SUPPORTING INFORMATION

Hypervalent Iodine (III) Reagents as Safe Alternatives to α-Nitro-α-Diazocarbonyls: A Comparison of Reactivities.

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General: All ylide reactions were run under oxygen containing atmospheres in open flasks, while the diazo mediated cyclopropanations were performed with rigid exclusion of moisture from reagents and glassware using standard techniques for manipulating air-sensitive compounds (although not necessary).[1] Anhydrous dichloromethane was obtained by filtration through drying columns on a GlassContour system (Irvine, CA). Analytical thin-layer chromatography (TLC) was performed on pre-coated, glass-backed silica gel (Merck 60 F_{254}). Visualization of the developed chromatogram was performed by UV absorbance, aqueous cerium molybdate or potassium permanganate. Flash column chromatography was performed using 230-400 mesh silica (EM Science or Silicycle) of the indicated solvent system according to standard technique.[2] Analytical Supercritical Fluid Chromatography was performed on a Berger SFC Analytical Instrument equipped with a diode array UV detector. Data is reported as follows: column type, eluent, flow rate and retention time (t_r).

Materials: $[Rh(OAc)_2]_2$ and $[Rh(OPiv)_2]_2$ complexes and iodobenzene diacetate (BAIB) were purchased from Aldrich Chemical Company. $[Rh(C_7H_{15}CO_2)_2]_2$ was purchased from Strem Chemicals. Olefin substrates are commercially available from Aldrich Chemical Company. Indene, styrene and α-methyl styrene were distilled prior to use while 4-vinyl anisole, *p*-chloro styrene, 1,1-diphenylethylene and methylene cyclopentane were used directly without further purification. α-Nitro carbonyl substrates **3**, **5d** can be purchased

[1]Shriver, D. F.; Drezdzon, M. A. *The manipulation of air-sensitive compounds*; 2nd Edition Ed.; Wiley: New York, 1986.
[2]Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.

from Aldrich Chemical Company or easily prepared according to Koyama *et al*.[3] and Putt *et al*[4] respectively, while **5a-c** have been previously reported in the literature.[5,6] Cyclopropanes **4, 6c,d** accord exactly with those that have been previously reported in the literature.[7],[8]

Typical Experimental Procedure for Rh(II) Carboxylate Catalyzed Cyclopropanation of Alkenes with a-Nitro Carbonyls and Iodobenzene Diacetate (Aqueous Reaction).

Styrene (485 mg, 0.53 mL, 5 equiv) was added to a 10 mL round bottomed flask containing the required amount of $[Rh(OPiv)_2]_2$ catalyst (2.8 mg, 4.7 x 10^{-3}mmol, 0.5 mol%) followed by the methyl nitro acetate (111 mg, 0.93 mmol, 1 equiv). To this homogeneous solution was added iodobenzene diacetate (330 mg, 1.03 mmol, 1.1 equiv) in one portion and the mixture allowed to stir 30 secs. Distilled water (5.0 mL) containing Na_2CO_3 (198 mg, 1.86 mmol, 2 equiv) was added over 5 secs and the biphasic reaction mixture was allowed to stir 2h. The crude reaction mixture was treated with a NaCl saturated solution (10 mL) and extracted with CH_2Cl_2 (3 x 5 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The resulting crude residue was then chromatographed on silica gel first eluting with hexanes (to remove excess alkene) then 5% EtOAc/hexanes affording **4** as a clear, colorless oil (177 mg, 86%) in a 93:7 *E:Z* ratio of the two diastereomers.

Typical Experimental Procedure for Rh(II) Carboxylate Catalyzed Cyclopropanation of Alkenes with a-Nitro Carbonyls and Iodobenzene Diacetate.

Styrene (232 mg, 0.25 mL, 5 equiv) was added to a 10 mL round bottomed flask containing the required amount of $[Rh(OPiv)_2]_2$ catalyst (1.4 mg, 2.2 x 10^{-3}mmol, 0.5 mol%) followed by the methyl nitro acetate (53 mg, 0.45 mmol, 1 equiv). To this

