue was triturated with cold ethyl acetate followed by methanol to afford pure 5 (46.1 g, 68%, yield over 2 steps) as an off-white solid.

\(^1\text{H NMR (400 MHz, CDCl}_3\) \(\delta 2.67 (s, 6H) \text{ ppm.} \) \(^{13}\text{C NMR (101 MHz, CDCl}_3\) \(\delta 68.3, -1.6 \text{ ppm.} \) [lit.³ \(^1\text{H NMR (250 MHz, CDCl}_3\) \(\delta 2.66 (s, 6H) \text{ ppm.} \) \(^{13}\text{C NMR (62.5 MHz, CDCl}_3\) \(\delta 68.2, 1.0 \text{ ppm.} \)]

1-azido-3-iodobicyclo[1.1.1]pentane (2).

\(\text{N}_3\)

1-azido-3-iodobicyclo[1.1.1]pentane (2) was synthesized from 5 according to the procedure reported by Wiberg and McMurdie.³ The synthesized compound was in a good agreement with the reported analytical data and was obtained in the yield of 76% (lit = 79%)

\(^1\text{H NMR (400 MHz, CDCl}_3\) \(\delta 2.44 (s, 6H) \text{ ppm.} \) \(^{13}\text{C NMR (101 MHz, CDCl}_3\) \(\delta 61.5, 55.3, -2.8 \text{ ppm.} \) [lit.³ \(^1\text{H NMR (CDCl}_3\) \(\delta 2.43 (s, 6H) \text{ ppm.} \) \(^{13}\text{C NMR (CDCl}_3\) \(\delta 61.3, 55.1, -3.1 \text{ ppm.} \)] DSC Normalized thermal potential exotherm: 2067 J/g; Onset temperature: 178 °C.
General procedure for optimization studies for the reduction of 1-azido-3-iodobicyclo[1.1.1]pentane (2) to bicyclo[1.1.1]pentan-1-amine hydroiodide (1).

To a heterogeneous mixture of 1-azido-3-iodobicyclo[1.1.1]pentane (2) (300 mg, 1.28 mmol) in water (2.6 mL) was added TTMSS (0.87 mL, 2.81 mmol) and the reaction mixture stirred for 15 min, during which precipitation of yellow solids occurred. 2-mercaptoethanol* (13 µL, 0.19 mmol) followed by AIBN* (21 mg, 0.13 mmol) were added and the resulting mixture was stirred for the specified period, either at rt or at 80 °C, with a water condenser attached regardless of conditions. The reaction mixture was allowed to cool to rt where water (5 mL) was added and the mixtures stirred for 5 min. EtOAc was added to aid phase separation, and the aqueous layer collected. The organic phase was washed with water, and the combined aqueous extracts evaporated to dryness. The yellow residue obtained was triturated with EtOAc, then dried in vacuo to give 1 (HI salt) as an off-white solid. The filtrate may be evaporated and re-triturated to recover a second crop of 1 (HI salt) with greater coloration** but with no significant amounts of impurities detected by 1H and 13C NMR.

*Additives may be omitted directly if conditions do not require them.

**1 (HI salt) slowly gains coloration over time on standing in open air at rt, but its purity by 1H and 13C NMR does not seem to be affected. The coloration is significantly slowed by storing over argon at -24 °C.

Procedure of bicyclo[1.1.1]pentane derivatives

Multigram scale synthesis of bicyclo[1.1.1]pentan-1-amine hydroiodide (1).

To a heterogeneous mixture of 1-azido-3-iodobicyclo[1.1.1]pentane (2) (14.09 g, 60 mmol) in water (75 mL) was added TTMSS (40.7 mL, 131.9 mmol) and the resulting mixture was stirred for 10 min, during which a gentle exotherm was observed. 2-mercaptoethanol (0.42 mL, 6 mmol) followed by AIBN (246 mg, 1.5 mmol) were added and stirred for another 10 min. The reaction mixture was heated at 80 °C and stirred for 4.5 h, during which an additional identical amount of AIBN was added at 1.5 h and 3 h interval. The reaction mixture was allowed to cool to rt, rinsed out with a small amount of EtOAc and water, and the aqueous layer collected. The organic layer was washed with water (2 x 10 mL), and the combined aqueous extracts washed with DCM (3 x 10 mL), EtOAc (3 x 10
mL), then fully evaporated to dryness. The yellow residue obtained was washed with EtOAc, then filtered and dried in vacuo to afford 1 (HI salt, 10.31 g) as an off-white powder. The EtOAc washings were evaporated and the residue washed with EtOAc to give a second crop of the 1 (136 mg, combined 82%) as an off-white powder (without any significant increase in coloration).

