Structure of *n*-Butyllithium in Mixtures of Ethers and Diamines: Influence of Mixed Solvation on 1,2-Additions to Imines

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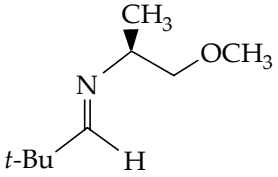
Supporting Information

Stereoselectivity of the 1,2-Addition Reactions

X-ray Crystallographic Data of 2•HCl

Structure Chart



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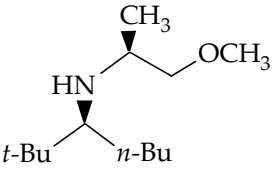

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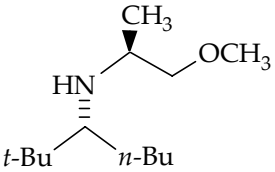

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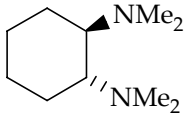
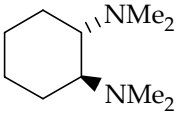
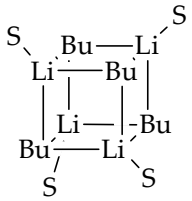


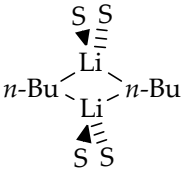
R,R-TMCDA



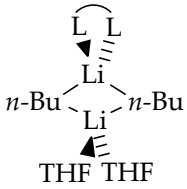
