Supporting Information for:

**A New Multicomponent Reaction Catalyzed by a [Lewis Acid]⁺[Co(CO)₄]⁻ Catalyst:
Stereospecific Synthesis of 1,3-Oxazinane-2,4-diones from Epoxides, Isocyanates,
and CO**

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Purification and Characterization of New Compounds

ODs were synthesized according to the test-scale procedure given in the experimental section, and details of the purification and characterization of individual compounds are given below. NMR spectra were recorded on Varian Mercury 300 (¹H, 300 MHz; ¹³C, 75 MHz), Bruker ASX 300 (¹H, 300 MHz; ¹³C, 75 MHz), or Varian INOVA-400 (¹H, 400 MHz; ¹³C, 100 MHz) spectrometers and referenced versus residual

non-deuterated solvent shifts. All ^1H NMR spectra, and most ^{13}C NMR spectra, were recorded using compounds in CDCl_3 solution. Due to the low solubility of some compounds in CDCl_3 , a few ^{13}C NMR spectra were collected in $\text{DMSO}-d_6$ solution. In the characterization data below, pd is the abbreviation for a pseudo-doublet. HRMS analyses were performed using EI^+ ionization. Compounds **4ac'**¹ and **4af'**^{1,2} have been reported.

6-Ethyl-3-phenyl-1,3-oxazinane-2,4-dione (4aa'; Table 2, entry 1)

Crystallized from $\text{CH}_2\text{Cl}_2/\text{hexanes}$. ^1H NMR (CDCl_3 , 300 MHz) δ 1.12 (3 H, t, $^3J = 7.5$ Hz), 1.88 (2 H, m), 2.83 (1 H, dd, $^2J = 16.9$ Hz, $^3J = 11.1$ Hz), 2.93 (1 H, dd, $^2J = 16.9$ Hz, $^3J = 3.8$ Hz), 4.60 (1 H, m), 7.19 (2 H, m), 7.45 (3 H, m). ^{13}C NMR (CDCl_3 , 75 MHz) δ 9.2, 27.6, 36.7, 75.8, 128.4, 129.2, 129.6, 151.7, 168.4. HRMS (EI^+) m/z calcd ($\text{C}_{12}\text{H}_{13}\text{NO}_3$) 219.08955, found 219.09088.

6-Ethyl-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4ab'; Table 2, entry 2)

Crystallized from $\text{CH}_2\text{Cl}_2/\text{hexanes}$. ^1H NMR (CDCl_3 , 300 MHz) δ 1.13 (3H, t, $^3J = 7.5$ Hz), 1.90 (2 H, m), 2.87 (1 H, dd, $^2J = 17.3$ Hz, $^3J = 11.0$ Hz), 2.96 (1 H, dd, $^2J = 17.3$ Hz, $^3J = 3.6$ Hz), 4.58 (1 H, m), 7.40 (2 H, pd), 8.34 (2 H, pd). ^{13}C NMR (CDCl_3 , 75 MHz) δ 9.1, 27.4, 36.5, 76.1, 124.7, 129.8, 140.3, 147.8, 150.7, 167.8. HRMS (EI^+) m/z calcd ($\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}_5$) 264.07462, found 264.07468.

3-(4-Bromophenyl)-6-ethyl-1,3-oxazinane-2,4-dione (4ad'; Table 2, entry 4)

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 1.11 (3 H, t, ³J = 7.4 Hz), 1.87 (2 H, m), 2.83 (1 H, dd, ²J = 16.9 Hz, ³J = 11.1 Hz), 2.93 (1 H, dd, ²J = 16.9 Hz, ³J = 3.5 Hz), 4.60 (1 H, m), 7.07 (2 H, pd), 7.60 (2 H, pd). ¹³C NMR (DMSO-*d*₆, 75 MHz) δ 8.9, 26.5, 35.6, 75.3, 121.4, 131.1, 131.9, 135.2, 151.1, 168.7. HRMS (EI+) *m/z* calcd (C₁₂H₁₂NO₃⁷⁹Br) 297.00005, found 297.00099; calcd (C₁₂H₁₂NO₃⁸¹Br) 298.99801, found 298.99847.

6-Ethyl-3-(4-methoxyphenyl)-1,3-oxazinane-2,4-dione (4ae'; Table 2, entry 5)

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 1.11 (3 H, t, ³J = 7.5 Hz), 1.87 (2 H, m), 2.82 (1 H, dd, ²J = 17.1 Hz, ³J = 11.3 Hz), 2.92 (1 H, dd, ²J = 17.1 Hz, ³J = 3.6 Hz), 3.83 (3 H, s), 4.58 (1 H, m), 6.97 (2 H, pd), 7.10 (2 H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ 9.2, 27.6, 36.7, 55.7, 75.8, 114.9, 127.3, 129.4, 159.9, 168.7. HRMS (EI+) *m/z* calcd (C₁₃H₁₅NO₄) 249.10011, found 249.09912.

