Supplementary Materials

One pot three-component cycloadditions of nonstabilized azomethine ylides to β -nitrostyrene, a highly electron deficient dipolarophile

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1. Materials and methods:

Column chromatography was performed on Merck silica gel (60-120 mesh, 0.120 mm-0.250 mm). Analytical TLC was performed on 0.25 mm Merck extra hard silica gel plates with UV254 fluorescent indicator. Melting points were recorded on an electrically heated Kfler Block apparatus. All melting points are uncorrected. ^1H -NMR and ^{13}C -NMR spectra were recorded at ambient temperature using Bruker Avance 300 spectrometer (300 MHz for ^1H and 75 MHz for ^{13}C). Chemical shifts are reported in δ , ppm with tetramethylsilane as internal reference, and coupling constants in Hz. Infrared spectra were recorded on Perkin Elmer Spectrum 100 FT-IR spectrometer in KBr pellets. EI-MS analysis was performed with a Perkin Elmer Clarus 600GC-MS instrument at 70eV using column Elite 5 MS (30 m x 0.25 mm x 0.25 μm) columns with maximum temperature 300 $^\circ\text{C}$. Single crystal X-ray diffraction studies of the crystalline heterocyclic compound were done with Xray Bruker Smart Apex-II diffractometer. Refinement was done by SHELXL-97 programme.

2. General procedure of Cycloaddition Reactions:

A solution of L-phenylglycine **1** (2 mmol) and arylaldehyde **2** (2 mmol) were heated along with dipolarophile, β -nitrostyrene **4** (2 mmol) at temperature 90 $^\circ\text{C}$ in dry DMF solvent. Azomethine ylides were derived from the reaction between arylaldehyde and α -amino acid *in situ* in the reaction medium. The heating was continued for few minutes depending on reactions keeping at constant temperature (details in Table no.1). The progress of reactions was monitored by thin-layer chromatography. After the period mentioned in Table no. **1** high boiling solvent DMF was removed by diluting reaction mixture with excess chloroform and washing with cold water. Following overnight drying with anhydrous sodium sulphate (Na_2SO_4), the chloroform was

removed by rotary evaporator under reduced pressure. The crude reaction mixture was resolved by column chromatography over silica gel using different percentages of ethyl acetate in petroleum ether (60-80 °C) as eluent.

Characterized data of compounds (1-11):

Compound **(1A)**: (2 α , 3 β , 4 α , 5 α)–3 β -nitro 2-(4-methoxyphenyl)-4,5-diphenylpyrrolidine; (C₂₃H₂₂N₂O₃); colourless crystal; isolated from 3% ethyl acetate in petroleum ether; m.p.: 112-114 °C; IR (KBr): 3326, 3030-2834, 1547, 1507, 1365, 1242, 1174, 1033, 698 cm⁻¹; ¹H-NMR (CDCl₃): δ 2.80-3.20 (1H, b, NH)^{*}, 4.90 (1H, d, J = 7.3 Hz, CH-NH), 5.15 (1H, dd, J = 7.3, 5.3 Hz, CHNO₂), 4.35 (1H, dd, J = 8.2, 5.3 Hz), 5.10 (1H, d, J = 8.2 Hz, CH-NH), 3.85 (3H, s, OMe), 6.95 (2H, d, J = 8.4 Hz), 7.00-7.02 (10H, m, Ar-H), 7.60 (2H, d, J = 8.2 Hz); ¹³C-NMR (CDCl₃): δ 55.22, 55.35, 65.32, 66.40, 97.10, 114.37, 127.00, 127.11, 127.33, 127.77, 127.98, 128.06, 128.97, 159.95. EI-MS: m/z 374 [M]⁺; Anal. calcd for (C₂₃H₂₂N₂O₃): C, 73.78; H, 5.92; N, 7.48%. Found: C, 73.63; H, 5.98; N, 7.52%.