S,S-TMCDA



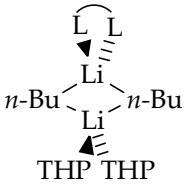
4a; S = THF
4b; S = THP
4c; S = Et_2O



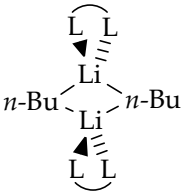
5a; S = THF
5b; S = THP



6a; L⌒L = TMEDA
6b; L⌒L = *R,R*-TMCDA

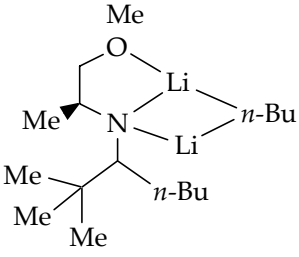


6c; L⌒L = *R,R*-TMCDA



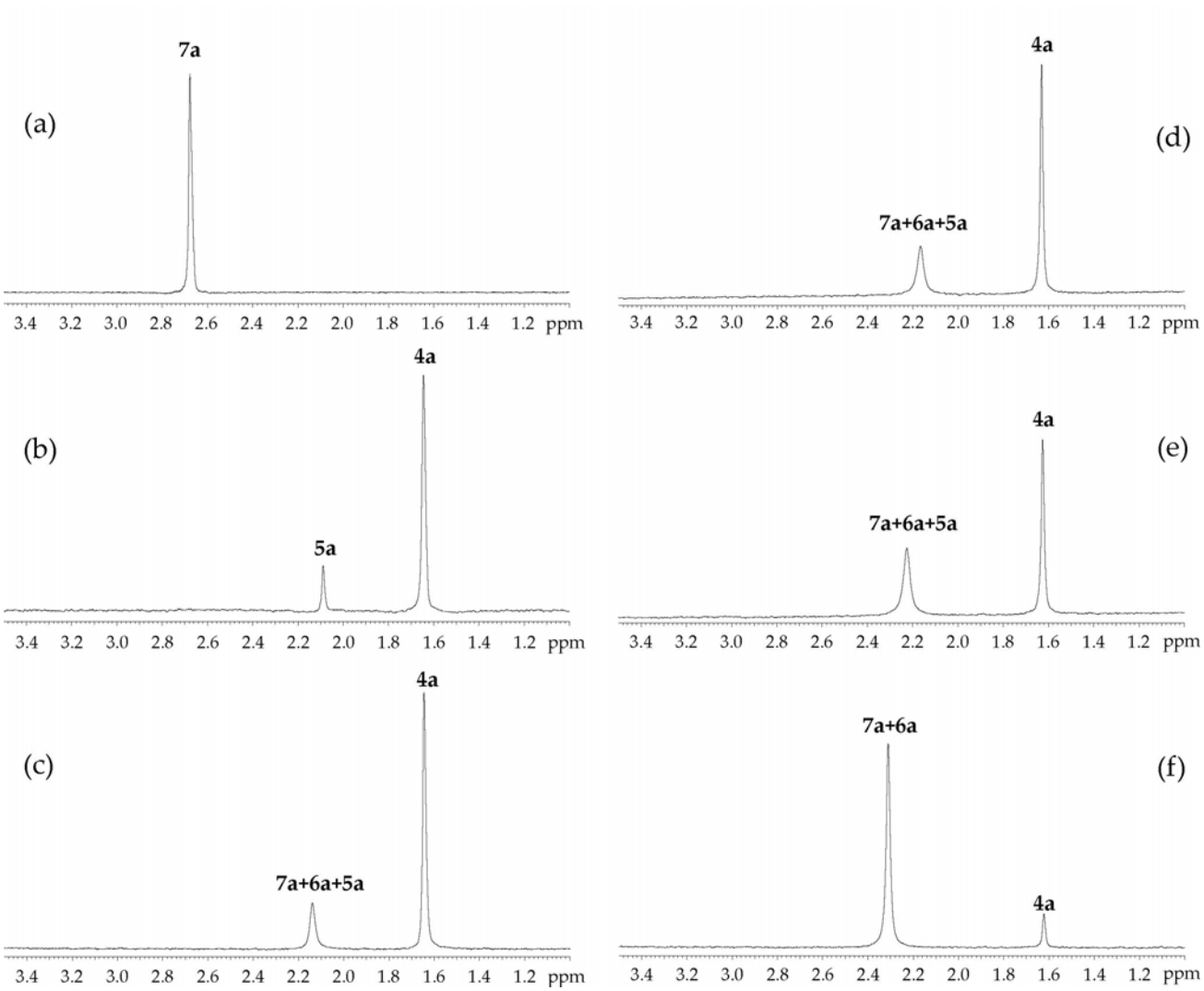
7a; L⌒L = TMEDA

7b; L⌒L = *R,R*-TMCDA

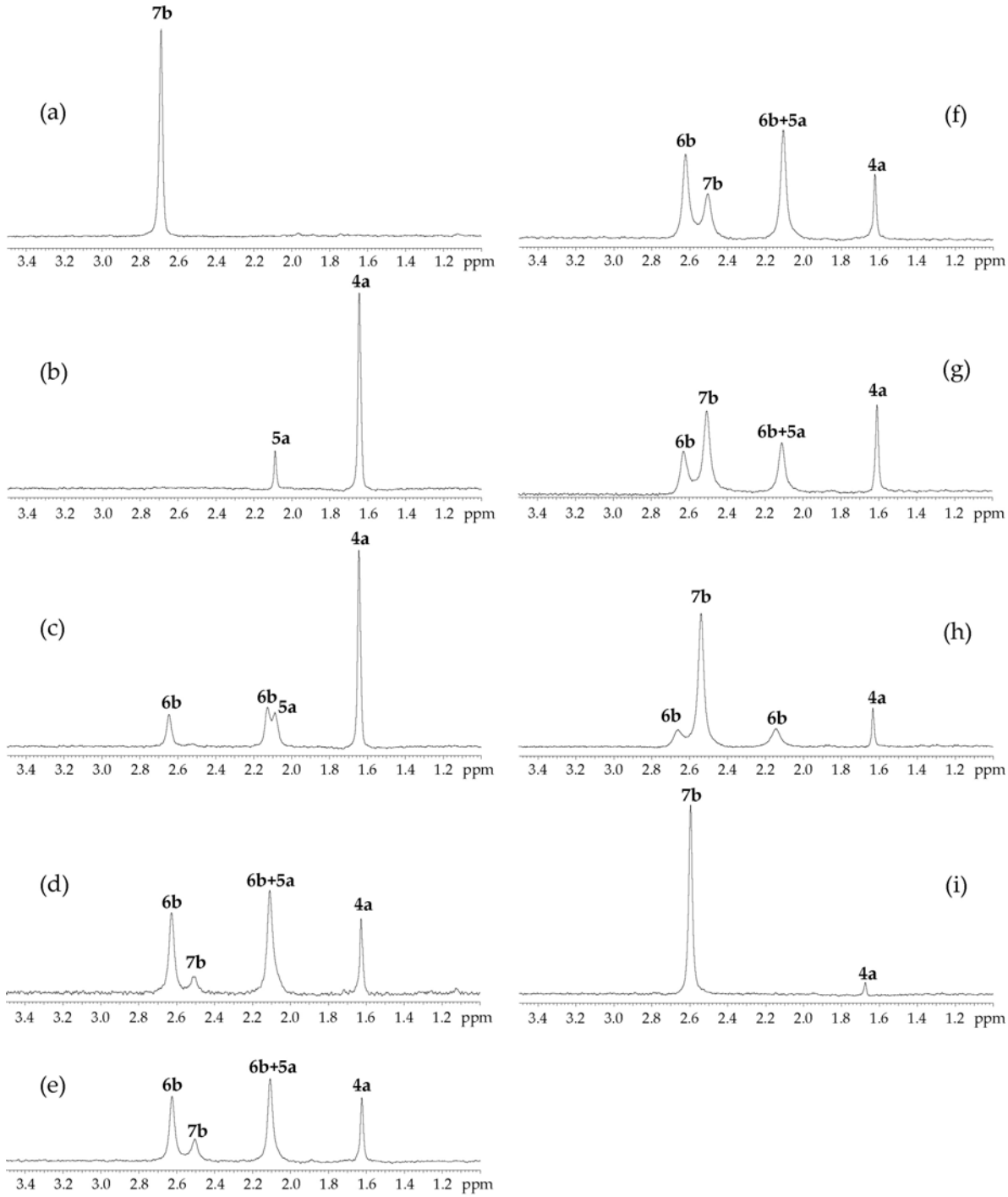