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[3]Koyama, Z. M.; Koto, S. *Org. Synth.* **1988**, C.V. 6, 797.
[4]Baker, D. C.; Putt S. R. *Synthesis* **1978**, 478.
[5]Sylvain, C.; Wagner, A.; Mioskowski C. *Tetrahedron Lett.* **1999**, *40*, 875.
[6]Charette, A. B.; Wurz, R. P.; Ollevier, T. *J. Org. Chem.* **2000**, *65*, 9252.
[7]Charette, A. B.; Wurz, R. P.; Ollevier, T. *Helv. Chim. Acta.* **2002**, *85*, 4468.
[8]Charette, A. B.; Wurz, R. P. *J. Mol. Cat. A: Chem.* **2002**, *article in press*.

homogeneous solution was added iodobenzene diacetate (158 mg, 0.49 mmol, 1.1 equiv) in one portion and the mixture allowed to stir 2h. The crude reaction mixture was then chromatographed directly on silica gel first using hexanes as an eluent (to remove excess alkene) then 5% EtOAc/hexanes affording **4** as a clear, colorless oil (82 mg, 83%) in a 92:8 *E:Z* ratio of the two diastereomers.

1-Cyclopropyl-2-nitro-ethanone (5e). The title compound was obtained according to the typical preparative procedure[4] (2.20 g, 74%) as a white crystalline solid: M.p. 73-75 ^{o}C; R_f 0.15 (4:1 hexanes/EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) d 5.44 (s, 2H), 1.93-1.98 (m, 1H), 1.24-1.28 (m, 2H), 1.10-1.15 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) d 196.5, 83.9, 19.2, 13.1; IR (solid) 1711 (C=O), 1553, 1377, 1300 (NO_2), 1068 cm^{-1}; HRMS (EI^{+}): $[M]^{+}$ $C_5H_7NO_3$: 129.042593, found 129.042834; Anal. $C_5H_7NO_3$ calcd C, 46.51; H, 5.46; N, 10.85; found: C, 46.39; H, 5.53; N, 10.89.

Characterization of Cyclopropanes

Allyl *E- and Z-1-nitro-2-phenylcyclopropanecarboxylate* (**6a**): Clear, colorless oil (86 mg, 71%): **allyl *E*-1-nitro-2-phenylcyclopropanecarboxylate**: R_f 0.58 (4:1 hexanes:EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) d 7.30 (s, 3H), 7.21-7.22 (m, 2H), 5.43-5.52 (m, 1H), 5.07-5.12 (m, 2H), 4.38-4.39 (m, 2H), 3.76-3.81 (m, 1H), 2.45-2.49 (m, 1H), 2.20-2.24 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) d 161.8, 132.1, 130.7, 128.7, 128.66, 128.64, 128.4, 119.3, 71.9, 67.1, 34.3, 20.9; IR (film) n 1742 (C=O), 1541, 1350, 1331 (NO_2) cm^{-1}; **allyl *Z*-1-nitro-2-phenylcyclopropanecarboxylate**: R_f 0.46 (4:1 hexanes:EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) d 7.32-7.34 (m, 3H), 7.22-7.27 (m, 2H) 5.91-5.98 (m, 1H), 5.36 (ddd, *J* = 30.0, 16.5, 1.2 Hz, 2H), 4.77-4.84 (m, 2H), 3.51 (t, *J* = 9.5 Hz, 1H), 2.71 (dd, *J* = 9.2, 7.0 Hz, 1H), 2.06 (dd, *J* = 9.9, 7.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) d 165.3, 131.5, 130.9, 128.92, 128.88, 128.6, 119.8, 75.8, 72.7, 67.6, 34.1, 20.3; IR (film) n 1740 (C=O), 1544, 1283 (NO_2), 1152 cm^{-1};

Benzyl *E- and Z-1-nitro-2-phenylcyclopropanecarboxylate* (6b): benzyl *E*-1-nitro-2-phenylcyclopropanecarboxylate: White crystalline solid; M.p. 49-50 ^{o}C; R_f 0.52 (4:1 hexanes:EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$) d 7.22-7.34 (m, 6H), 7.17-7.21 (m, 2H), 7.00-7.04 (m, 2H), 4.94 (dd, *J* = 21.7, 12.1 Hz, 1H), 3.80 (t, *J* = 10.0 Hz, 1H), 2.48 (dd, *J* = 9.2, 6.6 Hz, 1H), 2.23 (dd, *J* = 10.8, 6.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) d 162.1, 134.4, 132.0, 128.7, 128.64, 128.60, 128.54, 128.47, 128.45, 71.9, 68.4, 34.4, 21.1; IR

