Vinyldiazolactone as a Vinylcarbene Precursor: Highly Selective C-H Insertion and Cyclopropanation Reaction

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Supporting Information

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General Information: NMR spectra were recorded on a Bruker DRX-400 MHz instrument in a solution of deuterochloroform unless otherwise noted. Chemical shifts of ^{1}H NMR are quoted relative to internal Me_4Si (0.00 ppm), those of ^{13}C NMR are quoted relative to residual solvent (77.0 ppm). NMR spectra of **1** were obtained using deuterated dichloromethane, chemical shifts of the ^{1}H NMR are quoted relative to residual solvent (5.32 ppm), chemical shifts of the ^{13}C are quoted relative to residual solvent (54.0 ppm). High-resolution spectra were obtained on a JEOL SX102a spectrometer. Thin layer chromatography was performed on Merck Silica Gel 40 F_{254} glass backed plates, visualization was achieved with UV or $KMnO_4$ stain. Column chromatography was performed on 40-63 μm, 230-400 mesh, 60 A silica gel. Cyclopropanation substrates 1-vinylcyclohexene,[1] 1-phenylbutadiene,[2] 1-phenyl-3-methylbutadiene were prepared according to literature procedures, all other reagents were purchased from Aldrich. Anhydrous dichloromethane and tetrahydrofuran were obtained by dried by nitrogen forced-flow over activated alumina as described by Grubbs.[3]

Preparation of 1: A solution of 3,5-dihydro-pyran-2-one (400 mg, 4.10 mmol) in acetonitrile (40 mL) was stirred at 0 ^{o}C. 4-Acetamidobenzenesulfonyl azide (1.23 g, 5.10 mmol) and DBU (0.77 mL, 5.1 mmol) were added to the stirred solution. After 3h, the resulting brown mixture was evaporated to a thick oil. The mixture was purified *via* flash silica gel chromatography (2:1 hexanes/ethyl acetate, Rf = 0.47), eluting **1** as a bright yellow band. Fractions were evaporated and dried under vacuum to yield **1** as an orange solid (237 mg, 47%): mp 60 ^{o}C; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 6.20 (dt, J = 10.0, 1.8 Hz, 1H), 5.38 (td, J = 3.3, 10.0 Hz, 1H), 4.99 (dd, J = 3.3, 1.8 Hz, 2H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 164.5, 114.5, 113.5, 70.5 (C = N_2 missing); IR (neat) 3073, 2882, 2098, 1668 cm^{-1}; HRMS (EI) calcd for $C_5H_4N_2O_2$ 124.0273, found 124.0275 (M+).

General Procedure for Table 1: To a flame dried flask containing anhydrous dichloromethane (20 mL), dirhodium catalyst (.013 mmol), and 1,4-cyclohexadiene (0.61 mL, 6.5 mmol) were added. The apparatus was degassed and heated to reflux under nitrogen. A solution of **1** (160 mg, 1.29 mmol in 10 mL dichloromethane) was added over 8 h. Upon completion of addition, the reaction mixture was refluxed for an additional 1h, then filtered through a short silica gel plug to remove the dirhodium catalyst. Integration of diagnostic ^{1}H NMR signals of **2a** (4.80 ppm) and **2b** (4.98 ppm) were used to determine relative ratios of **2a**,**b**. GC was used to determine the enantiomeric excess of **2a** prior to column chromatographic purification. Silica gel column chromatography (3:1 hexanes/ethyl acetate Rf = 0.40) of the reaction mixture provided an unseparated mixture of **2a**,**b** from which isolated yields were calculated from the mass of the product. Pure samples of **2a** and **2b** were obtained by subsequent silica gel chromatography (10:1 hexane/ethyl acetate), but only partial separation was achieved.