i

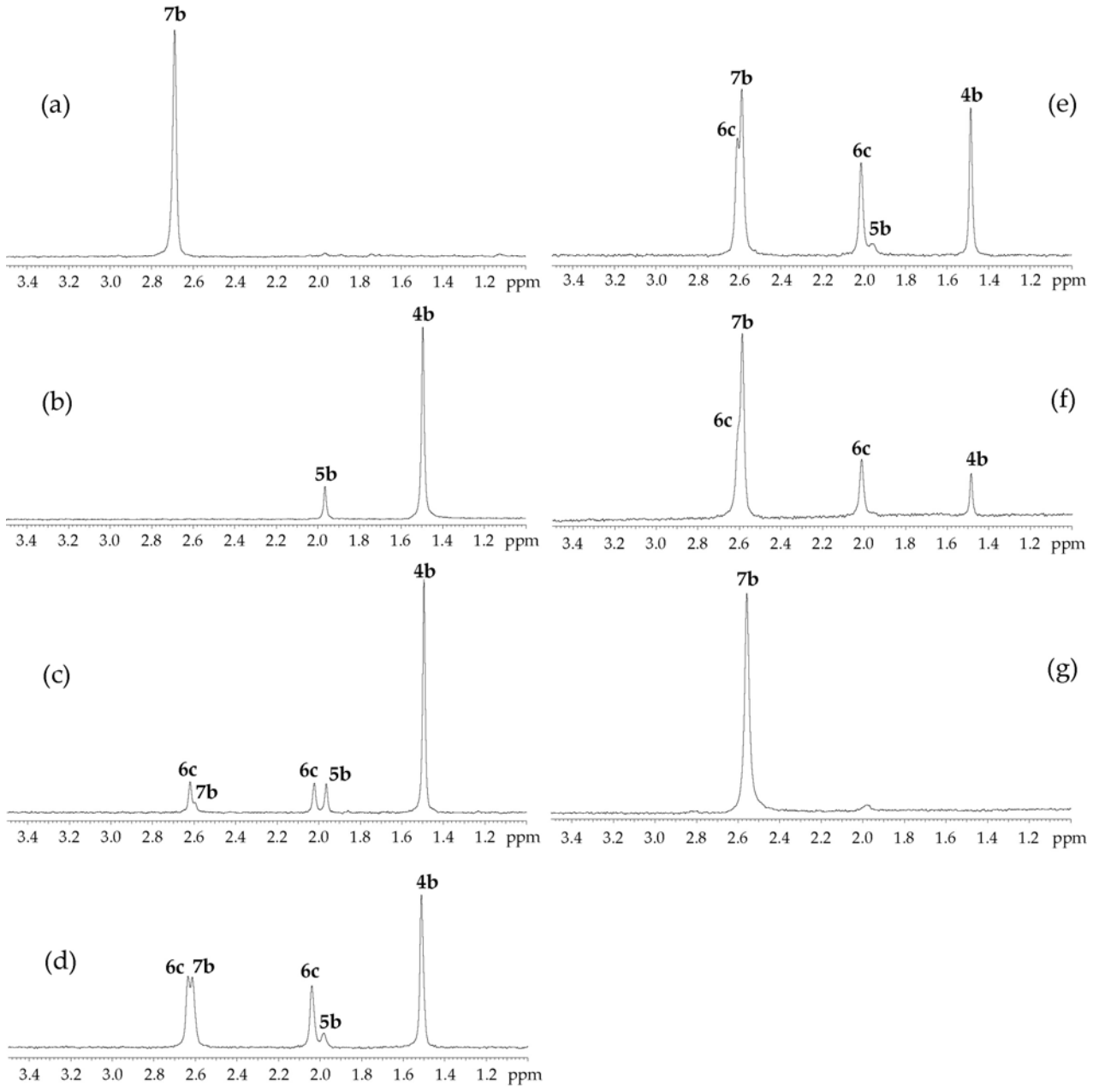
^{6}Li NMR Spectroscopic Studies



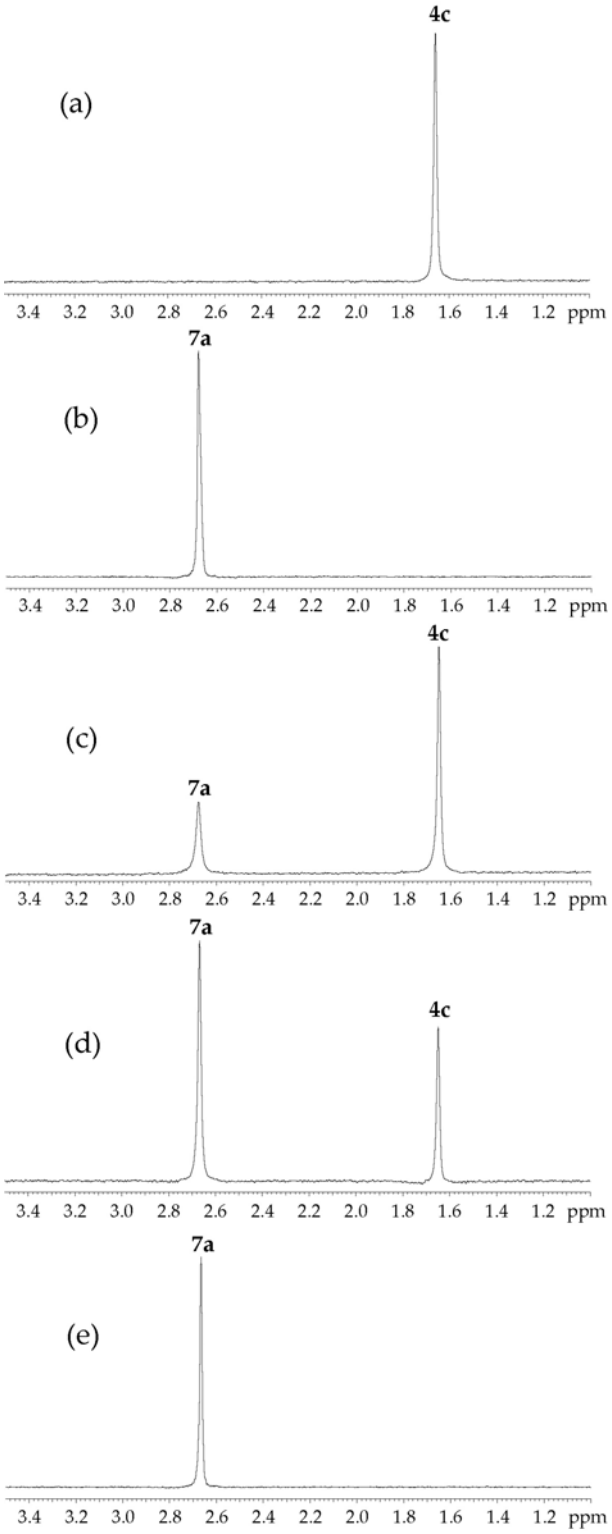
I. ^{6}Li NMR spectra of [^{6}Li]*n*-BuLi recorded on samples containing 0.10 M [^{6}Li]*n*-BuLi in pentane at -115 °C: (a) 0.20 M TMEDA; (b) 5.0 M THF; (c) 5.0 M THF, 0.02 M TMEDA; (d) 5.0 M THF, 0.05 M TMEDA; (e) 5.0 M THF, 0.12 M TMEDA; (f) 5.0 M THF, 0.4 M TMEDA.