\[ \text{1H NMR (600 MHz, D}_2\text{O) } \delta 2.66 \text{ (s, 1H), 2.10 (s, 6H) ppm.} \]

\[ \text{1H NMR (400 MHz, CD}_2\text{OD) } \delta 2.66 \text{ (s, 1H), 2.10 (s, 6H) ppm.} \]

\[ \text{13C NMR (151 MHz, D}_2\text{O) } \delta 51.0, 45.0, 23.3 \text{ ppm.} \]

\[ \text{13C NMR (101 MHz, CD}_2\text{OD) } \delta 52.4, 46.8, 24.5 \text{ ppm.} \]

HRMS (ESI TOF, m/z): [M-HI+H]+ Calcd for C15H10N m/z 84.0808; Found 84.0811. IR: 3446, 2942, 1634, 1234, 615 cm\(^{-1}\). DSC

Normalized thermal potential exotherm: 792 J/g; Onset temperature: 215 °C.

1-(3-iodobicyclo[1.1.1]pentan-1-yl)-4-phenyl-1H-1,2,3-triazole (7).

To a solution of 1-azido-3-iodobicyclo[1.1.1]pentane (2) (200 mg, 0.85 mmol) in anhydrous THF (8.5 mL) was added CuI (16.2 mg, 0.085 mmol), phenyl acetylene (140.2 µL, 1.28 mmol), and Et\(_3\)N (237.4 µL, 1.70 mmol) and the resulting mixture was allowed to stir at rt for 18 h. THF was removed under reduced pressure, diluted with DCM and filtered through a pad of Celite\textsuperscript®545. The filtrate was concentrated in vacuo and purified by silica gel flash chromatography (gradient elution from 0:50:50 to 10:40:50 with product eluting from 5:45:50 to 7.5:42.5:50 diethyl ether-hexanes-DCM), followed by a trituration with hexanes to afford 7 (254 mg, 89%) as a white solid.

\[ \text{1H NMR (400 MHz, CDCl}_3\text{) } \delta 7.85 – 7.77 \text{ (m, 2H), 7.69 (s, 1H), 7.47 – 7.39 (m, 2H), 7.38 – 7.31 (m, 1H), 2.87 (s, 6H) ppm.} \]

\[ \text{1H NMR (400 MHz, CD}_2\text{OD) } \delta 7.85 – 7.77 \text{ (m, 2H), 7.69 (s, 1H), 7.47 – 7.39 (m, 2H), 7.38 – 7.31 (m, 1H), 2.87 (s, 6H) ppm.} \]

\[ \text{13C NMR (101 MHz, CDCl}_3\text{) } \delta 147.9, 130.3, 129.0, 128.5, 126.0, 118.0, 62.1, 54.8, -2.7 \text{ ppm.} \]

\[ \text{13C NMR (101 MHz, CD}_2\text{OD) } \delta 148.9, 131.4, 130.0, 129.5, 128.5, 126.0, 118.0, 62.1, 54.8, -2.7 \text{ ppm.} \]

HRMS (ESI TOF, m/z): [M+H]+ Calcd for C\(_{13}\)H\(_{13}\)IN m/z 338.0149; Found 338.0158; [M+Na]+ Calcd for C\(_{13}\)H\(_{12}\)IN\(_2\)Na m/z 359.9968; Found 359.9977. IR: 1432, 1201, 1064, 874, 767, 693 cm\(^{-1}\).
reaction mixture stirred for another hour. The reaction mixture was allowed to cool down, then water was added and extracted with EtOAc (3 x 10 mL). The organics were dried over anhydrous sodium sulfate and concentrated in vacuo and purified by preparative TLC (20:80 EtOAc-hexanes), followed by a trituration with hexanes to afford 8 (10.2 mg, 65%) as a white solid.