2a (from Table 1, entry 6): Clear oil, GC: Chiraldex G-TA (30m x 0.25 mm), 140 ^{o}C/10 min, 1 ^{o}C/min ramp to 160 ^{o}C), retention times 26.2 min (*R*) and 29.2 min (*S*), 80% ee *R*; $[\alpha]_D^{21}$ +79.5^o (*c* = 0.31, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.97-5.76 (comp, 4H),

[1] Herz, W.; Juo, R.-R. *J. Org. Chem.* **1985**, *50*, 618.
[2] Okamoto, T.; Kobayashi, K.; Oka, S.; Tanimoto, S. *J. Org. Chem.* **1988**, *53*, 4897.
[3] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

5.65-5.50 (comp, 2H), 4.83-4.78 (comp, 2H), 3.62-3.55 (m, 1H), 3.09-3.04 (m, 1H), 2.64-2.58 (comp, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) 170.6, 127.5, 127.1, 125.87, 123.9, 124.0, 122.9, 68.8, 44.4, 38.1, 26.2; IR (neat) 3018, 1733 cm^{-1}; HRMS (EI) calcd for $C_{11}H_{12}O_2$ 176.0837, found 176.0835 (M+).

2b (from Table 1, entry 2): Clear oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.95 (dt, *J* = 10.5, 2.9 Hz, 1H), 5.63 (m, 2H), 5.42 (dt, *J* = 10.5, 1.9 Hz, 1H), 4.98 (dd, *J* = 2.9, 1.9 Hz, 2H), 2.54-2.42 (comp, 2H), 2.19-2.16 (m, 2H), 2.09-2.01 (m 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 124.2, 122.9, 121.1, 68.5, 28.0, 26.1, 19.6; IR (neat) 2088, 1712 cm^{-1}; HRMS (EI) calcd for $C_{11}H_{12}O_2$ 176.0837, found 176.0837 (M+).

General Procedure for Table 2: To a flame dried flask containing anhydrous dichloromethane (20 mL), Rh_2(*S*,*R*-MenthAZ)$_4$ (17 mg, 0.013 mmol) and olefin (3.3 mmol) were added. The apparatus was degassed and heated to reflux under nitrogen. A solution of **1** (160 mg, 1.29 mmol in 10 mL dichloromethane) was added over 8 h. Upon completion of addition, the reaction mixture was refluxed for a further 1h, then filtered through a short silica gel plug to remove the dirhodium catalyst. Analysis by ^{1}H NMR was used to determine the ratios of *E*- and *Z*-cyclopropane isomers. Enantiomeric excess was determined by GC or HPLC, as indicated. Isolated yields of **3a-d** were determined after purification by silica gel column chromatography.

3a: (6:1 hexanes/EtOAc, Rf = 0.22) 74%, white solid, mp 98 ^{o}C; GC: (B-DM, 30m x 0.25 mm; 0.25 μm film, 140 ^{o}C/10 min, 1 ^{o}C/min ramp to 160 ^{o}C), retention times of 45.3 min (minor) and 47.3 min (major), 84% ee; $[\alpha]_D^{23}$ +83.6^o (*c* = 0.59, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.20 (comp, 5H), 5.72 (dt, *J* = 10.0, 2.8 Hz, 1H), 5.01 (dd, *J* = 2.8, 1.8 Hz, 2H), 4.96 (dt, *J* = 10.0, 1.8 Hz, 1H), 3.25 (fortuitous t, *J* = 9.0, 7.7 Hz, 1H), 2.14 (dd, *J* = 9.0, 4.8 Hz), 1H), 1.56 (dd, *J* = 7.7, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 135.5, 129.1, 128.4, 127.2, 124.7, 121.2, 69.2, 35.2, 26.8, 22.7; IR (neat) 3021, 2885, 1716 cm^{-1}; HRMS (EI) calcd for $C_{13}H_{12}O_2$ 200.0837, found 200.0834 (M+).

3b: (6:1 hexanes/EtOAc, Rf = 0.36) 77%, clear oil, GC: (B-DM, 30m x 0.25 mm; 0.25 μm film, 160 ^{o}C isotherm) retention times of 82.4 min (minor) and 96.0 min (major), 80% ee; $[\alpha]_D^{21}$ +68.2^o (*c* = 0.63, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.80 (dt, *J* = 10.2, 2.8 Hz, 1H), 5.57-5.42 (m, 1H), 5.26 (dt, *J* = 10.2, 2.0 Hz, 1H), 5.01-4.99 (comp, 2H), 2.47-2.43 (m, 1H), 2.10-1.45 (comp, 9H), 1.27 (dd, *J* = 7.4, 4.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 133.0, 125.1, 125.0, 120.6, 69.1, 37.5, 29.5, 25.2, 25.1, 22.7, 22.31, 22.25; IR (neat) 3020, 2879, 1716 cm^{-1}; HRMS (EI) calcd for $C_{13}H_{16}O_2$ 204.1150, found 204.1148 (M^+).