Compound **(2C)**: (2 α , 3 α , 4 β , 5 α)–3 α -nitro 5-(4-chlorophenyl)-2,4-diphenylpyrrolidine; (C₂₂H₁₉ClN₂O₂); colourless solid; isolated from 20% ethyl acetate in petroleum ether; m.p.: 198-190 °C; IR (KBr): 3370, 3031-2841, 1551, 1371, 1087, 827, 697 cm⁻¹; ¹H-NMR (CDCl₃): δ 2.50-2.75 (1H, b, NH), 5.00 (1H, d, J = 7.7 Hz, CH-NH), 5.40 (1H, dd, J = 7.7, 5.3 Hz, CHNO₂), 4.00 (1H, dd, J = 9.1, 5.3 Hz), 4.35 (1H, d, J = 9.2 Hz), 7.10 (2H, d, J = 7.6 Hz), 7.20-7.40 (10H,

*NH-proton replaced by DCl, present in experimentally used CDCl₃ that help us to eliminate complexity generated by the coupling of NH-proton with near by protons. For the same reason NH-peak is very weak in nature and broad.

m, Ar-H), 7.45 (2H, d, $J = 7.7$ Hz); ^{13}C -NMR (CDCl_3): δ 58.47, 66.50, 69.03, 96.96, 127.10, 127.82, 127.95, 128.60, 128.84, 129.13, 134.01, 135.33, 137.72; EIMS: m/z 378 $[\text{M}]^+$, 380 $[\text{M}+2]^+$; Anal. calcd for ($\text{C}_{22}\text{H}_{19}\text{ClNO}_2$): C, 69.75; H, 5.05; N, 7.39%. Found: C, 69.58; H, 5.01; N, 7.42%.

Compound (**2D**): (2α , 3β , 4α , 5α)- 3α -nitro-5-(4-chlorophenyl)-2,4-diphenylpyrrolidine; ($\text{C}_{22}\text{H}_{19}\text{ClN}_2\text{O}_2$); colourless solid; isolated from 10% ethyl acetate in petroleum ether; m.p.: 162-164 °C; IR (KBr): 3378, 3012-2842, 1565, 1368, 1062, 832 cm^{-1} ; ^1H -NMR (CDCl_3): δ 2.50-2.70 (1H, b, NH), 4.95 (1H, d, $J = 7.8$ Hz, CH-NH), 5.38 (1H, dd, $J = 7.7, 5.4$ Hz, CHNO_2), 3.95 (1H, dd, $J = 7.9, 5.3$ Hz), 4.37 (1H, d, $J = 8.2$ Hz), 7.05 (2H, d, $J = 7.8$ Hz), 7.18-7.50 (12H, m, Ar-H); ^{13}C -NMR (CDCl_3): δ 58.65, 66.57, 69.11, 97.13, 127.12, 127.25, 127.84, 127.89, 128.24, 128.54, 128.61, 128.75, 129.04, 133.87, 135.68, 138.20; EIMS: m/z 378 $[\text{M}]^+$, 380 $[\text{M}]^+$; Anal. calcd for ($\text{C}_{22}\text{H}_{19}\text{ClNO}_2$): C, 69.75; H, 5.05; N, 7.39%. Found: C, 69.84; H, 5.08; N, 7.12%.

Compound-(**3B**): (2α , 3α , 4β , 5α)- 3α -nitro-2, 4, 5-triphenylpyrrolidine; ($\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}_2$); colourless solid; isolated from 3% ethyl acetate in petroleum ether ; m.p.: 118-120 °C; IR (KBr): 3323, 3029-2841, 1153, 1493, 1453, 1372, 757, 697 cm^{-1} ; ^1H -NMR (CDCl_3): δ 2.40-2.80 (1H, b, -NH), 5.00 (1H, d, $J = 7.9$ Hz, CH-NH), 5.43 (1H, dd, $J = 7.6, 5.2$ Hz, CHNO_2), 4.05 (1H, dd, $J = 9.1, 5.2$ Hz), 4.40 (1H, d, $J = 9.1$ Hz, CH-NH), 7.10-7.50 (15H, m, Ar-H). ^{13}C -NMR (CDCl_3): δ 58.65, 66.74, 69.86, 97.36, 127.04, 127.35, 127.82, 128.29, 128.59, 128.75, 128.75, 129.07, 135.44, 138.17, 139.17; EIMS: m/z 344 $[\text{M}]^+$; Anal. calcd for ($\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}_2$): C, 76.72; H, 5.85; N, 8.13%. Found: C, 76.69; H, 5.89; N, 8.08%.