\[^1\text{H} \text{NMR (400 MHz, CDCl}_3 \text{)} \delta 7.87 - 7.77 (m, 2H), 7.73 (s, 1H), 7.46 - 7.37 (m, 2H), 7.36 - 7.29 (m, 1H), 2.73 (s, 1H), 2.44 (s, 6H) \text{ppm.} \]^1\text{C} \text{NMR (101 MHz, CDCl}_3 \text{)} \delta 147.6, 130.8, 128.9, 128.2, 126.0, 118.0, 53.4, 52.1, 23.6 ppm. HRMS (ESI TOF, m/z): [M+H]^+ Calcd for C\text{11}H\text{10}N\text{3} m/z 212.1182; Found 212.1189. IR: 2998, 1430, 1250, 1223, 1031, 907, 766, 694 cm\textsuperscript{-1}.

Alternative method for the synthesis of 1-(bicyclo[1.1.1]pentan-1-yl)-4-phenyl-1H,1,2,3-triazole (8).

To a heterogeneous mixture of 1-(3-iodobicyclo[1.1.1]pentan-1-yl)-4-phenyl-1H,1,2,3-triazole (7) (20 mg, 0.059 mmol) in water (0.6 mL) was added TTMSS (32.8 µL, 0.107 mmol) and AIBN (1.95 mg, 0.012 mmol), and the resulting mixture was heated at 75 °C for 5 h. The reaction mixture was allowed to cool down, diluted with DCM, washed with saturated sodium thiosulphate, brine, dried over sodium sulphate and concentrated in vacuo. The residue was purified by silica gel flash chromatography (gradient elution from 5:95 to 20:80 with product eluting from 10:90 to 15:85 diethyl ether-hexanes), followed by a trituration with hexanes to afford 8 (12.5 mg, 72%) as a white solid.

\[^1\text{H} \text{NMR (600 MHz, CDCl}_3 \text{)} d 7.85 - 7.80 (m, 2H), 7.73 (s, 1H), 7.45 - 7.39 (m, 2H), 7.36 - 7.30 (m, 1H), 3.70 (s, 3H), 2.40 (t, J = 7.5 Hz, 2H), 2.29 (s, 6H), 2.06 (t, J = 7.5 Hz, 2H) ppm. \]^1\text{C} \text{NMR (151 MHz, CDCl}_3 \text{)} d 173.5, 147.6, 130.5, 129.0, 128.3, 125.9, 118.2, 53.4, 52.0, 49.7, 36.0, 25.0 ppm. HRMS (ESI TOF, m/z): [M+H]^+ Calcd for C\text{17}H\text{20}N\text{3}O\text{2} m/z
298.155; Found 298.1551.; [M+Na]$^+$ Calcd for C$_{17}$H$_{13}$N$_3$NaO$_2$ m/z 320.1369; Found 320.1369. IR: 2921, 1732, 1431, 1246, 1178, 769, 696 cm$^{-1}$.

**Proof for the formation of 1-azidobicyclo[1.1.1]pentane (10)**

1-(bicyclo[1.1.1]pentan-1-yl)-4-ethoxy-1H-1,2,3-triazole (11).

To neat 1-azido-3-iodobicyclo[1.1.1]pentane (2) (100 mg, 0.425 mmol) was added TTMSS (509 µL, 1.66 mmol), and the resulting mixture was stirred for 15 min where complete consumption of 2 by $^1$H NMR was observed. The reaction mixture was washed with cold saturated bicarbonate and the organics extracted with THF (1.5 mL). To this THF organic extract was added ethoxyethyne (73 µL, 0.51 mmol, 50% w/w in hexane), Et$_3$N (119 µL, 0.85 mmol) followed by CuI (32.4 mg, 0.17 mmol), then allowed to stir at r.t. for 1.5 h. THF was removed under reduced pressure, diluted with DCM and filtered through a pad of Celite®545. The filtrate was concentrated in vacuo and purified by silica gel flash chromatography (gradient elution from 10:90 to 25:75 with product eluting from 20:80 to 25:75 diethyl ether-hexanes) to afford 11 (70 mg, 91%) as a pale yellow oil.