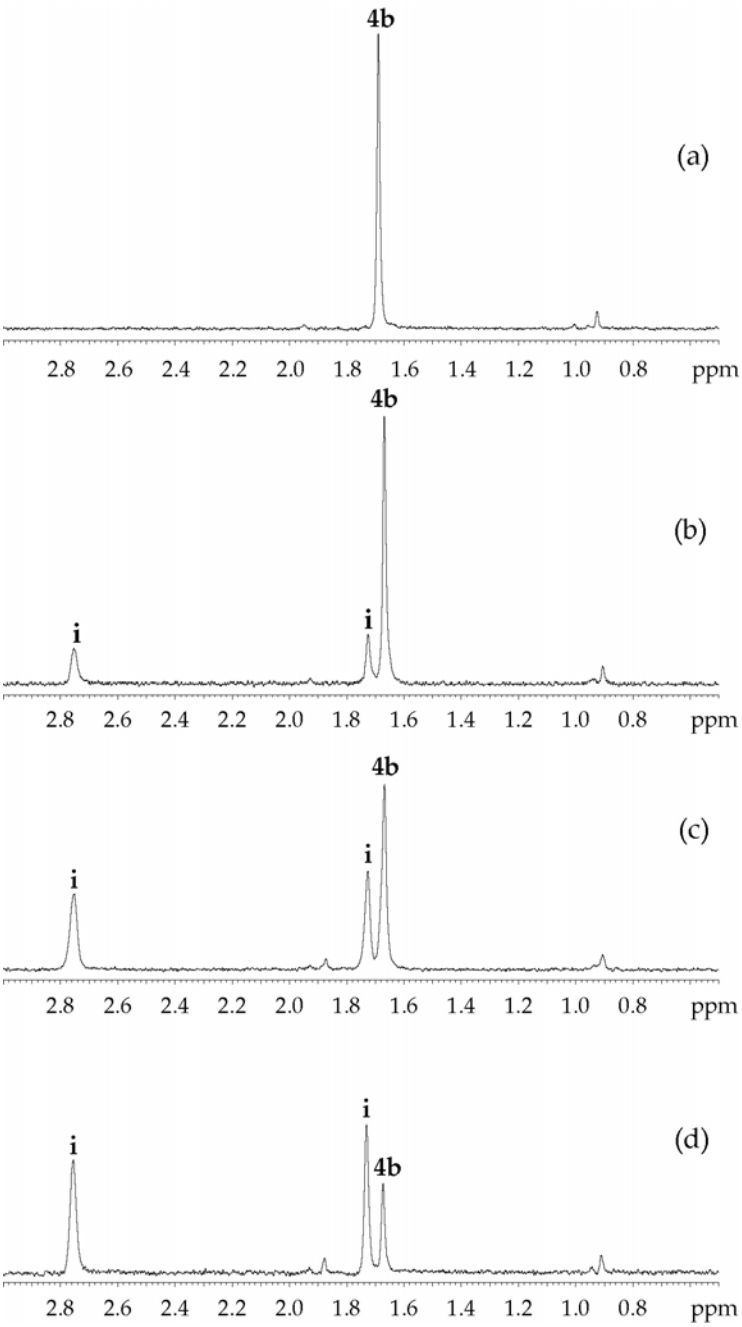
II. ^{6}Li NMR spectra of [^{6}Li]*n*-BuLi recorded on samples containing 0.10 M [^{6}Li]*n*-BuLi in pentane at -115 °C: (a) 0.2 M *R,R*-TMCDA; (b) 5.0 M THF; (c) 0.02 M *R,R*-TMCDA, 5.0 M THF; (d) 0.05 M *R,R*-TMCDA, 5.0 M THF; (e) 0.08 M *R,R*-TMCDA, 5.0 M THF; (f) 0.12 M *R,R*-TMCDA, 5.0 M THF; (g) 0.12 M *R,R*-TMCDA, 2.0 M THF; (h) 0.12 M *R,R*-TMCDA, 1.0 M THF; (i) 0.12 M R,R-TMCDA, 0.2 M THF.



III. ^{6}Li NMR spectra of [^{6}Li]*n*-BuLi recorded on samples containing 0.10 M [^{6}Li]*n*-BuLi in pentane at -115 °C: (a) 0.02 M *R,R*-TMCDA; (b) 5.0 M THP; (c) 5.0 M THP, 0.02 M *R,R*-TMCDA; (d) 5.0 M THP, 0.05 M *R,R*-TMCDA; (e) 5.0 M THP, 0.08 M *R,R*-TMCDA; (f) 5.0 M THP, 0.12 M *R,R*-TMCDA; (g) 5.0 M THP, 0.5 M *R,R*-TMCDA.