3c: (6:1 hexanes/EtOAc, Rf = 0.29) 81%, clear oil, 8:1 mixture *E*,*Z*-**3c** diastereomers. Diastereomeric ratio determined by ^{1}H NMR prior to chromatography (*E*-**3c** 6.38 ppm, *Z*-**3c** 6.47 ppm). Isolated yield of *E*,*Z*-**3c** obtained upon initial column chromatography (6:1 hexanes/EtOAc). Diastereomers isolated by further silica gel column chromatography (*E*-**3c** Rf = 0.44, dichloromethane).

E-**3c**: Clear oil, HPLC: (AD-H, 98:2 hexanes/2-propanol, 1 mL/min) retention times of 11.8 min (major) and 13.2 min (minor), 86% ee; $[\alpha]_D^{21}$ +77.8^o (*c* = 0.73, $CHCl_3$); ^{1}H

NMR (400 MHz, $CDCl_3$) δ 7.36-7.23 (comp, 5H), 6.38 (s, 1H), 5.84 (dt, *J* = 10.2, 2.8 Hz, 1H), 5.33 (br d, *J* = 10.2 Hz, 1H), 5.05-5.03 (comp, 2H), 2.72 (fortuitous t, *J* = 7.9 Hz, 1H), 1.96 (dd, *J* = 8.7, 4.7 Hz, 1H), 1.91 (s, 3H), 1.47 (dd, *J* = 7.4, 4.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 137.1, 133.6, 128.8, 128.2, 128.0, 126.6, 124.6, 121.2, 69.1, 39.7, 25.8, 23.1, 19.1; IR (neat) 3050, 2884, 1722 cm^{-1}; HRMS (EI) calcd for $C_{16}H_{16}O_2$ 240.1150, found 204.1141 (M^+).

3d: (6:1 hexanes/EtOAc, Rf = 0.33) 86%, clear oil, 5:1 mixture of *E*,*Z*-**3d** diastereomers. Diastereomeric ratio determined by ^1H NMR prior to chromatography (*E*-**3d** 1.28 ppm, *Z*-**3d** 1.45 ppm). Isolated yield of *E*,*Z*-**3d** obtained upon initial column chromatography (6:1 hexanes/EtOAc). Diastereomers separated by further chromatography (*E*-**3d** Rf = 0.54, *Z*-**3d** Rf = 0.25, dichloromethane).
Z-**3d**: Clear oil, ^1H NMR (400 MHz, $CDCl_3$) δ 7.36-7.19 (comp, 5H), 6.59 (d, *J* = 15.9 Hz, 1H), 6.34 (dd, *J* = 15.9, 9.1 Hz, 1H), 5.84 (ddd, *J* = 9.9, 3.6, 2.4 Hz, 1H), 5.31 (ddd, *J* = 9.9, 2.4, 1.2 Hz, 1H), 5.03 (dt, *J* = 16.5, 2.4, 1H), 4.95 (ddd, *J* = 16.5, 3.6, 1.2 Hz, 1H), 2.16-2.10 (m, 1H), 2.05 (dd, *J* = 7.6, 4.8 Hz, 1H), 1.45 (dd, *J* = 8.5, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.3, 136.9, 132.3, 128.7, 128.5, 127.3, 126.10, 126.06, 120.8, 69.1, 36.6, 28.8, 24.9; IR (neat) 3020, 1722 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{14}O_2$ 226.0994, found 226.0989 (M^+).
E-**3d**: Clear oil, HPLC (OD-H, 90:10 hexanes/2-propanol, 1 mL/min) retention times of 15.0 min (minor) and 18.9 min (major), 73% ee; $[\alpha]_D^{22}$ +59.3° (*c* = 0.69, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.36-7.21 (comp, 5H), 6.60 (d, *J* = 15.8 Hz, 1H), 5.96 (dd, *J* = 15.8, 8.3 Hz, 1H), 5.86 (dt, *J* = 10.0, 2.9, 1H) 5.46 (dt, *J* = 10.0, 2.1 Hz, 1H), 5.02-5.01 (comp, 2H), 2.76-2.70 (m, 1H), 2.11 (dd, *J* = 8.9, 4.6 Hz, 1H), 1.28 (dd, *J* = 7.3, 4.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 136.6, 133.7, 128.6, 127.6, 126.0, 125.7, 125.9, 121.7, 69.1, 34.4, 27.4, 25.0; IR (neat) 3024, 1721 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{14}O_2$ 226.0994, found 226.0989 (M^+).