3-Benzyl-6-ethyl-1,3-oxazinane-2,4-dione (4ag'; Table 2, entry 7)

Purified by column chromatography on silica gel (28% EtOAc/hexanes, R_f = 0.30). ¹H NMR (CDCl₃, 300 MHz) δ 1.03 (3 H, t, ³J = 7.7 Hz), 1.73 (2 H, m), 2.64 (1 H, dd, ²J = 16.9 Hz, ³J = 11.4 Hz), 2.77 (1 H, dd, ²J = 16.9 Hz, ³J = 3.5 Hz), 4.36 (1 H, dddd, ³J = 11.4 Hz, 6.9 Hz, 5.6 Hz, 3.5 Hz), 4.93 (1 H, d, ²J = 14.1 Hz), 4.99 (1 H, d, ²J = 14.1 Hz), 7.30 (3 H, m), 7.41 (2 H, m). ¹³C NMR (CDCl₃, 75 MHz) δ 9.1, 27.5, 36.5, 45.2, 75.3, 128.0, 128.7, 129.1, 136.7, 151.9, 168.2. HRMS (EI+) *m/z* calcd (C₁₃H₁₅NO₃) 233.10520, found 233.10431.

3,6-Diethyl-1,3-oxazinane-2,4-dione (4ah'; Table 2, entry 8)

Filtered through silica in EtOAc to remove catalyst residue, then sublimed under vacuum to yield a mixture of **4ah'** and 1,3,5-triethylisocyanurate.³ ¹H NMR (CDCl₃, 300 MHz) δ 1.04 (3 H, t, ³J = 7.5 Hz), 1.19 (3 H, t, ³J = 7.1 Hz), 1.77 (2 H, m), 2.61 (1 H, dd, ²J = 17.1 Hz, ³J = 11.4 Hz), 2.74 (1 H, dd, ²J = 17.1 Hz, ³J = 3.6 Hz), 3.83 (2 H, m), 4.36 (1 H, m). ¹³C NMR (CDCl₃, 75 MHz) δ 9.1, 13.4, 27.5, 36.5, 37.5, 75.3, 151.8, 168.1.

6-Methyl-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4bb'; Table 3, entry 2)

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 1.60 (3 H, d, ³J = 6.3 Hz), 2.86 (1 H, dd, ²J = 17.1 Hz, ³J = 11.1 Hz), 2.99 (1 H, dd, ²J = 17.1 Hz, ³J = 3.6 Hz), 4.86 (1 H, m), 7.40 (2 H, pd), 8.34 (2 H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ 20.4, 38.8, 71.5, 124.7, 130.0, 140.6, 148.3, 150.5, 167.4. HRMS (EI+) *m/z* calcd (C₁₁H₁₀N₂O₅) 250.05897, found 250.06071.

6-Butyl-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4cb'; Table 3, entry 3)

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 0.96 (3 H, t, ³J = 7.1 Hz), 1.43 (4 H, m), 1.84 (2 H, m), 2.87 (1 H, dd, ²J = 17.1 Hz, ³J = 11.1 Hz), 2.97 (1 H, dd, ²J = 17.1 Hz, ³J = 3.6 Hz), 4.69 (1 H, m), 7.40 (2 H, pd), 8.34 (2 H, pd). ¹³C NMR (DMSO-*d*₆, 75 MHz) δ 14.5, 22.5, 26.9, 33.7, 36.6, 75.0, 124.8, 131.2, 142.4, 147.8, 151.5, 169.3. HRMS (EI+) *m/z* calcd (C₁₄H₁₆N₂O₅) 292.10592, found 292.10577.

6-Cyclohexyl-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4db'; Table 3, entry 4)

Filtered through silica in 33 % EtOAc/hexanes. ^1H NMR (CDCl_3 , 400 MHz) δ 1.24 (6 H, m), 1.76 (2 H, m), 1.86 (2 H, m), 2.02 (1 H, m), 2.88 (1 H, dd, $^2J = 17.2$ Hz, $^3J = 10.4$ Hz), 2.93 (1 H, dd, $^2J = 17.2$ Hz, $^3J = 5.2$ Hz) 4.46 (1 H, m), 7.40 (2 H, pd), 8.33 (2 H, pd). ^{13}C NMR (CDCl_3 , 75 MHz) δ 25.7, 25.8, 26.2, 28.1, 34.6, 79.0, 124.8, 129.9, 140.4, 147.9, 151.0, 168.3. HRMS (EI+) m/z calcd ($\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_5$) 318.1216, found 318.1220.

6-tert-Butyl-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4eb'; Table 3, entry 5)

Filtered through silica in 50 % EtOAc/hexanes, crystallized from CH_2Cl_2 /hexanes. ^1H NMR (CDCl_3 , 300 MHz) δ 1.10 (9 H, s), 2.88 (1 H, d, $^3J = 4.1$ Hz), 2.91 (1 H, s), 4.36 (1 H, m), 7.41 (2 H, pd), 8.33 (2 H, pd). ^{13}C NMR (CDCl_3 , 75 MHz) δ 25.3, 32.9, 34.0, 82.2, 124.8, 129.9, 140.4, 147.9, 151.0, 168.5. HRMS (EI+) m/z calcd ($\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_5$) 292.1059, found 292.1062.