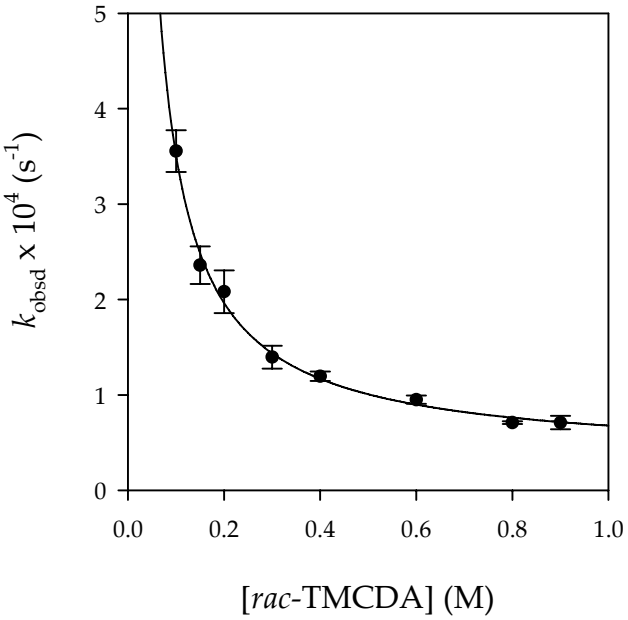


IV. ^{6}Li NMR spectra of 0.10 M [^{6}Li]*n*-BuLi at -115 °C containing: (a) 5.0 M Et_2O; (b) 0.2 M TMEDA; (c) 5.0 M Et_2O and 0.02 M TMEDA; (d) 5.0 M Et_2O and 0.05 M TMEDA; (e) 5.0 M Et_2O and 0.12 M TMEDA.



V. ^{6}Li NMR spectra of 0.10 M [^{6}Li]*n*-BuLi at -90 °C containing 0.50 M THP and imine **1** in pentane cosolvent: (a) 0.0 M **1**; (b) 0.02 M **1**; (c) 0.05 M **1**; (d) 0.08 M **1**.

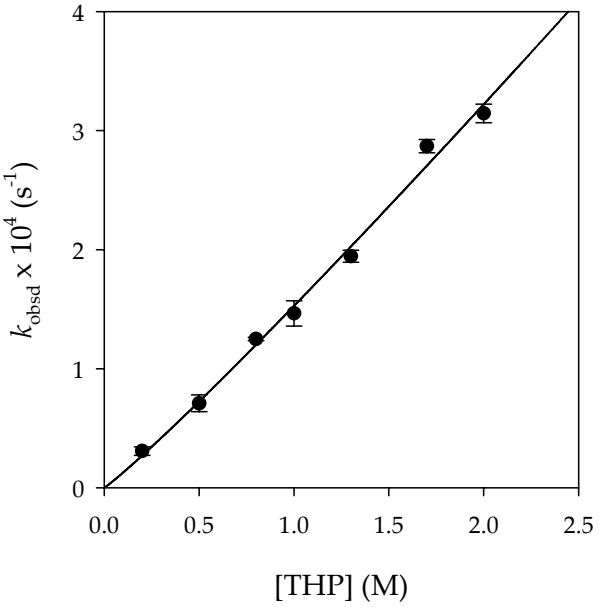
Rate Studies



VI. Plot of k_{obsd} vs [*rac*-TMCDA] for the 1,2-addition of *n*-BuLi (0.10 M) to imine **1** (0.007 M) in 0.50 M THP and toluene cosolvent at -78 °C. The curve depicts an unweighted least-squares fit to $k_{obsd} = a[rac\text{-TMCDA}]^n + b$ ($a = 4.0 \pm 1.0 \times 10^{-5}$, $n = -0.97 \pm 0.12$, $b = 3.3 \pm 2.0 \times 10^{-5}$)