(solid) ν 1744 (C=O), 1543, 1351 (NO_2) cm^{-1}; **benzyl *Z*-1-nitro-2-phenylcyclopropanecarboxylate**: White crystalline solid; M.p. 88-90 °C; R_f 0.48 (4:1 hexanes:EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.39 (m, 4H), 7.30-7.34 (m, 4H), 7.21-7.27 (m, 2H), 5.33 (dd, *J* = 17.4, 12.3 Hz, 2H), 3.51 (t, *J* = 9.6 Hz, 1H), 2.70 (dd, *J* = 9.2, 7.0 Hz, 1H), 2.05 (dd, *J* = 9.9, 7.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.4, 134.6, 131.5, 129.0, 128.92, 128.89, 128.6, 128.4, 72.8, 68.8, 34.1, 20.4; IR (solid) ν 1739 (C=O), 1532, 1298 (NO_2), 1160 cm^{-1}; Anal. calcd for $C_{17}H_{15}NO_4$: C, 68.68; H, 5.09; N, 4.71; found C, 68.39; H, 5.18; N, 4.72.

***E*- and *Z*-Cyclopropyl-(1-nitro-2-phenyl-cyclopropyl)-methanone (6e):** (115 mg, 72%) ***E*-cyclopropyl-(1-nitro-2-phenyl-cyclopropyl)-methanone.** Pale yellow crystalline solid; M.p. 69-70 °C; R_f 0.65 (4:1 hexanes/EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25-7.31 (m, 3H), 7.11-7.17 (m, 2H), 3.86 (t, *J* = 9.9 Hz, 1H), 2.58 (dd, *J* = 9.3, 6.3 Hz, 1H), 2.22 (dd, *J* = 10.6, 6.3 Hz, 1H), 1.99-2.07 (m, 1H), 1.08-1.16 (m, 1H), 0.89-0.99 (m, 1H), 0.40-0.49 (m, 1H), 0.09-0.17 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.1, 131.9, 128.7, 128.66, 128.4, 77.7, 37.0, 21.9, 20.1, 13.6, 11.8; IR (solid) 1697 (C=O), 1526, 1345 (NO_2), 1057 cm^{-1}; HRMS (EI^+): $[M]^+$ $C_{13}H_{13}NO_3$: 231.089543, found 231.089556; ***Z*-cyclopropyl-(1-nitro-2-phenyl-cyclopropyl)-methanone.** Semi-solid; R_f 0.62 (4:1 hexanes/EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21-7.33 (m, 5H), 3.61 (t, *J* = 9.5 Hz, 1H), 2.75 (dd, *J* = 9.3, 6.5 Hz, 1H), 1.93-2.07 (m, 2H), 1.21-1.32 (m, 1H), 0.90-0.99 (m, 1H), 0.40-0.49 (m, 1H), 0.09-0.18 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.6, 131.8, 128.8, 128.5, 76.7, 35.6, 22.6, 18.7, 13.5; IR (film) 1696 (C=O), 1529, 1392, 1346 (NO_2), 1057 cm^{-1}; Anal. $C_{13}H_{13}NO_3$ calcd C, 67.52; H, 5.67; N, 6.06; found: C, 67.19; H, 5.65; N, 6.00.

Methyl *exo*-1-nitro-2,3-indenecyclopropanecarboxylate (8a): White crystalline solid (150 mg, 91%)**:** M.p. 80-82 °C; R_f 0.51 (4:1 hexanes:EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.44 (m, 1H), 7.16-7.24 (m, 3H), 3.84 (d, *J* = 7.3 Hz, 1H), 3.43-3.47 (m, 2H), 3.41 (s, 3H), 3.11-3.16 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 141.8, 137.7, 128.3, 127.6, 125.9, 125.0, 72.9, 53.0, 42.0, 34.43, 34.36; IR (film) ν 1747 (C=O), 1543, 1346 (NO_2) cm^{-1}; HRMS (EI^+): *m/z*: 233.069010, $C_{12}H_{11}NO_4$ requires 233.068808; Anal. calcd for $C_{12}H_{11}NO_4$: C, 61.80; H, 4.75; N, 6.01; found C, 61.90; H, 4.73; N, 6.02. The structure was further confirmed by x-ray crystallography.[9]

5-(4-Methoxyphenyl)-2-oxy-4,5-dihydro-isoxazole-3-carboxylic acid methyl ester (8b): White platelets (94 mg, 82%): M.p. 117-118 °C; R_f 0.19 (4:1 hexanes:EtOAc); ^{1}H

[9]Charette, A. B.; Wurz, R. P.; Ollevier, T. *Helv. Chim. Acta.* **2002**, *85*, 4468.