$^1$H NMR (400 MHz, CDCl$_3$) δ 6.95 (s, 1H), 4.27 (q, $J = 7.0$ Hz, 2H), 2.68 (s, 1H), 2.36 (s, 6H), 1.40 (t, $J = 7.0$ Hz, 3H) ppm. $^{13}$C NMR (101 MHz, CDCl$_3$) δ 160.9, 104.4, 66.5, 53.1, 52.4, 23.4, 15.0 ppm. HRMS (ESI TOF, m/z): [M+H]$^+$ Calcd for C$_9$H$_{14}$N$_3$O m/z 180.1131; Found 180.1137; [M+Na]$^+$ Calcd for C$_{9}$H$_{13}$N$_3$NaO m/z 202.0951; Found 202.0956. IR: 2920, 1568, 1441, 1346, 1212, 1046 cm$^{-1}$.

bicyclo[1.1.1]pentan-1-amine hydrochloride (1).

To neat 1-azido-3-iodobicyclo[1.1.1]pentane (2) (100 mg, 0.425 mmol) was added TTMSS (509 µL, 1.66 mmol), and the resulting mixture was stirred for 15 min where complete consumption of 2 by $^1$H NMR was observed. The reaction mixture was washed with cold saturated bicarbonate and the organics extracted with THF (1.5 mL). To this THF organic layer was added 6 N HCl (1.5 mL) and triphenylphosphine (223 mg, 0.85 mmol), then allowed to stir vigorously at rt for 3 h. The water layer was washed with EtOAc (1 mL x 2) and fully evaporated to dryness. This yellow residue obtained was triturated with EtOAc followed by DCM to afford 1 (HCl salt, 35.5 mg, 70%) as an off-white solid.
**1**H NMR (400 MHz, CD$_3$OD) δ 2.66 (s, 1H), 2.11 (s, 6H) ppm. **13**C NMR (101 MHz, CD$_3$OD) δ 52.4, 46.8, 24.5 ppm. 
**1**H NMR (600 MHz, D$_2$O) δ 2.66 (s, 1H), 2.10 (s, 6H) ppm. **13**C NMR (151 MHz, D$_2$O) δ 50.9, 44.9, 23.3 ppm. [lit.$^4$] 
**1**H NMR (400 MHz, DMSO-$d_6$) δ 2.58 (s, 1H), 1.97 (s, 6H) ppm. **13**C NMR (151 MHz, DMSO-$d_6$) δ 51.08, 45.53, 23.74 ppm. ] HRMS (ESI TOF, m/z): [M+Na]$^+$ Calcd for C$_5$H$_{10}$NNa m/z 142.0394; Found 142.0394. [lit.$^4$] 
**DSC** Normalized thermal potential exotherm: 2015 J/g; Onset temperature: 193 °C.] 
*NMR spectra of 1 (HCl salt) have not been reported in CD$_3$OD. For spectra, see S16.

**methyl 3-(3-azidobicyclo[1.1.1]pentan-1-yl)propanoate (12).**

![Chemical Structure](image)

To a heterogeneous mixture of 1-azido-3-iodobicyclo[1.1.1]pentane (2) (500 mg, 2.13 mmol) in water (2.7 mL) in a quartz tube was added methyl acrylate (578.4 µL, 6.38 mmol), TTMSS (718 µL, 2.34 mmol), followed by AlBN (34.9 mg, 0.213 mmol). The reaction mixture was subjected to UV irradiation (254 nm) over 16 h at 4 °C (cold room). The reaction mixture was diluted with EtOAc, washed with saturated sodium thiosulphate, brine, dried over sodium sulphate and concentrated in vacuo. Rigorous purification was required and silica gel flash chromatography was performed 3 times (gradient elution from 0:100 to 4:96 with product eluting from 2:98 to 3:97 diethyl ether-hexanes) to afford 12 (130 mg, 31%) as a colorless oil.

**1**H NMR (400 MHz, CDCl$_3$) δ 3.67 (s, 3H), 2.30 (t, J = 7.5 Hz, 2H), 1.93 (t, J = 7.5 Hz, 2H), 1.84 (s, 6H) ppm. **13**C NMR (101 MHz, CDCl$_3$) δ 173.6, 52.3, 51.8, 51.1, 34.8, 31.7, 25.3 ppm. HRMS (ESI TOF, m/z): [M-N$_2$+H]$^+$ Calcd for C$_9$H$_{14}$NO$_2$ m/z 168.1019; Found 168.1012; [M-N$_2$+Na]$^+$ Calcd for C$_9$H$_{13}$NNaO$_2$ m/z 190.0838; Found 190.0839. IR: 2982, 2110, 1740, 1438, 1263, 1204, 1176, 1052, 841 cm$^{-1}$.