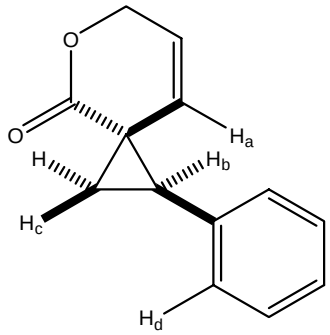
Reduction and Cope rearrangement of 3d: Unseparated *E*,*Z*- **3d** (5:1 *E*:*Z*) (203 mg, 0.90 mmol) was stirred in anhydrous THF (10 mL) at 0 °C. $LiAlH_4$ (93 mg, 2.7 mmol) was added portionwise and the reaction mixture was heated to reflux under nitrogen. After 36 h the solution was cooled to 0 °C and quenched *via* dropwise addition of water until gas was no longer observed evolving. An aqueous solution of NaOH (0.5 mL, 50 wt%) was added, and the mixture was allowed to stir for 10 min. Anhydrous Na_2SO_4 was added and the mixture stirred 2h. The resulting aluminium salts were removed by filtration through Celite, washing with Et_2O five times (20 mL). Solvent was removed from the filtrate by rotary evaporation, and the residue was recrystallized from a minimal amount of dichloromethane and hexanes to yield **4** (110 mg, 53%, 92% ee) as a white crystalline solid: mp 114 °C, GC: (B-DM, 30m x 0.25 mm; 0.25 μm film, 140 °C/10 min, 1 °C/min ramp to 160 °C) retention times of 68.7 min (minor) and 70.5 min (major), 92% ee; $[\alpha]_D^{20}$ -60.6° (*c* = 0.34, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.30-7.20 (comp, 5H), 5.80-5.73 (comp, 2H), 5.61-5.57 (m, 1H), 4.05 (s, 2H), 3.58-3.53 (m, 1H), 3.43 (dd, *J* = 10.4, 4.9 Hz, 1H), 3.37 (dd, *J* = 10.4, 6.2 Hz, 1H), 3.13-3.08 (m, 1H), 2.95-2.89 (m, 1H), 2.77 (dd, *J* = 18.9, 6.5 Hz, 1H), 1.99 (br s, 1H), 1.67 (br s, 1H); ^{13}C NMR (100 MHz,

$CDCl_3$) δ 144.0, 142.9, 133.8, 128.5, 128.2, 126.8, 126.5, 126.2, 68.0, 65.0, 45.6, 45.0, 28.6; IR (neat) 3020 cm^{-1}.

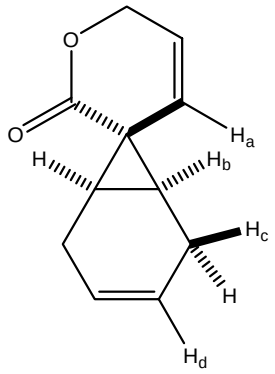
Determination of absolute stereochemistry of 2a: A sample of **2a**,**b** (9:1, 84 mg, 0.48 mmol) obtained from the Rh_2(4*S*,*R*-MenthAZ) catalyzed C-H insertion (80% ee as measured by GC) was stirred in toluene (10 mL). 2,3-Dichloro-5.6-dicyano-1,4-benzoquinone (216 mg, 0.96 mmol) was added. After 2 h, the toluene was evaporated and the reaction mixture was quickly passed through a short silica gel plug (3:1 hexane/ethyl acetate) to remove a bright red colored baseline impurity. The resulting solution was evaporated to a yellow oil. Ethyl acetate (5 mL) was added, followed by 10% Pd/C (10 mg). The atmosphere was purged and replaced with hydrogen. After stirring 12 h, the mixture was filtered through a celite pad, washing with ethyl acetate, and evaporated. Column chromatography (4:1 hexane:ethyl acetate, Rf = 0.30) provided 2-phenyl-δ-valerolactone (71 mg, 86% yield).[4] The optical rotation of 2-phenyl-δ-valerolactone was measured: $[\alpha]_D^{23}$ +14.1° (*c* = 0.28, $CHCl_3$). A previous report lists the optical rotation of the *R* enantiomer (72% ee) to be: $[\alpha]_D^{22}$ +32.6° (*c* = 0.424, $CHCl_3$), indicating the absolute stereochemistry of the predominant enantiomer of **2a** to be *R*.[5]