Compound (**4C**): (2 α , 3 α , 4 β , 5 α)–3 α -nitro 5-(*m*-nitrophenyl)-2,4-diphenylpyrrolidine; (C₂₂H₁₉N₃O₄); yellow crystal; isolated from 20% ethyl acetate in petroleum ether ; m.p.: 132-134 °C; IR (KBr): 3370, 3083-2853, 1150, 1527, 1350, 1090, 752, 696 cm⁻¹; ¹H-NMR (CDCl₃): δ 2.60-3.10 (1H, b, NH), 5.09 (1H, d, *J* = 8.0, CH-NH), 5.45 (1H, dd, *J* = 8.0, 5.5, CHNO₂), 4.07 (1H, dd, *J* = 9.1, 5.6), 4.54 (1H, d, *J* = 9.1 CH-NH), 7.10-7.20 (2H, m, Ar-H), 7.30-7.45 (7H, m, Ar-H), 7.50-7.60 (2H, m, Ar-H), 7.82 (1H, d, *J* = 7.7), 8.18 (1H, dd, *J* = 7.6, 3.4), 8.30 (1H, s); ¹³C-NMR (CDCl₃): δ 58.29, 66.29, 68.52, 96.71, 122.22, 123.23, 127.24, 127.86, 128.23, 128.62, 128.97, 129.31, 129.63, 133.41, 135.53, 137.20, 142.16, 148.41; EIMS: *m/z* 389 [M]⁺; Anal. calcd for (C₂₂H₁₉N₃O₄): C, 67.86; H, 4.92; N, 10.79%. Found: C, 67.88; H, 4.79; N, 10.82%.

Compound (**5C**): (2 α , 3 α , 4 β , 5 α)–3 α -nitro 5-(*p*-nitrophenyl)-2,4-diphenylpyrrolidine; (C₂₂H₁₉N₃O₄); colourless solid; isolated from 20% ethyl acetate in petroleum ether; m.p.: 106-108 °C; IR (KBr): 3346, 3017-2848, 1536, 1512, 1378, 1202, 1148, 1032, 623 cm⁻¹; ¹H-NMR (CDCl₃): δ 2.50-2.70 (1H, b, NH), 5.10 (1H, d, *J* = 8.5 Hz, CH-NH), 5.53 (1H, dd, *J* = 8.1, 6.1 Hz, CHNO₂), 4.10 (1H, dd, *J* = 7.8, 5.9 Hz), 4.45 (1H, d, *J* = 8.3 Hz, CH-NH), 7.14 (2H, dd, *J*=7.5, 2.1 Hz), 7.30-7.42 (6H, m, Ar-H), 7.56 (2H, dd, *J*=7.2, 2.1 Hz), 7.62 (2H, d, *J* = 8.7 Hz), 8.19 (2H, d, *J* = 8.8 Hz); ¹³C-NMR (CDCl₃): δ 58.45, 66.35, 68.65, 96.74, 123.75, 127.21, 127.83, 127.97, 128.55, 128.76, 128.92, 129.28, 137.30, 137.38, 148.76; EIMS: *m/z* 389 [M]⁺; Anal. calcd for (C₂₂H₁₉N₃O₄): C, 67.86; H, 4.92; N, 10.79%. Found: C, 67.68; H, 4.56; N, 10.98%.