NMR (400 MHz, $CDCl_3$) d 7.32 (d, *J* = 8.7 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 5.66 (t, *J* = 8.9 Hz, 1H), 3.85 (s, 3H), 3.81 (s, 3H), 3.71 (dd, *J* = 16.9, 9.5 Hz, 1H), 3.42 (dd, *J* = 16.9, 8.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) d 160.5, 159.6, 129.3, 127.7, 114.5, 108.3, 77.3, 55.5, 52.8, 38.2; IR (solid): n 1736 (C=O), 1611 (N-oxide) cm^{-1}; HRMS (EI^+): *m/z*: $C_{12}H_{13}NO_5$ requires 251.079373 found 251.080215. Anal. calcd for $C_{12}H_{13}NO_5$: C, 57.37; H, 5.22; N, 5.58; found C, 57.27; H, 5.19; N, 5.53. ***E*-Methyl 1-nitro-2-(4-methoxyphenyl)cyclopropane carboxylate (8b):** Found in the crude reaction mixture ^{1}H NMR (400 MHz, $CDCl_3$) d 7.13 (d, *J* = 8.9 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 3.80 (s, 3H), 3.73 (t, *J* = 9.9 Hz, 1H), 3.54 (s, 3H), 2.42 (dd, *J* = 9.1, 6.6 Hz, 1H), 2.21 (dd, *J* = 10.8, 6.6 Hz, 1H). ***Z*-Methyl 1-nitro-2-(4-methoxyphenyl)cyclopropane carboxylate (8b):** ^{1}H NMR (400 MHz, $CDCl_3$) d 7.15 (d, *J* = 8.2 Hz, 2H), 6.85 (d, *J* = 8.2 Hz, 2H), 3.89 (s, 3H), 3.82 (s, 3H), 3.45 (t, *J* = 9.6 Hz, 1H) 2.65 (dd, *J* = 9.2, 7.0 Hz, 1H), 2.03 (dd, *J* = 9.9, 6.9 Hz, 1H).

Methyl *E*- and ***Z*-1-nitro-2-(4-chlorophenyl)-cyclopropanecarboxylate (8c)**: (154 mg, 75%): **methyl *E*-1-nitro-2-(4-chlorophenyl)-cyclopropanecarboxylate**: M.p. 33-35 °C; R_f 0.57 (4:1 hexanes:EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) d 7.29 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 8.3 Hz, 2H), 3.71 (t, *J* = 9.9 Hz, 1H), 3.55 (s, 3H), 2.40 (dd, *J* = 9.1, 6.8 Hz, 1H), 2.22 (dd, *J* = 10.7, 6.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) d 162.5, 134.4, 130.8, 129.9, 128.9, 71.7, 53.5, 33.5, 21.0; IR (film) n 1746 (C=O), 1546, 1347 (NO_2) cm^{-1}; HRMS (EI^+) calcd for $C_{11}H_{10}NO_4Cl$ requires 255.029836 found 255.029762; **methyl *Z*-1-nitro-2-(4-chlorophenyl)-cyclopropane carboxylate**: Clear, colorless oil, R_f 0.30 (4:1 hexanes:EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) d 7.29 (d, *J* = 6.5 Hz, 2H), 7.15 (d, *J* = 6.5 Hz, 2H), 3.90 (s, 3H), 3.45 (t, *J* = 9.5 Hz, 1H), 2.65 (dd, *J* = 9.1, 7.1 Hz, 1H), 2.05 (dd, *J* = 9.9, 7.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) d 165.8, 135.0, 130.0, 129.9, 129.2, 72.5, 54.1, 33.2, 20.3; IR (film) n 1745 (C=O), 1544, 1296 (NO_2) cm^{-1}; Anal. calcd for $C_{11}H_{10}NO_4Cl$: C, 51.68; H, 3.94; N, 5.48; found C, 51.51; H, 3.91; N, 5.49.