[4] Betancourt de Perez, R. M.; Fuentes, L. M.; Larson, G. L.; Barnes, C. L.; Heeg, M. J. *J. Org. Chem.* **1986**, *51*, 2039.
[5] Nakamura, Y.; Takeuchi, S.; Ohgo, Y.; Yamaoka, M.; Yoshida, A.; Mikami, K. *Tetrahedron* **1999**, *55*, 4959.

Determination of relative stereochemistry of 2b, 3a:
nOe experiments were performed on a Bruker AM-400 instrument using a Gauss1.1000 shape pulse and gradient selection. Observed correlations were used to assign the relative stereochemistry of **2b**, **3a** as those shown.



Irradiated Signal (ppm)/H_x	**Correlated Signals (ppm)/H_x**	**% Correlation**
4.96/H_a	1.56/H_c	1.3
	7.21 (d, *J* = 7.3 Hz)/H_d	0.8
	3.25/H_b	0
3.25/H_b	7.21 (d, *J* = 7.3 Hz)/H_e	1.1
1.56/H_c	4.96/H_a	0.9
	7.21 (d, *J* = 7.3 Hz)/H_d	2.1



Irradiated Signal (ppm)/H_x	**Correlated Signals (ppm)/H_x**	**% Correlation**
5.63/H_d	5.42/H_a	0.1
5.42/H_a	5.63/H_d	0.3
	2.09-2.01/H_c	1.1
	2.19-2.16/H_b	0
2.09-2.01/H_c	5.42/H_a	0.7

4.98
4.99
4.99
4.99
5.36
5.36
5.37
5.38
5.39
5.40
6.18
6.18
6.19
6.20
6.21
6.21
2.16
1.00
0.97
5.0
0.0
ppm (t1)
N2
O
O
1

2.59
2.60
2.60
2.61
2.61
2.62
3.06
3.06
3.07
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4.80
4.81
5.45
5.58
5.74
5.74
5.88
5.88
5.89
2a
O
O
2.30
0.88
0.86
2.00
1.82
3.56
0.0
5.0
ppm (t1)

2.03
2.03
2.07
2.08
2.17
2.17
2.18
2.19
2.46
2.51
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5.63
5.93
5.93
5.94
5.96
2.07
1.89
2.00
1.95
0.98
1.95
1.00
0.0
5.0
ppm (t1)
O
O
2b

1.54
1.56
1.56
1.58
2.12
2.13
2.14
2.15
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3.27
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4.94
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4.97
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5.72
5.73
5.74
7.20
7.35
1.09
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1.00
1.00
2.09
1.02
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0.0
5.0
ppm (t1)
Ph
O
O
3a

1.25
1.27
1.27
1.28
1.49
1.50
1.50
1.51
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5.55
5.56
5.56
5.78
5.78
5.79
5.80
5.81
5.82
3b
1.20
10.00
0.91
2.00
1.02
1.04
1.02
0.0
5.0
ppm (t1)

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1.47
1.48
1.91
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1.96
1.97
1.98
2.70
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7.23
7.25
7.32
7.34
2.88
1.01
0.94
2.07
0.98
0.98
1.00
5.30
0.0
5.0
10.0
15.0
ppm (t1)
Ph
O
O
E-3c

1.27
1.28
1.28
1.30
2.09
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Ph
O
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E-3d
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1.01
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5.0
ppm (t1)