**References**


(4) Bunker, K. D.; Sach, N. W.; Huang, Q.; Richardson, P. F. *Org. Lett.* **2011**, 17, 4746
$^1$H NMR and $^{13}$C NMR Spectra

bicyclo[1.1.1]pentan-1-amine hydroiodide (1), $^1$H NMR (400 MHz, CD$_3$OD)

bicyclo[1.1.1]pentan-1-amine hydroiodide (1), $^{13}$C NMR (101 MHz, CD$_3$OD)
1-(3-iodobicyclo[1.1.1]pentan-1-yl)-4-phenyl-1H-1,2,3-triazole (7), $^1$H NMR (400 MHz, CDCl$_3$)

1-(3-iodobicyclo[1.1.1]pentan-1-yl)-4-phenyl-1H-1,2,3-triazole (7), $^{13}$C NMR (101 MHz, CDCl$_3$)
1-(3-iodobicyclo[1.1.1]pentan-1-yl)-4-phenyl-1H-1,2,3-triazole (7), $^1$H NMR (400 MHz, CD$_3$OD)

1-(3-iodobicyclo[1.1.1]pentan-1-yl)-4-phenyl-1H-1,2,3-triazole (7), $^{13}$C NMR (101 MHz, CD$_3$OD)
1-(bicyclo[1.1.1]pentan-1-yl)-4-phenyl-1H-1,2,3-triazole (8), $^1$H NMR (400 MHz, CDCl$_3$)

$^1$H NMR (400 MHz, CDCl$_3$)

1-(bicyclo[1.1.1]pentan-1-yl)-4-phenyl-1H-1,2,3-triazole (8), $^{13}$C NMR (101 MHz, CDCl$_3$)

$^{13}$C NMR (101 MHz, CDCl$_3$)
methyl 3-{3-(4-phenyl-1H-1,2,3-triazol-1-yl)bicyclo[1.1.1]pentan-1-yl}propanoate (9), $^1$H NMR (600 MHz, CDCl$_3$)

\[\text{\includegraphics{methyl_3-(3-(4-phenyl-1H-1,2,3-triazol-1-yl)bicyclo[1.1.1]pentan-1-yl)propanoate_(9)_1H_NMR_(600_MHz,_CDCl_3).png}}\]

methyl 3-{3-(4-phenyl-1H-1,2,3-triazol-1-yl)bicyclo[1.1.1]pentan-1-yl}propanoate (9), $^{13}$C NMR (151 MHz, CDCl$_3$)

\[\text{\includegraphics{methyl_3-(3-(4-phenyl-1H-1,2,3-triazol-1-yl)bicyclo[1.1.1]pentan-1-yl)propanoate_(9)_13C_NMR_(151_MHz,_CDCl_3).png}}\]
1-(bicyclo[1.1.1]pentan-1-yl)-4-ethoxy-1H-1,2,3-triazole (11), \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3})

![NMR spectrum of 1-(bicyclo[1.1.1]pentan-1-yl)-4-ethoxy-1H-1,2,3-triazole (11)]

1-(bicyclo[1.1.1]pentan-1-yl)-4-ethoxy-1H-1,2,3-triazole (11), \textsuperscript{13}C NMR (101 MHz, CDCl\textsubscript{3})

![NMR spectrum of 1-(bicyclo[1.1.1]pentan-1-yl)-4-ethoxy-1H-1,2,3-triazole (11)]
methyl 3-(3-azidobicyclo[1.1.1]pentan-1-yl)propanoate (12), $^1$H NMR (400 MHz, CDCl$_3$)

methyl 3-(3-azidobicyclo[1.1.1]pentan-1-yl)propanoate (12), $^{13}$C NMR (101 MHz, CDCl$_3$)
bicyclo[1.1.1]pentan-1-amine hydrochloride (1), $^1$H NMR (400 MHz, CD$_3$OD)

bicyclo[1.1.1]pentan-1-amine hydrochloride (1), $^{13}$C NMR (101 MHz, CD$_3$OD)