Methyl 1-nitro-2-phenyl-2-methyl-cyclopropanecarboxylate (8d): Clear, colorless oil (130 mg, 80%): R_f 0.60 (4:1 hexanes:EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) d 7.26-7.34 (m, 5H), 3.49 (s, 3H), 2.42 (d, *J* = 6.8 Hz, 1H), 2.17 (d, *J* = 6.8 Hz, 1H), 1.54 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) d 164.2, 139.0, 128.8, 127.9, 127.8, 76.2, 53.2, 39.4, 26.7, 24.9; IR (film): n 1745 (C=O), 1544, 1363, 1312 (NO_2) cm^{-1}; HRMS (EI^+): *m/z*: $C_{12}H_{13}NO_4$ (M+1) requires 236.092283, found 236.091740. Anal. calcd for $C_{12}H_{13}NO_4$: C, 61.27; H, 5.57; N, 5.95; found C, 61.00; H, 5.55; N, 5.91.

5-phenyl-5-methyl-2-oxy-4,5-dihydro-isoxazole-3-carboxylic acid methyl ester (8d): Clear colorless oil (25 mg, quantity varies on silica gel used): R_f 0.24 (4:1 hexanes:EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) d 7.31-7.44 (m, 5H), 3.83 (s, 3H), 3.57

(dd, J = 28.0, 16.7 Hz, 2H), 1.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) d 159.7, 143.0, 129.1, 128.4, 124.3, 109.0, 83.1, 52.8, 44.4, 28.2; IR (film): n 1739, 1704 (C=O), 1621 (N-oxide), 1445, 1256 cm^{-1}.

5,5-diphenyl-2-oxy-4,5-dihydro-isoxazole-3-carboxylic acid methyl ester (8e): White crystalline solid (120 mg, 63%): M.p. 116-117 ^{o}C; R_f 0.28 (4:1 hexanes:EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) d 7.32-7.45 (m, 10H), 4.05 (s, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) d 159.6, 141.5, 129.0, 128.7, 125.9, 109.0, 86.2, 52.9, 43.7; IR (solid): n 1700 (C=O), 1631 (N-oxide). 1441 cm^{-1}; HRMS (EI^{+}): *m/z*: $C_{17}H_{15}NO_4$ requires 297.100108 found 297.099994; Anal. calcd for $C_{17}H_{15}NO_4$: C, 68.68; H, 5.09; N, 4.71; found C, 68.59; H, 5.05; N, 4.62.

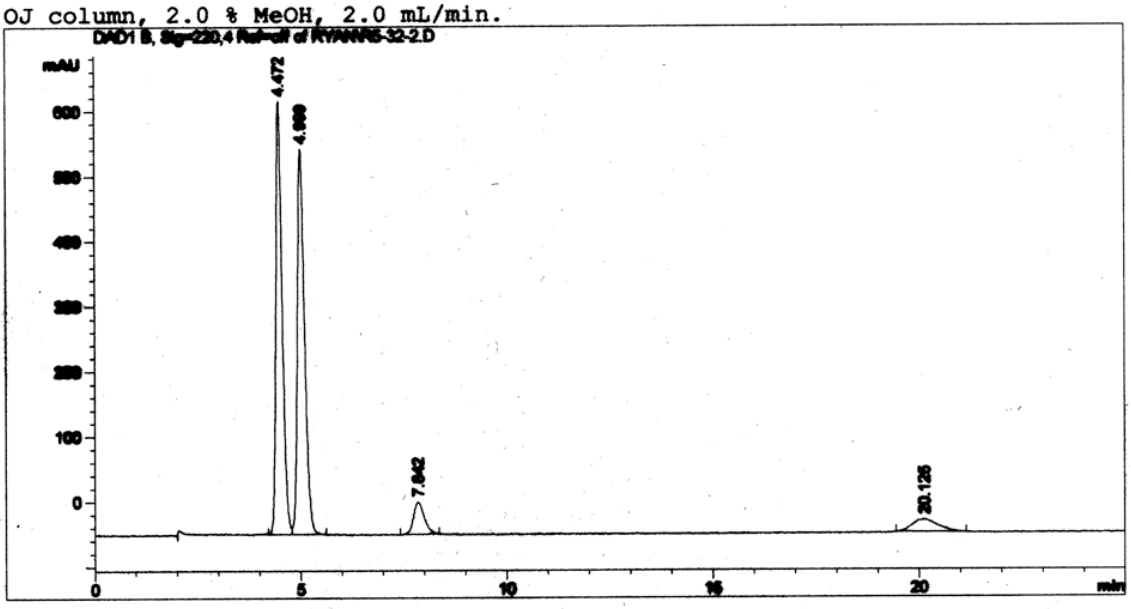
Methyl 1-nitro-2,2'-diphenylcyclopropane carboxylate (8e): The cyclopropane was found in the crude reaction mixture ^{1}H NMR (400 MHz, $CDCl_3$) d 7.24-7.47 (m, 10H), 3.60 (s, 3H), 3.04 (d, J = 6.9 Hz, 1H), 2.67 (d, J = 6.9 Hz, 1H).