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1.47
2.03
2.05
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2.06
2.10
2.12
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Ph
O
O
Z-3d
1.12
1.07
1.12
2.33
1.00
0.93
0.96
1.04
5.33
0.0
5.0
ppm (t1)

2.73
2.74
2.75
2.75
2.78
2.78
2.80
2.80
2.89
2.90
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5.57
5.57
5.61
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5.73
5.74
5.74
5.80
7.24
7.26
7.29
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2.00
1.00
1.98
5.24
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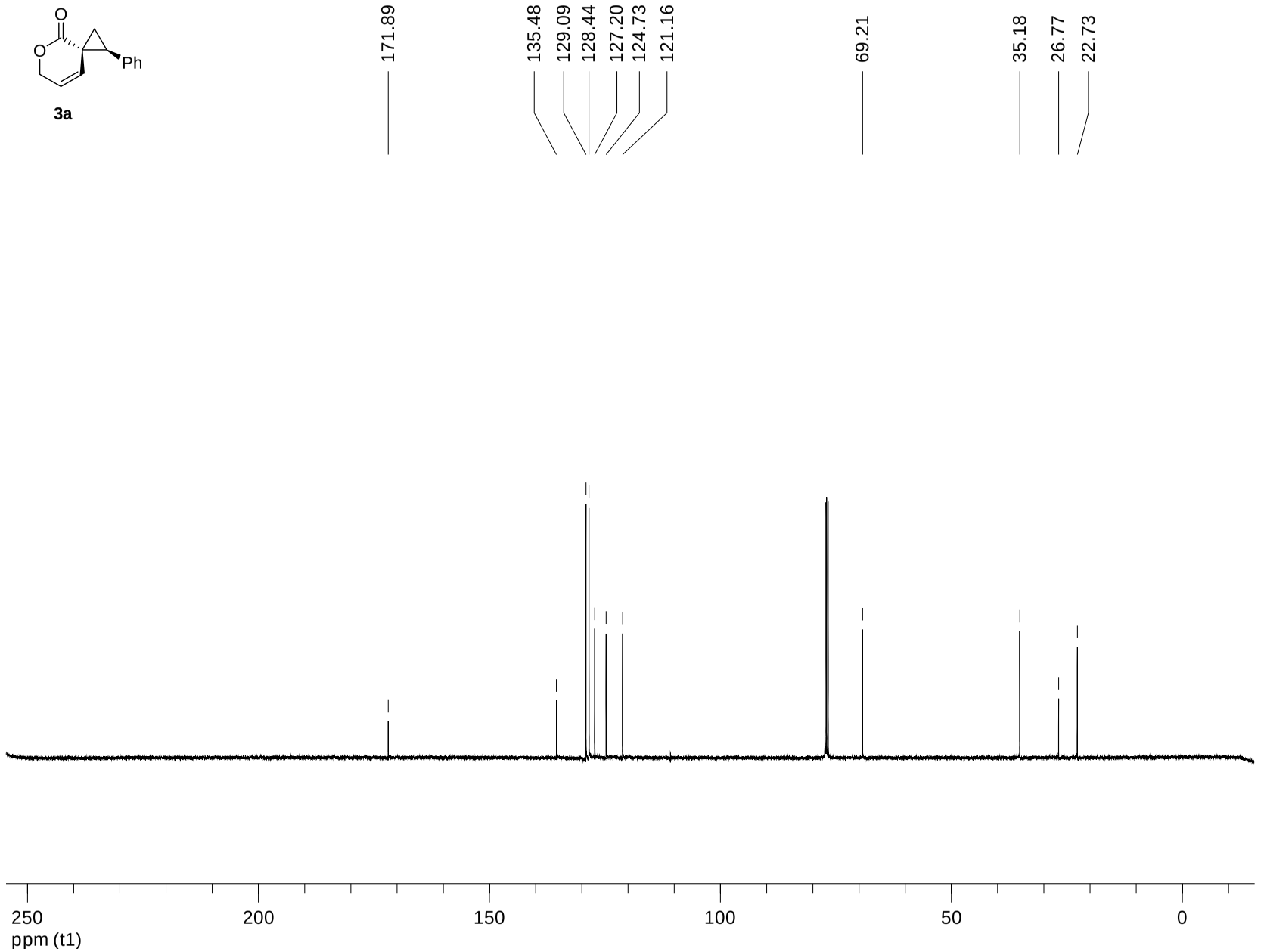
5.0
ppm (t1)
OH
OH
Ph
4