6-(But-3-en-yl)-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4fb'; Table 3, entry 6)

Crystallized from CH_2Cl_2 /hexanes. ^1H NMR (CDCl_3 , 300 MHz) δ 1.86 (1 H, m), 2.03 (1 H, m) 2.34 (2 H, m), 2.88 (1 H, dd, $^2J = 17.1$ Hz, $^3J = 11.1$ Hz), 2.97 (1 H, dd, $^2J = 17.1$ Hz, $^3J = 3.9$ Hz), 4.71 (1 H, m), 5.10 (1 H, dq, $^3J = 10.4$ Hz, $^4J = 1.5$ Hz), 5.16 (1 H, dq, $^3J = 17.0$ Hz, $^4J = 1.5$ Hz), 5.83 (1 H, ddt, $^3J = 17.0$ Hz, 10.4 Hz, 6.8 Hz), 7.41 (2 H, pd), 8.34 (2 H, pd). ^{13}C NMR (CDCl_3 , 75 MHz) δ 28.8, 33.5, 37.0, 74.2, 116.9, 124.8, 129.9, 136.2, 140.3, 148.0, 150.7, 167.7. HRMS (EI+) m/z calcd ($\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_5$) 290.09027, found 290.09003.

6-(Allyloxymethyl)-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4gb'; Table 3, entry 7)

Purified by column chromatography on silica gel (50 % EtOAc/hexanes, R_f = 0.19). ^1H NMR (CDCl_3 , 300 MHz) δ 3.09 (2 H, m), 3.70 (1 H, dd, 2J = 11.0 Hz, 3J = 3.0 Hz), 3.94 (1 H, dd, 2J = 11.0 Hz, 3J = 3.0 Hz), 4.10 (2 H, m), 4.82 (1 H, m), 5.27 (1 H, dd, 2J = 10.5 Hz, 3J = 1.0 Hz), 5.31 (1H, dd, 2J = 17.1 Hz, 3J = 1.3 Hz), 5.89 (1 H, m), 7.39 (2 H, pd), 8.32 (2H, pd). ^{13}C NMR (CDCl_3 , 75 MHz) δ 33.5, 71.1, 73.0, 73.6, 118.9, 124.7, 129.9, 133.6, 140.5, 147.9, 150.6, 167.4. HRMS (EI+) m/z calcd ($\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_6$) 306.0852, found 306.0853.

6-Butoxymethyl-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4hb'; Table 3, entry 8)

Crystallized from CH_2Cl_2 /hexanes. ^1H NMR (CDCl_3 , 300 MHz) δ 0.92 (3 H, t, 3J = 7.2 Hz), 1.36 (2 H, m), 1.58 (2 H, m), 3.04 (1 H, dd, 2J = 17.2 Hz, 3J = 6.8 Hz), 3.10 (1 H, dd, 2J = 17.2 Hz, 3J = 5.7 Hz), 3.52 (1 H, dt, 2J = 9.6 Hz, 3J = 6.6 Hz), 3.57 (1 H, dt, 2J = 9.6 Hz, 3J = 6.6 Hz), 3.66 (1 H, dd, 2J = 10.9 Hz, 3J = 3.2 Hz), 3.92 (1 H, dd, 2J = 10.9 Hz, 3J = 3.0 Hz), 4.81 (1 H, m), 7.39 (2 H, pd), 8.33 (2 H, pd). ^{13}C NMR (CDCl_3 , 75 MHz) δ 14.1, 19.3, 31.8, 33.5, 71.9, 72.3, 73.6, 124.7, 129.8, 140.5, 147.9, 150.7, 167.4. HRMS (EI+) m/z calcd ($\text{C}_{15}\text{H}_{18}\text{N}_2\text{O}_6$) 322.1165, found 322.1167.

6-(Benzyloxymethyl)-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4ib'; Table 3, entry 9)

Crystallized from CH_2Cl_2 /hexanes. ^1H NMR (CDCl_3 , 300 MHz) δ 3.03 (1 H, dd, 2J = 17.2 Hz, 3J = 5.3 Hz), 3.15 (1 H, dd, 2J = 17.2 Hz, 3J = 6.3 Hz), 3.74 (1 H, dd, 2J = 10.8 Hz, 3J = 3.0 Hz), 4.03 (1 H, dd, 2J = 10.8 Hz, 3J = 2.4 Hz), 4.59 (3 H, s), 4.83 (1 H, dd, 3J

= 6.3, 5.3, 3.0, 2.4 Hz), 7.06 (2 H, pd), 7.35 (5 H, br m), 8.16 (2 H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ 33.5, 71.8, 73.4, 74.5, 124.6, 128.7, 129.0, 129.8, 136.8, 140.4, 147.8, 150.6, 167.2. HRMS (EI+) *m/z* calcd (C₁₈H₁₆N₂O₆) 356.1008, found 356.1014.