Compound-(**5D**): (2 α , 3 β , 4 α , 5 α)–3 β -nitro 5-(*p*-nitrophenyl)-2,4-diphenylpyrrolidine; (C₂₂H₁₉N₃O₄); colourless crystal; isolated from 30% ethyl acetate in petroleum ether; m.p.: 128-

130 °C; IR (KBr): 3334, 3015-2832, 1542, 1528, 1368, 1214, 1141, 1016, 608 cm^{-1} ; ^1H -NMR (CDCl_3): δ 2.50-2.85 (1H, b, NH), 5.10 (1H, d, J = 8.7 Hz, CH-NH), 5.45 (1H, dd, J = 7.8, 5.6 Hz, CHNO_2), 4.04 (1H, dd, J = 7.8, 5.9 Hz), 4.52 (1H, d, J = 9.1 Hz, CH-NH), 7.15 (2H, dd, J = 7.6, 1.8 Hz), 7.30-7.42 (6H, m, Ar-H), 7.47 (2H, dd, J = 7.3, 2.1 Hz), 7.73 (2H, d, J = 8.7 Hz), 8.22 (2H, d, J = 8.8 Hz); ^{13}C -NMR (CDCl_3): δ 58.26, 65.05, 69.30, 96.80, 123.58, 127.15, 127.87, 127.92, 128.15, 128.29, 128.64, 129.05, 129.23, 135.76, 139.19, 148.02; EIMS: m/z 389 $[\text{M}]^+$; Anal. calcd for ($\text{C}_{22}\text{H}_{19}\text{N}_3\text{O}_4$): C, 67.86; H, 4.92; N, 10.79%. Found: C, 67.72; H, 4.90; N, 10.92%.

Compound (**6B**): (2 α , 3 α , 4 β , 5 α)-3 α -nitro-2-(*m*-bromophenyl)-4,5-diphenyl pyrrolidine; ($\text{C}_{22}\text{H}_{19}\text{BrN}_2\text{O}_2$); colourless solid ; isolated from petroleum ether ; m.p.: 122-124 °C; IR (KBr): 3324, 3029-2821, 1550, 1482, 1371, 1328, 1263, 1079, 751, 696 cm^{-1} ; ^1H -NMR (CDCl_3): δ 2.20-2.60 (1H, b, NH), 4.95 (1H, d, J = 7.7 Hz, CH-NH), 5.35 (1H, dd, J = 8.0, 5.3 Hz, CHNO_2), 4.00 (1H, dd, J = 9.0, 5.3 Hz), 4.30 (1H, d, J = 9.1 Hz, CH-NH), 7.05-7.45 (13H, m, Ar-H), 7.60 (1H, s, Ar-H); ^{13}C -NMR (CDCl_3): δ 58.47, 66.54, 69.07, 97.09, 118.12, 122.74, 126.00, 127.12, 127.25, 127.83, 127.93, 128.56, 128.79, 129.13, 130.21, 131.30, 135.61, 137.79, 142.04; EIMS: m/z 422 $[\text{M}]^+$, 224 $[\text{M}+2]^+$; Anal. calcd for ($\text{C}_{22}\text{H}_{19}\text{BrN}_2\text{O}_2$): C, 62.42; H, 4.52; N, 6.62%. Found: C, 61.84; H, 4.37; N, 6.94%.

Compound (**7B**): (2 α , 3 α , 4 β , 5 α)-3 α -nitro-2 α -(*o*-methoxyphenyl)-4, 5-diphenyl pyrrolidine; ($\text{C}_{23}\text{H}_{22}\text{N}_2\text{O}_3$); colourless solid; isolated from 3% ethyl acetate in petroleum ether ; m.p.: 128-130 °C; IR (KBr): 3360, 3072-2842, 1550, 1442, 1380, 1238, 1018, 750, 698 cm^{-1} ; ^1H -NMR (CDCl_3): δ 3.10-3.33 (1H, b, NH), 5.05 (1H, d, J = 7.6 Hz, CH-NH), 5.55 (1H, dd, J = 7.6, 5.4