VII. Data for plot in Section VI.

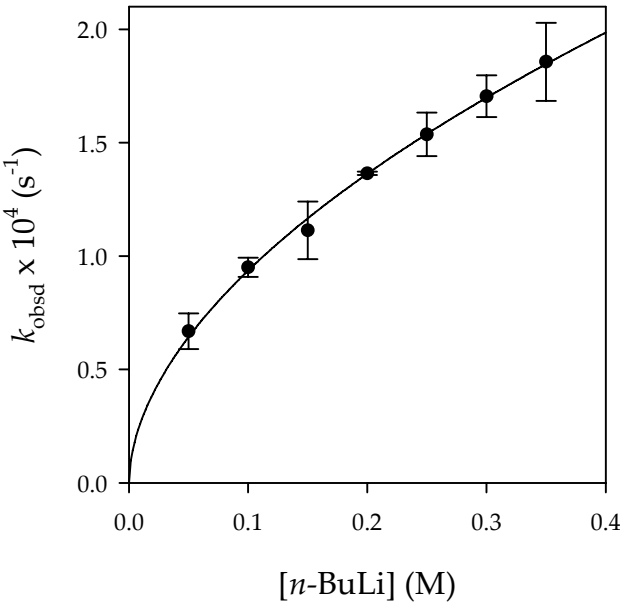
[*rac*-TMCDA] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} 3 (s^{-1})	k_{obsd} (av) (s^{-1})
0.10	0.000371 ± 5E-6	0.00034 ± 1E-5		0.00036 ± 2E-5
0.15	0.000222 ± 3E-6	0.000250 ± 3E-6		0.00024 ± 2E-5
0.20	0.0001845 ± 8E-7	0.000229 ± 3E-6	0.000211 ± 2E-6	0.00021 ± 2E-5
0.30	0.0001310 ± 6E-7	0.000148 ± 1E-6		0.00014 ± 1E-5
0.40	0.000123 ± 1E-6	0.000116 ± 3E-6		0.000120 ± 5E-6
0.60	0.000098 ± 2E-6	0.000092 ± 2E-6		0.000095 ± 4E-6
0.80	0.000072 ± 1E-6	0.000070 ± 1E-6		0.000071 ± 1E-6
0.90	0.000066 ± 2E-6	0.000076 ± 1E-6		0.000071 ± 7E-6



VIII. Plot of k_{obsd} vs [THP] for the 1,2-addition of *n*-BuLi (0.10 M) to imine **1** (0.007 M) in 0.90 M *rac*-TMCDA and toluene cosolvent at -78 °C. The curve depicts an unweighted least-squares fit to $k_{obsd} = a[THP]^n$ ($a = 1.53 \pm 0.05$ x 10^{-4}, $n = 1.08 \pm 0.05$).

IX. Data for plot in Section VIII.

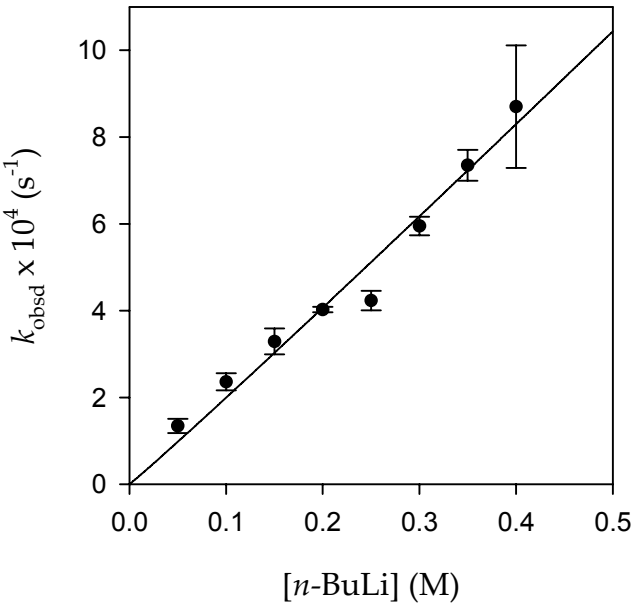
[THP] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} 3 (s^{-1})	k_{obsd} (av) (s^{-1})
0.20	0.0000284 ± 5E-7	0.0000334 ± 4E-7		0.000031 ± 4E-6
0.50	0.000066 ± 2E-6	0.000076 ± 1E-6		0.000071 ± 7E-6
0.80	0.000126 ± 1E-6	0.000124 ± 1E-6		0.000125 ± 1E-6
1.00	0.000139 ± 1E-6	0.000154 ± 2E-6		0.000146 ± 1E-5
1.30	0.000191 ± 2E-6	0.000198 ± 2E-6		0.0001945 ± 5E-6
1.70	0.000227 ± 3E-6	0.000339 ± 8E-6	0.000291 ± 3E-6	0.0002857 ± 2E-5
2.00	0.000320 ± 6E-6	0.000309 ± 6E-6		0.0003145 ± 8E-6



X. Plot of k_{obsd} vs [*n*-BuLi] for the 1,2-addition to imine **1** (0.007 M) in 0.60 M *rac*-TMCDA and 0.50 M THP and toluene cosolvent at -78 °C. The curve depicts an unweighted least-squares fit to $k_{obsd} = a[n\text{-BuLi}]^b$ (a = 3.3 ± 0.1 x 10^{-4}, b = 0.54 ± 0.02).