6-(*tert*-Butyldimethylsiloxyethyl)-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4jb'; Table 3, entry 10)

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 0.12 (3 H, s), 0.13 (3H, s), 0.92 (9 H, s), 3.01 (1 H, dd, ²*J* = 17.4 Hz, ³*J* = 5.1 Hz), 3.08 (1 H, dd, ²*J* = 17.4 Hz, ³*J* = 8.9 Hz), 3.88 (1 H, dd, ²*J* = 11.4 Hz, ³*J* = 8.1 Hz), 4.06 (1 H, dd, ²*J* = 11.4 Hz, ³*J* = 3.3 Hz), 4.73 (1 H, m), 7.41 (2 H, pd), 8.33 (2 H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ -5.24, -5.19, 18.7, 26.0, 33.3, 64.6, 74.8, 124.7, 129.9, 140.4, 147.4, 150.6, 167.8. HRMS (EI+): An M⁺ peak for this compound could not be detected. The major ion peak had a mass consistent with (M⁺ – 57), which corresponds to the loss of the ^tBu group. This fragmentation is favored by the formation of a relatively stable, branched ^tBu radical, and by the presence of an oxygen atom that can stabilize the silyl ether cation.⁴ *m/z* calcd (M⁺ – 57) = *m/z* calcd (C₁₃H₁₅N₂O₆Si) 323.0699, found 323.0688.

6-Chloromethyl-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (4kb'; Table 3, entry 11)

Crystallized from EtOAc. ¹H NMR (CDCl₃, 300 MHz) δ 3.11 (1 H, dd, ²*J* = 17.1 Hz, ³*J* = 4.7 Hz), 3.16 (1 H, dd, ²*J* = 17.1 Hz, ³*J* = 10.1 Hz), 3.85 (1 H, dd, ²*J* = 12.2 Hz, ³*J* = 4.2 Hz), 3.92 (1 H, dd, ²*J* = 12.2 Hz, ³*J* = 5.0 Hz), 4.97 (1 H, m), 7.41 (2 H, pd), 8.35 (2 H, pd). ¹³C NMR (DMSO-*d*₆, 75 MHz) δ 33.4, 45.8, 73.3, 124.2, 130.5, 141.5, 147.2, 150.3, 167.8. HRMS (EI+) *m/z* calcd (C₁₁H₉N₂O₅Cl) 284.02000, found 284.02031.

Propyl 3-(3-(4-nitrophenyl)-2,4-dioxo-1,3-oxazinan-6-yl) Propanoate (4lb'; Table 3, entry 12)

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 0.96 (3 H, t, ³J = 7.2 Hz), 1.68 (2 H, qt, ³J = 7.2 Hz, 6.9 Hz), 2.14 (2 H, td, ³J = 7.0 Hz, 6.4 Hz), 2.64 (2 H, m), 2.88 (1 H, dd, ²J = 17.3 Hz, ³J = 11.3 Hz), 3.01 (1 H, dd, ²J = 17.3 Hz, ³J = 3.5 Hz), 4.08 (2 H, t, ³J = 6.9 Hz), 4.83 (1 H, dtd, ³J = 11.3 Hz, 6.4 Hz, 3.5 Hz), 7.41 (2 H, pd), 8.34 (2 H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ 10.6, 22.1, 29.1, 29.5, 37.1, 66.8, 73.9, 124.8, 129.9, 140.2, 148.0, 150.5, 167.4, 172.6. HRMS (EI+) *m/z* calcd (C₁₆H₁₈N₂O₇) 350.11140, found 350.10969.

3-(3-(4-Nitrophenyl)-2,4-dioxo-1,3-oxazinan-6-yl)propyl Butanoate (4mb'; Table 3, entry 13)

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 0.96 (3H, t, ³J = 7.4 Hz), 1.69 (2H, sextet, ³J = 7.4 Hz), 1.92 (4H, br m), 2.31 (2H, t, ³J = 7.4 Hz), 2.89 (1H, dd, ²J = 17.1 Hz, ³J = 11.1 Hz), 2.97 (1H, dd, ²J = 17.1 Hz, ³J = 3.8 Hz), 4.18 (2 H, m), 4.74 (1 H, ddd, ³J = 11.1 Hz, 7.4 Hz, 3.8 Hz), 7.40 (2H, pd), 8.34 (2H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ 13.9, 18.7, 24.2, 31.1, 36.3, 37.1, 63.2, 74.4, 124.8, 129.9, 140.2, 148.0, 150.6, 167.5, 173.9. HRMS (EI+) *m/z* calcd (C₁₇H₂₀N₂O₇) 364.1271, found 364.1277.

***trans*-5,6-Dimethyl-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (*trans*-4pb'; Table 4, entry 1)**

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 1.34 (3 H, d, ³*J* = 7.0 Hz), 1.59 (3 H, d, ³*J* = 6.2 Hz), 2.76 (1 H, dq, ³*J* = 11.0, 7.0 Hz), 4.52 (1 H, dq, ³*J* = 11.0, 6.2 Hz), 7.39 (2 H, pd), 8.33 (2 H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ 11.3, 19.2, 42.2, 76.7, 124.8, 129.9, 140.7, 147.9, 150.6, 171.0. HRMS (EI+) *m/z* calcd (C₁₂H₁₂N₂O₅) 264.07462, found 264.07419.

***cis*-5,6-Dimethyl-3-(4-nitrophenyl)-1,3-oxazinane-2,4-dione (*cis*-4pb'; Table 4, entry 2)**

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 1.36 (3 H, d, ³*J* = 7.2 Hz), 1.51 (3 H, d, ³*J* = 6.6 Hz), 3.05 (1 H, qd, ³*J* = 7.2, 4.1 Hz), 4.87 (1 H, qd, ³*J* = 6.6, 4.1 Hz), 7.38 (2 H, pd), 8.33 (2 H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ 10.0, 15.9, 40.3, 74.5, 124.8, 129.9, 140.5, 147.9, 150.5, 171.4. HRMS (EI+) *m/z* calcd (C₁₂H₁₂N₂O₅) 264.07462, found 264.07460.