Hz, CHNO₂), 4.35 (1H, dd, $J = 9.3, 5.4$ Hz), 4.85 (1H, d, $J = 5.4$ Hz, CH-NH), 3.73 (3H, s, OMe), 6.92 (1H, d, $J = 8.2$ Hz), 7.05 (1H, t, $J = 7.5$ Hz), 7.22-7.46 (9H, m, Ar-H), 7.53 (2H, d, $J = 7.6$ Hz), 7.64 (1H, d, $J = 7.6$ Hz); ¹³C-NMR (CDCl₃): δ 55.26, 57.53, 64.59, 67.07, 98.06, 110.89, 121.04, 126.78, 126.92, 127.4, 127.65, 128.41, 128.46, 128.60, 128.70, 129.07, 135.86, 138.74, 157.53; EIMS: m/z 374 [M]⁺; Anal. calcd for (C₂₃H₂₂N₂O₃): C, 73.78; H, 5.92; N, 7.48%. Found: C, 73.69; H, 5.99; N, 7.42%.

Compound (**8C**): (2 α , 3 α , 4 β , 5 α)-3 α -nitro 5-(*p*-methylphenyl)-2,4-diphenylpyrrolidine; (C₂₃H₂₂N₂O₂); colourless solid; isolated from 5% ethyl acetate in petroleum ether; m.p.: 134-136 °C; IR (KBr): 3320, 3028-2854, 1552, 1370, 1081, 698 cm⁻¹; ¹H-NMR (CDCl₃): δ 2.50-2.75 (1H, b, NH), 5.00 (1H, d, $J = 7.8$ Hz, CH-NH), 5.45 (1H, dd, $J = 8.8, 5.6$ Hz, CHNO₂), 4.08 (1H, dd, $J = 8.1, 5.3$ Hz), 4.40 (1H, d, $J = 9.3$ Hz, CH-NH), 2.17 (3H, s, CH₃), 7.12-7.53 (14H, m, Ar-H); ¹³C-NMR (CDCl₃): δ 21.12, 58.68, 66.77, 69.75, 97.58, 126.89, 127.02, 127.29, 127.57, 127.66, 128.14, 128.63, 129.34, 132.65, 136.40, 138.42, 139.56; EIMS: m/z 358 [M]⁺; Anal. calcd for (C₂₃H₂₂N₂O₂): C, 77.07; H, 6.19; N, 7.82%. Found: C, 76.89; H, 6.28; N, 7.87%.

Compound (**8D**): (2 α , 3 β , 4 α , 5 α)-3 β -nitro 5-(*p*-methylphenyl)-2,4-diphenylpyrrolidine; (C₂₃H₂₂N₂O₂); colourless solid; isolated from 3% ethyl acetate in petroleum ether; m.p.: 158-160 °C; IR (KBr): 3367, 3008-2862, 1545, 1378, 1085, 755, 685 cm⁻¹; ¹H-NMR (CDCl₃): δ 2.60-2.85 (1H, b, NH), 5.03 (1H, d, $J = 7.9$ Hz, CH-NH), 5.47 (1H, dd, $J = 7.8, 5.5$ Hz, CHNO₂), 4.12 (1H, dd, $J = 7.9, 5.4$ Hz), 4.43 (1H, d, $J = 8.3$ Hz, CH-NH), 2.35 (3H, s, CH₃), 7.14-7.50 (14H, m, Ar-H); ¹³C-NMR (CDCl₃): δ 30.86, 58.42, 66.84, 69.97, 97.32, 126.82, 127.08, 127.32,

127.76, 127.83, 127.98, 128.32, 128.56, 129.38, 135.72, 138.46, 139.57; EIMS: m/z 358 $[M]^+$; Anal. calcd for ($C_{23}H_{22}N_2O_2$): C, 77.07; H, 6.19; N, 7.82%. Found: C, 76.95; H, 6.02; N, 7.75%.