70.5
113.5
114.5
164.5
0
50
100
150
200
250
ppm (t1)
O
O
N2
1

170.6
127.5
127.1
125.7
124.4
123.9
122.9
68.8
44.4
38.1
26.2
2a
250
200
150
100
50
0
ppm (t1)

172.7
124.3
123.0
121.3
68.7
28.2
26.3
19.7
2b
250
200
150
100
50
0
ppm (f1)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O
Ph
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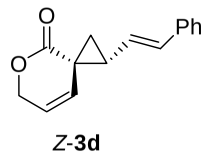
135.48
129.09
128.44
127.20
124.73
121.16
69.21
35.18
26.77
22.73
250
200
150
100
50
0
ppm (t1)

172.5
133.0
125.1
125.0
120.6
69.1
37.5
29.5
25.1
22.7
22.3
22.3
3b
0
50
100
150
200
250
ppm (t1)

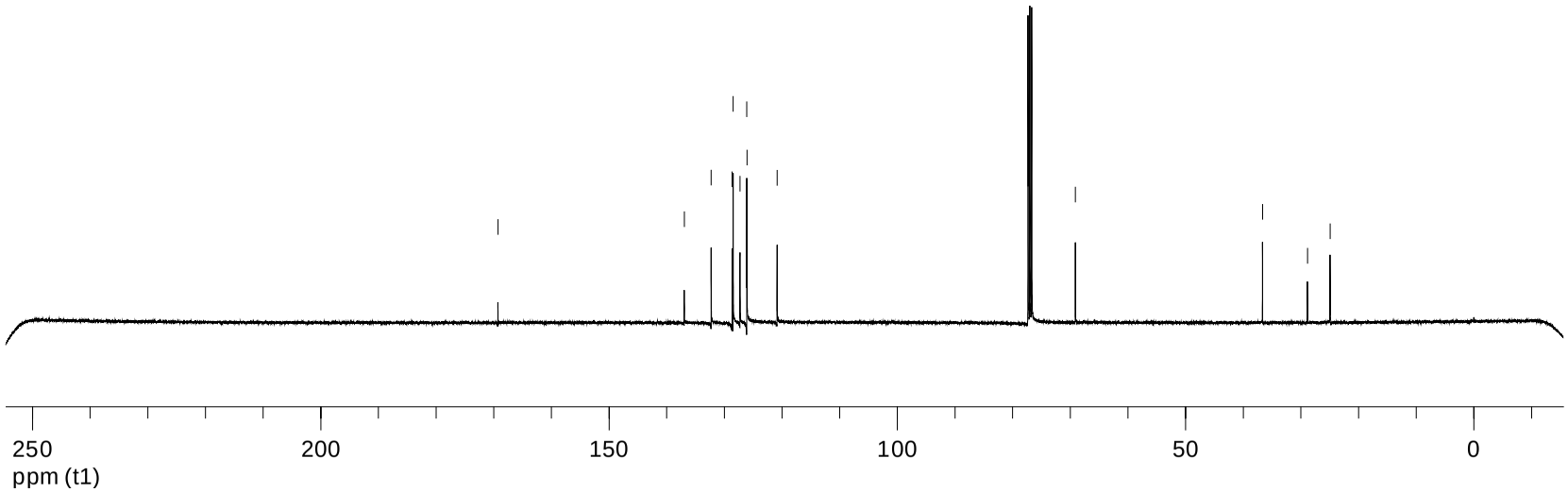
Ph
O
O
E-3c
172.2
137.1
133.6
128.8
128.2
128.0
126.6
124.6
121.2
69.1
39.7
25.8
23.1
19.1
250
200
150
100
50
0
ppm (t1)

171.6
136.6
133.7
128.6
127.6
126.0
125.7
124.9
121.7
69.1
34.4
27.4
25.0
Ph
O
O
E-3d
ppm (t1)
200
150
100
50
0