***trans*-3-(4-Nitrophenyl)tetrahydrocyclopenta[*e*][1,3]oxazine-2,4(3*H*,4*aH*)-dione (*trans*-4qb'; Table 4, entry 3)**

Crystallized from EtOAc. ¹H NMR (CDCl₃, 400 MHz) δ 1.77 – 2.02 (3 H, m), 2.07 (1 H, m), 2.20 (1 H, m), 2.32 (1 H, m), 2.93 (1 H, ddd ³*J* = 12.3, 11.0, 8.2 Hz), 4.58 (1 H, ddd, ³*J* = 12.3, 10.1, 6.9), 7.39 (2 H, pd), 8.33 (2 H, pd). ¹³C NMR (DMSO-*d*₆, 75 MHz) δ 18.7, 19.0, 27.3, 45.2, 77.7, 123.9, 130.3, 142.1, 146.8, 151.7, 170.3. HRMS (ESI+) *m/z* calcd (C₁₃H₁₂N₂O₅) 276.0746, found 276.0747.

***trans*-(4-Nitrophenyl)hexahydro-2*H*-benzo[*e*][1,3]oxazine-2,4(3*H*)-dione** (*trans*-4rb';

Table 4, entry 4)

Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 1.23-1.71 (4 H, m), 1.95 (2 H, m), 2.38 (2 H, m), 2.60 (1 H, td, ³*J* = 11.5 Hz, 4.1 Hz), 4.36 (1 H, td, ³*J* = 11.5 Hz, 4.7 Hz), 7.39 (2 H, pd), 8.33 (2 H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ 23.7, 24.3, 24.6, 31.3, 44.7, 77.6, 124.8, 129.9, 140.6, 147.9, 150.8, 170.3. HRMS (EI+) *m/z* calcd (C₁₄H₁₄N₂O₅) 290.09027, found 290.08953.

(*E*)-4-(4-Nitrophenylamino)-4-oxobut-2-enyl Butanoate (**5**). Crystallized from CH₂Cl₂/hexanes. ¹H NMR (CDCl₃, 300 MHz) δ 0.99 (3 H, t, ³*J* = 7.5 Hz), 1.71 (2 H, sextet, ³*J* = 7.5), 2.39 (2 H, t, ³*J* = 7.5 Hz), 4.82 (2 H, m), 6.18 (1 H, d, ³*J* = 15.5 Hz), 7.07 (1 H, dt, ³*J* = 15.5 Hz, 4.5 Hz), 7.47 (1 H, br s), 7.76 (2 H, pd), 8.37 (2 H, pd). ¹³C NMR (CDCl₃, 75 MHz) δ 13.9, 18.6, 36.2, 62.6, 119.5, 124.0, 125.4, 141.1, 143.7, 143.9, 163.2, 173.2. HRMS (EI+) *m/z* calcd (C₁₄H₁₆N₂O₅) 292.1059, found 292.1054.

¹H NMR Spectra of New Compounds

Figure S1. ¹H NMR Spectrum of **4aa'** in CDCl₃.

Figure S2. ¹H NMR Spectrum of **4ab'** in CDCl₃.

Figure S3. ^1H NMR Spectrum of **4ad'** in CDCl_3 .

Figure S4. ^1H NMR Spectrum of **4ae'** in CDCl_3 .

Figure S5. ^1H NMR Spectrum of **4ag'** in CDCl_3 .

Figure S6. ^1H NMR Spectrum of **4ah'** and triethylisocyanurate in CDCl_3 .

Figure S7. ^1H NMR Spectrum of **4bb'** in CDCl_3 .

Figure S8. ^1H NMR Spectrum of **4cb'** in CDCl_3 .

Figure S9. ¹H NMR Spectrum of **4db'** in CDCl₃.

Figure S10. ¹H NMR Spectrum of **4eb'** in CDCl₃.

Figure S11. ¹H NMR Spectrum of **4fb'** in CDCl₃.

Figure S12. ¹H NMR Spectrum of **4gb'** in CDCl₃.

Figure S13. ¹H NMR Spectrum of **4hb'** in CDCl₃.

Figure S14 ¹H NMR Spectrum of **4ib'** in CDCl₃.

Figure S15. ^1H NMR Spectrum of **4jb'** in CDCl_3 .

Figure S16. ^1H NMR Spectrum of **4kb'** in CDCl_3 .

Figure S17. ¹H NMR Spectrum of **4lb'** in CDCl₃.

Figure S18. ¹H NMR Spectrum of **4mb'** in CDCl₃.

Figure S19. ^1H NMR Spectrum of *trans*-4pb'.

Figure S20. ^1H NMR Spectrum of *cis*-4pb' in CDCl_3 .

Figure S21. ^1H NMR Spectrum of *trans*-4qb'.

Figure S22. ^1H NMR Spectrum of *trans*-4rb'.