Compound-(**9A**): (2β , 3α , 4β , 5β)- 3α -nitro 2-(4-methoxyphenyl)-4-(4-chlorophenyl)-5-phenylpyrrolidine; ($C_{23}H_{21}ClN_2O_3$); colourless crystal; isolated from 3% Ethyl Acetate in Petroleum Ether; m.p.: 146-148 °C; IR (KBr): 3323, 3034-2801, 1547, 1504, 1327, 1250, 828,706 cm^{-1} ; 1H -NMR ($CDCl_3$): δ 2.30-2.90 (1H, b, NH), 4.87 (1H, d, $J = 7.2$ Hz, CH-NH), 5.00 (1H, dd, $J = 7.2, 5.0$ Hz, $CHNO_2$), 4.14 (1H, dd, $J = 8.1, 4.9$ Hz), 5.08 (1H, d, $J = 8.2$ Hz, CH-NH), 3.82 (3H, s, OMe), 6.90 (2H, d, $J = 8.4$ Hz), 7.98-7.18 (9H, m, Ar-H), 7.57 (2H, d, $J = 8.5$ Hz); ^{13}C -NMR ($CDCl_3$): δ 54.56, 55.34, 65.09, 66.29, 97.21, 114.38, 127.28, 127.94, 128.12, 130.26, 130.88, 132.90, 136.62, 138.54, 159.95; EI-MS: m/z 408 $[M]^+$, 410 $[M+2]^+$; Anal. calcd for ($C_{23}H_{21}ClN_2O_3$): C, 67.56; H, 5.18; N, 6.85% Found: C, 67.50; H, 5.12; N, 6.89%.

Compound (**10C**): (2α , 3α , 4β , 5α)- 3α -nitro 4,5-di-(4-chlorophenyl)-2-phenylpyrrolidine; ($C_{22}H_{18}Cl_2N_2O_2$); colourless solid; isolated from 10% ethyl acetate in petroleum ether; m.p.: 156-158 °C; IR (KBr): 3358, 3034-2812, 1552, 1374, 1073, 685 cm^{-1} ; 1H -NMR ($CDCl_3$): δ 2.60-2.90 (1H, b, NH), 4.93 (1H, d, $J = 7.8$ Hz, CH-NH), 5.34 (1H, dd, $J = 7.8, 5.8$ Hz, $CHNO_2$), 4.02 (1H, dd, $J = 8.1, 5.9$ Hz), 4.26 (1H, d, $J = 8.3$ Hz), 7.05 (2H, d, $J = 8.3$ Hz), 7.20-7.45 (11H, m, Ar-H), ^{13}C -NMR ($CDCl_3$): δ 57.96, 65.57, 69.01, 96.71, 127.12, 127.20, 127.56, 127.84, 128.32, 128.60, 128.79, 129.20, 129.25, 129.32; EIMS: m/z 412 $[M]^+$, 414 $[M+2]^+$, 416 $[M+4]^+$; Anal. calcd for ($C_{22}H_{18}Cl_2N_2O_2$): C, 63.93; H, 4.39; N, 6.78%. Found: C, 63.98; H, 4.37; N, 6.79%.

Compound (**11C**): (2 α , 3 α , 4 β , 5 α)–3 α -nitro-4-(4-chlorophenyl)-5-(4-methylphenyl)-2-phenylpyrrolidine; (C₂₃H₂₁ClN₂O₂); colourless solid; isolated from 3% ethyl acetate in petroleum ether; m.p.: 124-126 °C; IR (KBr): 3333, 3030-2863, 1554, 1366, 1093, 816, 701, 516 cm⁻¹; ¹H-NMR (CDCl₃): δ 2.60-2.90 (1H, b, NH), 5.00 (1H, d, J = 7.5 Hz, CH-NH), 5.40 (1H, dd, J = 7.3, 6.0 Hz, CHNO₂), 4.05 (1H, dd, J = 8.8, 5.9 Hz), 4.35 (1H, d, J = 9.0 Hz), 2.34 (3H, s, CH₃), 7.09 (2H, d, J = 8.0 Hz), 7.18 (2H, d, J = 7.4 Hz), 7.25-7.45 (9H, m, Ar-H), ¹³C-NMR (CDCl₃): δ 21.18, 58.10, 66.50, 69.84, 97.18, 126.74, 126.96, 127.09, 127.14, 128.14, 128.32, 128.55, 128.72, 129.18, 132.60, 138.56, 139.21; EI-MS: m/z 392 [M]⁺, 394 [M+2]⁺; Anal. calcd for (C₂₃H₂₁ClN₂O₂): C, 70.31; H, 5.39; N, 7.13%. Found: C, 70.34; H, 5.43; N, 7.11%.