1-Nitro-spiro[2.4]heptane-1-carboxylic acid methyl ester (8f): (111 mg, 73%). Slightly volatile, clear colorless oil. R_f 0.57 (4:1 hexanes:EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$) d 3.80 (s, 3H), 2.05 (d, J = 6.4 Hz, 1H), 1.96-2.02 (m, 1H), 1.78 (d, J = 6.4 Hz, 1H), 1.56-1.75 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$) d 165.6, 74.7, 53.3, 41.7, 33.7, 32.3, 29.2, 26.2, 25.8; IR (solid): n 1742 (C=O), 1542, 1438, 1365 (NO_2) cm^{-1}.

Asymmetric Cyclopropanation

The enantiomeric excess was determined by super-critical fluid chromatography (SFC) analysis (Chiracel OJ, 2.0% MeOH in supercritical CO_2, 2.0 mL/min)
E-**4** t_r 4.47, 5.00 min; *Z*-**4** t_r 7.84, 20.13 min.

Racemic **4**:



Area Percent Report

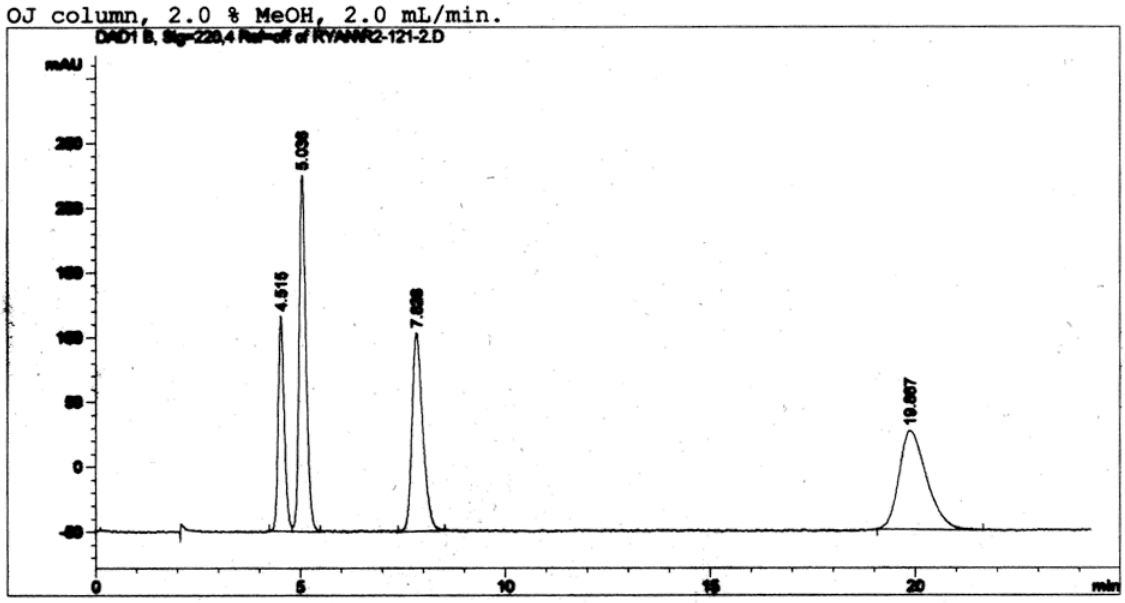
Sorted by Retention Time
Multiplier : 1.000000

Signal 1: DAD1 B, Sig=220,4 Ref=off

Peak #	RT [min]	Sig	Type	Area [mAU*sec]	Height [mAU]	Area %
1	4.472	1	BV	7063.00684	665.03156	44.3539
2	4.999	1	VB	7113.17432	592.15875	44.6690
3	7.842	1	BB	921.01257	49.93417	5.7837
4	20.125	1	BB	827.00409	19.08804	5.1934
Totals :				15924.19824	1326.21252	

4 prepared in dichloromethane with $[Rh(S\text{-PTPA})_2]_2$ catalysis and diazo compound *E*-**4** t_r 4.52, 5.04 min; *Z*-**4** t_r 7.83, 19.87 min.