Figure S23. ¹H NMR Spectrum of **5** in CDCl₃.

Figure S24. ORTEP structure of OD **4qb'** showing the *trans* stereochemistry of ring fusion. Thermal ellipsoids drawn at the 30% probability level.

X-Ray Crystal Structure Data for 4qb'

Table 1. Crystal data and structure refinement for 4qb'.

Identification code	tc12	
Empirical formula	C13 H12 N2 O5	
Formula weight	276.25	
Temperature	295(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbcn	
Unit cell dimensions	a = 17.4307(13) Å	$\alpha = 90^\circ$
	b = 5.3287(3) Å	$\beta = 90^\circ$
	c = 26.7589(19) Å	$\gamma = 90^\circ$
Volume	2485.4(3) Å ³	
Z	8	
Density (calculated)	1.476 Mg/m ³	
Absorption coefficient	0.115 mm ⁻¹	
F(000)	1152	
Crystal size	1.00 x 0.15 x 0.02 mm ³	
Theta range for data collection	1.52 to 21.02°	
Index ranges	-17 ≤ h ≤ 17, -5 ≤ k ≤ 5, -26 ≤ l ≤ 26	
Reflections collected	16703	
Independent reflections	1335 [R(int) = 0.0558]	
Completeness to theta = 21.02°	99.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9977 and 0.8933	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	1335 / 0 / 209	
Goodness-of-fit on F ²	1.021	
Final R indices [I > 2sigma(I)]	R1 = 0.0379, wR2 = 0.0981	
R indices (all data)	R1 = 0.0619, wR2 = 0.1149	
Largest diff. peak and hole	0.251 and -0.204 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4qb'**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	1994(1)	2418(4)	3103(1)	48(1)
O(2)	1775(1)	447(4)	3808(1)	47(1)
O(3)	388(1)	-4339(4)	3060(1)	51(1)
O(4)	1799(2)	-2404(7)	754(1)	108(1)
O(5)	982(2)	546(7)	737(1)	107(1)
N(1)	1238(1)	-1080(4)	3063(1)	36(1)
N(2)	1381(2)	-913(7)	959(1)	66(1)
C(1)	1690(2)	694(6)	3314(1)	38(1)
C(2)	1522(2)	-1910(8)	4020(1)	54(1)
C(3)	1478(2)	-1897(7)	4582(1)	67(1)
C(4)	866(2)	-3887(8)	4689(1)	88(1)
C(5)	487(3)	-4589(8)	4190(1)	58(1)
C(6)	735(2)	-2493(6)	3848(1)	49(1)
C(7)	750(2)	-2831(6)	3298(1)	40(1)
C(8)	1271(2)	-1022(5)	2522(1)	34(1)
C(9)	1650(2)	-2919(7)	2276(1)	40(1)
C(10)	1692(2)	-2883(7)	1760(1)	44(1)
C(11)	1344(2)	-944(6)	1510(1)	42(1)
C(12)	962(2)	957(7)	1747(1)	43(1)
C(13)	930(2)	910(6)	2264(1)	41(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for **4qb'**.

O(1)-C(1)	1.201(3)	O(5)-N(2)-C(11)	118.6(4)
O(2)-C(1)	1.338(3)	O(4)-N(2)-C(11)	118.2(3)
O(2)-C(2)	1.447(4)	O(1)-C(1)-O(2)	119.4(3)
O(3)-C(7)	1.204(3)	O(1)-C(1)-N(1)	122.5(3)
O(4)-N(2)	1.211(4)	O(2)-C(1)-N(1)	118.0(3)
O(5)-N(2)	1.201(4)	O(2)-C(2)-C(6)	110.1(3)
N(1)-C(1)	1.402(4)	O(2)-C(2)-C(3)	113.7(3)
N(1)-C(7)	1.411(4)	C(6)-C(2)-C(3)	105.3(3)
N(1)-C(8)	1.447(4)	C(2)-C(3)-C(4)	102.7(3)
N(2)-C(11)	1.474(4)	C(3)-C(4)-C(5)	107.8(3)
C(2)-C(6)	1.478(5)	C(6)-C(5)-C(4)	102.9(3)
C(2)-C(3)	1.505(5)	C(7)-C(6)-C(2)	108.5(3)
C(3)-C(4)	1.530(5)	C(7)-C(6)-C(5)	121.2(3)
C(4)-C(5)	1.535(5)	C(2)-C(6)-C(5)	103.5(3)
C(5)-C(6)	1.507(5)	O(3)-C(7)-N(1)	121.4(3)
C(6)-C(7)	1.484(4)	O(3)-C(7)-C(6)	126.6(3)
C(8)-C(13)	1.375(4)	N(1)-C(7)-C(6)	111.9(3)
C(8)-C(9)	1.376(4)	C(13)-C(8)-C(9)	121.1(3)
C(9)-C(10)	1.382(4)	C(13)-C(8)-N(1)	120.1(3)
C(10)-C(11)	1.372(4)	C(9)-C(8)-N(1)	118.8(3)
C(11)-C(12)	1.369(4)	C(8)-C(9)-C(10)	119.6(3)
C(12)-C(13)	1.384(4)	C(11)-C(10)-C(9)	118.4(3)
		C(12)-C(11)-C(10)	123.0(3)
C(1)-O(2)-C(2)	116.0(3)	C(12)-C(11)-N(2)	118.5(3)
C(1)-N(1)-C(7)	124.8(3)	C(10)-C(11)-N(2)	118.5(3)
C(1)-N(1)-C(8)	116.3(2)	C(11)-C(12)-C(13)	118.1(3)
C(7)-N(1)-C(8)	118.9(2)	C(8)-C(13)-C(12)	119.9(3)
O(5)-N(2)-O(4)	123.2(3)		