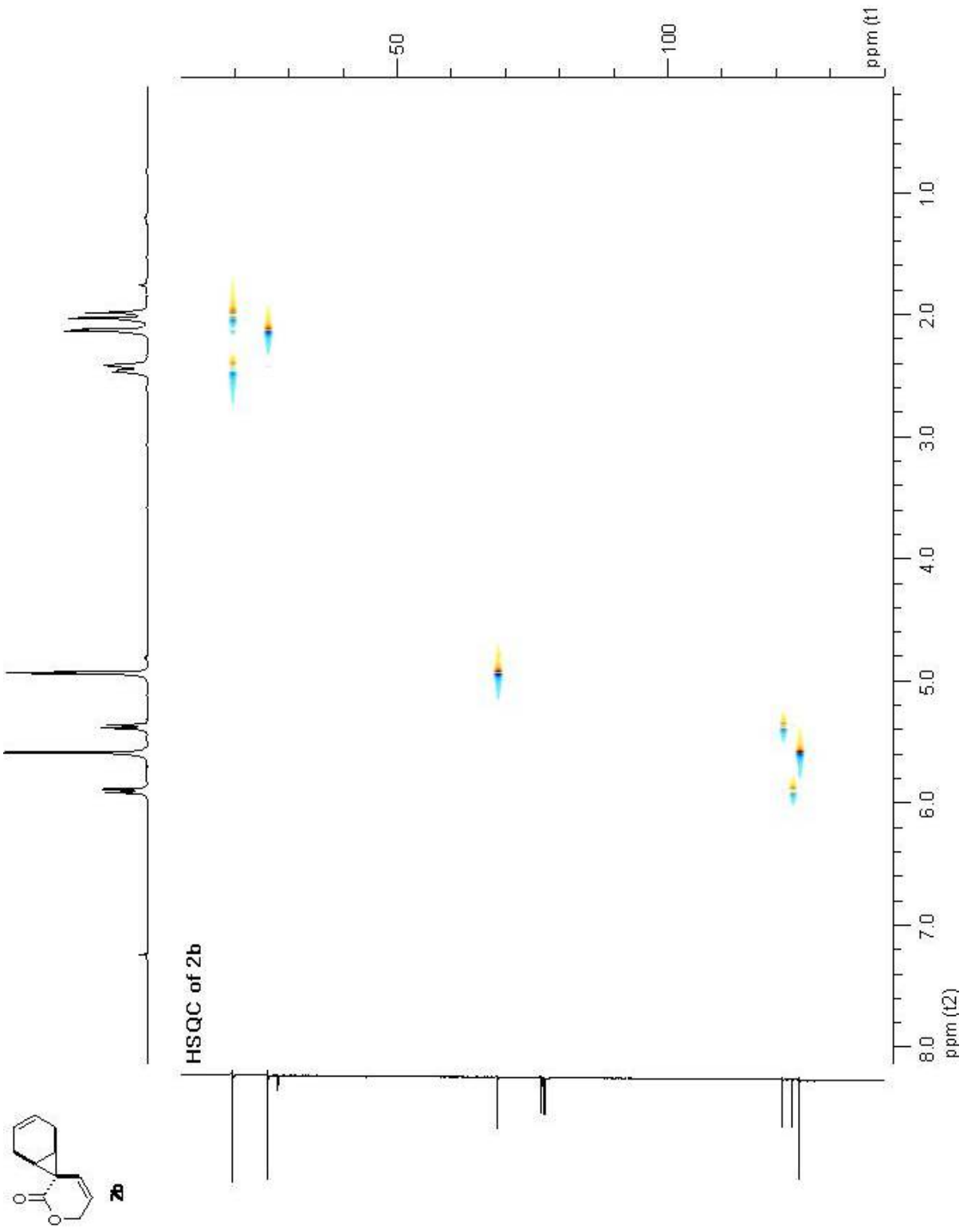


Z-**3d**

169.3
136.9
132.3
128.7
128.5
127.3
126.1
126.1
120.8
69.1
36.6
28.8
24.9



144.0
142.9
133.8
128.5
128.2
126.8
126.5
126.2
68.0
65.0
45.6
45.0
28.6
0
50
100
150
200
250
ppm (t1)
OH
OH
Ph
4


HSQC of 2b
50
100
ppm (t1)
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
ppm (t2)
O
O
O