OJ column, 2.0 % MeOH, 2.0 mL/min.



Area Percent Report

Sorted by Retention Time
Multiplier : 1.000000

Signal 1: DAD1 B, Sig=220,4 Ref=off

Peak #	RT [min]	Sig	Type	Area [mAU*sec]	Height [mAU]	Area %
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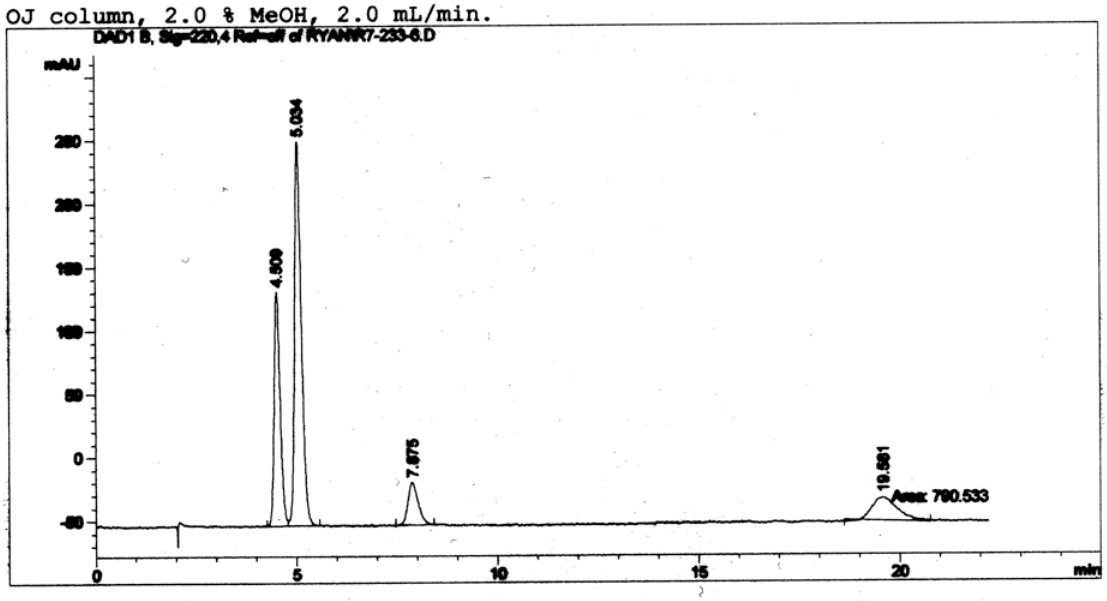
2	5.036	1	VB	3199.60229	275.40839	28.1811
3	7.828	1	BB	2824.56543	153.29242	24.8779
4	19.867	1	BB	3606.98999	76.41223	31.7693
Totals :				11353.70605	671.30359	

4 prepared using iodonium ylides with $[Rh(S\text{-PTPA})_2]_2$ catalysis

E-**4** t_r 4.51, 5.03 min; *Z*-**4** t_r 7.88, 19.58 min.



Area Percent Report

Sorted by Retention Time
Multiplier : 1.000000

Signal 1: DAD1 B, Sig=220,4 Ref=off

Peak #	RT [min]	Sig	Type	Area [mAU*sec]	Height [mAU]	Area %
1	4.509	1	BV	1922.03857	184.62891	27.9880
2	5.034	1	VB	3545.68921	302.68597	51.6309
3	7.875	1	BB	609.11365	33.95066	8.8697
4	19.581	1	MM	790.53345	18.12743	11.5114
Totals :				6867.37500	539.39294	

Benzyloxy-nitro-acetic acid methyl ester (9): (57 mg, 33%). Clear, colorless oil. R_f 0.33 (4:1 hexanes:EtOAc); ^{1}H-NMR (300 MHz, $CDCl_3$) d 7.37-7.41 (s, 5H), 5.61 (s, 1H), 5.06 (d, *J* = 11.5 Hz, 1H), 4.73 (d, *J* = 11.5 Hz, 1H), 3.85 (s, 3H); ^{13}C-NMR (75 MHz, $CDCl_3$) d 162.1, 133.9, 129.5, 129.1, 129.0, 103.0, 74.7, 54.2; IR (film) 1759 (C=O), 1568, 1215, 1163 (NO_2).