Symmetry transformations used to generate equivalent atoms

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4qb'**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
O(1)	47(1)	46(1)	51(1)	7(1)	-4(1)	-12(1)
O(2)	52(2)	53(2)	35(1)	-1(1)	1(1)	-19(1)
O(3)	53(1)	53(1)	45(1)	-4(1)	2(1)	-18(1)
O(4)	156(3)	123(3)	47(2)	-7(2)	19(2)	36(3)
O(5)	125(3)	147(3)	50(2)	36(2)	-1(2)	37(2)
N(1)	38(2)	39(2)	30(2)	1(1)	0(1)	-8(1)
N(2)	74(2)	80(3)	44(2)	6(2)	3(2)	-5(2)
C(1)	36(2)	42(2)	36(2)	-1(2)	2(2)	0(2)
C(2)	60(3)	64(3)	36(2)	4(2)	-1(2)	-17(2)
C(3)	76(3)	89(3)	36(2)	4(2)	-2(2)	-22(2)
C(4)	114(4)	101(3)	49(2)	13(2)	1(2)	-45(3)
C(5)	74(3)	56(3)	44(2)	3(2)	11(2)	-15(2)
C(6)	52(2)	54(2)	42(2)	-2(2)	5(2)	-14(2)
C(7)	42(2)	38(2)	40(2)	-1(2)	0(2)	-4(2)
C(8)	33(2)	35(2)	34(2)	1(2)	0(2)	-3(2)
C(9)	40(2)	39(2)	41(2)	7(2)	-2(2)	3(2)
C(10)	44(2)	45(2)	42(2)	-3(2)	6(2)	2(2)
C(11)	45(2)	53(2)	29(2)	9(2)	0(2)	-9(2)
C(12)	41(2)	44(2)	45(3)	8(2)	-6(2)	-1(2)
C(13)	36(2)	40(2)	46(2)	-2(2)	1(2)	1(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4qb'**.

	x	y	z	U(eq)
H(2)	1760(20)	-3420(80)	3876(14)	90(14)
H(3A)	1322	-266	4706	81
H(3B)	1966	-2351	4730	81
H(4A)	486	-3228	4918	105
H(4B)	1100	-5356	4839	105
H(6A)	413	-1031	3921	59
H(9)	1861(15)	-4170(50)	2446(11)	38(9)
H(10)	1958(16)	-4100(50)	1589(11)	38(9)
H(12)	700(20)	2290(70)	1575(12)	69(11)
H(13)	670(16)	2190(60)	2435(10)	43(9)
H(5B)	680(20)	-6160(90)	4086(15)	98(16)
H(5A)	-40(20)	-4710(70)	4216(13)	83(14)

Figure S25. ORTEP structure of (*R*)-**4bd'** showing absolute stereochemistry. Thermal ellipsoids drawn at the 40% probability level.

X-Ray Crystal Structure Data for (*R*)-4bd'

Table 1. Crystal data and structure refinement for (*R*)-4bd'.

Identification code	tc13	
Empirical formula	C11 H10 Br N O3	
Formula weight	284.11	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 5.3536(4) Å	$\alpha = 90^\circ$
	b = 9.2517(7) Å	$\beta = 90^\circ$
	c = 22.0423(16) Å	$\gamma = 90^\circ$
Volume	1091.75(14) Å ³	
Z	4	
Density (calculated)	1.729 Mg/m ³	
Absorption coefficient	3.755 mm ⁻¹	
F(000)	568	
Crystal size	0.60 x 0.10 x 0.10 mm ³	
Theta range for data collection	1.85 to 30.51°	
Index ranges	-7 ≤ h ≤ 7, -12 ≤ k ≤ 13, -31 ≤ l ≤ 31	
Reflections collected	16068	
Independent reflections	3298 [R(int) = 0.0423]	
Completeness to theta = 30.51°	99.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7052 and 0.2116	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3298 / 0 / 185	
Goodness-of-fit on F ²	0.964	
Final R indices [I > 2sigma(I)]	R1 = 0.0277, wR2 = 0.0578	
R indices (all data)	R1 = 0.0397, wR2 = 0.0611	
Absolute structure parameter	0.006(8)	
Largest diff. peak and hole	0.532 and -0.263 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for (*R*)-**4bd**'. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Br(1)	6738(1)	2606(1)	2035(1)	33(1)
O(1)	6076(3)	607(2)	5591(1)	26(1)
O(2)	2180(3)	-1421(2)	4297(1)	28(1)
O(3)	8596(3)	1439(2)	4880(1)	27(1)
N(1)	5069(3)	242(2)	4575(1)	21(1)
C(1)	3472(4)	269(2)	5729(1)	24(1)
C(2)	2782(4)	-1045(2)	5364(1)	24(1)
C(3)	3232(4)	-792(2)	4702(1)	22(1)
C(4)	6702(4)	816(2)	5011(1)	22(1)
C(5)	3316(6)	49(3)	6406(1)	36(1)
C(6)	5437(4)	702(2)	3954(1)	21(1)
C(7)	3700(4)	1645(2)	3708(1)	26(1)
C(8)	4058(4)	2174(2)	3129(1)	27(1)
C(9)	6142(4)	1753(2)	2804(1)	25(1)
C(10)	7832(4)	778(2)	3038(1)	26(1)
C(11)	7464(4)	244(2)	3622(1)	25(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for (*R*)-**4bd'**.