Expansion

¹H-NMR of Compound 1A

Chemical structure of Compound 1A is shown, featuring a 5-membered ring with a nitro group (NO₂), a methoxy group (OMe), and two phenyl rings.

4. Summary of crystal data:

Figure S1: Crystal structure of cycloadduct **1A**

Figure S2: Crystal structure of cycloadduct **4C**

Figure S3: Crystal structure of cycloadduct **7B**

Figure S4: Crystal structure of cycloadduct **9A**

5. Crystal data for cycloadduct 1A, 4C, 7B and 9A

	Cycloadduct-1A	Cycloadduct-4C	Cycloadduct-7B	Cycloadduct-9A
Formula	C ₂₃ H ₂₂ N ₂ O ₃	C ₂₂ H ₁₉ N ₃ O ₄	C ₂₃ H ₂₂ N ₂ O ₃	C ₂₃ H ₂₁ ClN ₂ O ₃
Formula Weight	374.27	389.40	374.43	408.87
Crystal System	Triclinic	Orthorhombic	Triclinic	Monoclinic
Space group	P-1	Pca21	P-1	P21/c
a [Å]	8.906(3)	19.831(2)	9.3000(18)	11.401(4)
b [Å]	10.567(3)	7.1756(8)	9.884(2)	12.039(4)
c [Å]	10.870(3)	14.0051(17)	11.521(2)	15.249(5)
α [°]	80.300(9)	1992.9(4)	67.956(6)	90.000
β [°]	83.224(10)	90.000	84.343(6)	94.020(11)
γ [°]	76.637(9)	90.000	81.395(6)	90.000
V [Å ³]	977.7(5)	90.000	969.5(3)	2087.9(12)
Z	2	4	2	4
D(calc) [g/cm ⁻³]	1.271	1.298	1.283	1.301
Absorption coefficient μ [mm ⁻¹]	0.085	0.091	0.086	0.209
Absorption correction	Multi-scan	Multi-scan	Multi-scan	Multi-scan
F(000)	396	816	396	856
Crystal Size [mm]	0.67 x 0.76 x 0.87	0.45 x 0.54 x 0.65	0.45 x 0.56 x 0.67	0.54 x 0.65 x 0.65
Temperature (K)	296	296	296	296
Radiation [Angstrom]	MoK α (λ =0.71073)	MoK α (λ =0.71073)	MoK α (λ =0.71073)	MoK α (λ =0.71073)
Theta Min-Max [°]	1.9, 27.5	2.0, 20.8	1.9, 27.5	1.8, 22.9
Collection range	-11 $\leq h \leq$ 10; -13 $\leq k \leq$ 12; -13 $\leq l \leq$ 14	-19 $\leq h \leq$ 17; -6 $\leq k \leq$ 6; -12 $\leq l \leq$ 13	-12 $\leq h \leq$ 12; -12 $\leq k \leq$ 12; -14 $\leq l \leq$ 14	-12 $\leq h \leq$ 12; -13 $\leq k \leq$ 13; -16 $\leq l \leq$ 16
Reflections collected	12795	5578	12732	19119
Independent reflections (R_{int})	4302, (0.021)	1749, (0.026)	4261, (0.024)	2844, (0.034)
R, wR ₂ , S	0.0475, 0.1186, 1.86	0.0396, 0.0578, 1.44	0.0512, 0.1005, 2.71	0.0412, 0.0892, 2.78