Br(1)-C(9)	1.8968(19)	C(4)-N(1)-C(6)	116.84(16)
O(1)-C(4)	1.337(2)	O(1)-C(1)-C(2)	107.23(17)
O(1)-C(1)	1.461(3)	O(1)-C(1)-C(5)	106.71(19)
O(2)-C(3)	1.205(2)	C(2)-C(1)-C(5)	114.04(18)
O(3)-C(4)	1.202(3)	C(3)-C(2)-C(1)	110.85(16)
N(1)-C(3)	1.400(3)	O(2)-C(3)-N(1)	120.65(18)
N(1)-C(4)	1.404(3)	O(2)-C(3)-C(2)	124.78(19)
N(1)-C(6)	1.447(2)	N(1)-C(3)-C(2)	114.56(17)
C(1)-C(2)	1.504(3)	O(3)-C(4)-O(1)	120.77(18)
C(1)-C(5)	1.507(3)	O(3)-C(4)-N(1)	122.81(17)
C(2)-C(3)	1.497(3)	O(1)-C(4)-N(1)	116.40(18)
C(6)-C(11)	1.375(3)	C(11)-C(6)-C(7)	121.03(19)
C(6)-C(7)	1.386(3)	C(11)-C(6)-N(1)	121.29(19)
C(7)-C(8)	1.378(3)	C(7)-C(6)-N(1)	117.66(18)
C(8)-C(9)	1.383(3)	C(8)-C(7)-C(6)	119.5(2)
C(9)-C(10)	1.378(3)	C(7)-C(8)-C(9)	119.46(19)
C(10)-C(11)	1.392(3)	C(10)-C(9)-C(8)	121.28(18)
		C(10)-C(9)-Br(1)	119.83(16)
C(4)-O(1)-C(1)	117.98(16)	C(8)-C(9)-Br(1)	118.84(15)
C(3)-N(1)-C(4)	123.95(16)	C(9)-C(10)-C(11)	119.1(2)
C(3)-N(1)-C(6)	119.11(16)	C(6)-C(11)-C(10)	119.6(2)

Symmetry transformations used to generate equivalent atoms

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for (*R*)-**4bd'**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
Br(1)	35(1)	37(1)	27(1)	7(1)	3(1)	1(1)
O(1)	23(1)	31(1)	25(1)	1(1)	-7(1)	-4(1)
O(2)	27(1)	25(1)	32(1)	-4(1)	-5(1)	-7(1)
O(3)	18(1)	32(1)	32(1)	0(1)	-4(1)	-5(1)
N(1)	19(1)	22(1)	24(1)	1(1)	-4(1)	-1(1)
C(1)	23(1)	22(1)	25(1)	1(1)	-1(1)	3(1)
C(2)	23(1)	18(1)	31(1)	4(1)	-1(1)	-3(1)
C(3)	20(1)	18(1)	30(1)	1(1)	-3(1)	4(1)
C(4)	20(1)	20(1)	26(1)	0(1)	-6(1)	3(1)
C(5)	41(2)	40(1)	26(1)	4(1)	-3(1)	-5(1)
C(6)	17(1)	21(1)	24(1)	-2(1)	-4(1)	-3(1)
C(7)	21(1)	26(1)	29(1)	0(1)	0(1)	6(1)
C(8)	22(1)	27(1)	30(1)	4(1)	-1(1)	7(1)
C(9)	26(1)	25(1)	23(1)	1(1)	-1(1)	-2(1)
C(10)	20(1)	31(1)	27(1)	-2(1)	0(1)	2(1)
C(11)	21(1)	25(1)	30(1)	1(1)	-1(1)	4(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for (*R*)-**4bd**.

	x	y	z	U(eq)
H(1)	2520(40)	1060(20)	5603(9)	17(6)
H(2B)	3840(40)	-1860(20)	5493(10)	25(6)
H(2A)	1040(40)	-1360(20)	5387(9)	18(5)
H(5C)	1640(60)	-220(30)	6499(13)	58(9)
H(5B)	3820(50)	920(30)	6629(11)	41(7)
H(5A)	4510(60)	-810(30)	6516(13)	62(9)
H(7)	2220(50)	1930(30)	3968(10)	33(6)
H(6)	2960(50)	2850(30)	2972(10)	43(7)
H(10)	9220(50)	500(30)	2826(11)	41(7)
H(11)	8470(60)	-460(30)	3793(11)	46(7)

Notes and References

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