XI. Data for plot in Section X.

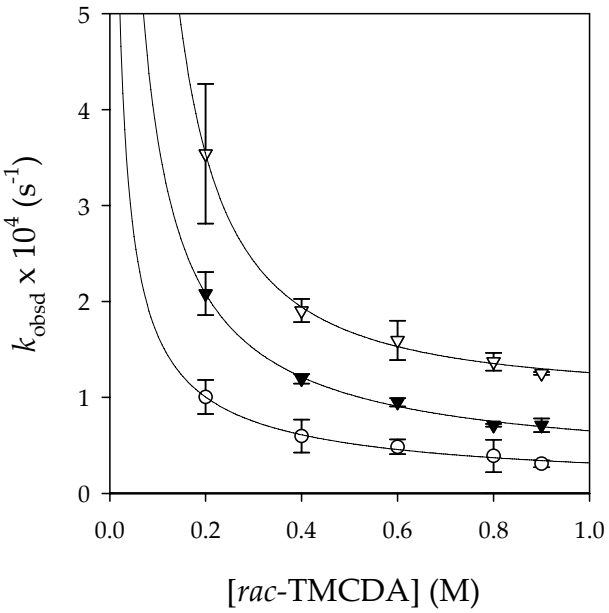
[*n*-BuLi] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} 3 (s^{-1})	k_{obsd} (av) (s^{-1})
0.05	0.000075 ± 5E-6	0.0000593 ± 5E-7	0.0000662 ± 6E-7	0.000067 ± 8E-6
0.10	0.000098 ± 2E-6	0.000092 ± 2E-6		0.000095 ± 4E-6
0.15	0.000121 ± 1E-6	0.000116 ± 2E-6	0.000097 ± 2E-6	0.00011 ± 1E-5
0.20	0.000137 ± 2E-6	0.000136 ± 2E-6		0.0001365 ± 7E-7
0.25	0.000152 ± 2E-6	0.000145 ± 2E-6	0.000164 ± 2E-6	0.00015 ± 1E-5
0.30	0.000164 ± 2E-5	0.000177 ± 2E-6		0.000171 ± 9E-6
0.35	0.000205 ± 2E-6	0.000180 ± 2E-6	0.000172 ± 2E-6	0.00019 ± 2E-5



XII. Plot of k_{obsd} vs [*n*-BuLi] for the 1,2-addition to imine **1** (0.007 M) in 0.15 M *rac*-TMCDA and 0.50 M THP and toluene cosolvent at -78 °C. The curve depicts an unweighted least-squares fit to $k_{obsd} = a[n\text{-BuLi}]^b$ (a = 2.1 ± 0.3 x 10^{-3}, b = 1.03 ± 0.10). Alternatively, fit to $k_{obsd} = a[n\text{-BuLi}]^b + k'[n\text{-BuLi}]^{1/2}$ (a = 2.1 ± 0.3 x 10^{-3}, b = 1.05 ± 0.10), k' = 3.3 x 10^{-5} which is the b term from Section VI).

XIII. Data for plot in Section XII.

[*n*-BuLi] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} (av) (s^{-1})
0.05	0.000146 ± 5E-6	0.000123 ± 2E-6	0.00013 ± 2E-5
0.10	0.000222 ± 3E-6	0.000222 ± 3E-6	0.00024 ± 2E-5
0.15	0.000308 ± 4E-6	0.000350 ± 5E-6	0.000329 ± 3E-5
0.20	0.000407 ± 7E-6	0.000398 ± 8E-6	0.000403 ± 6E-6
0.25	0.000439 ± 8E-6	0.000407 ± 7E-6	0.00042 ± 2E-5
0.30	0.00061 ± 2E-5	0.000580 ± 9E-6	0.000595 ± 2E-5
0.35	0.00071 ± 1E-5	0.00076 ± 2E-5	0.00074 ± 4E-5
0.40	0.00077 ± 2E-5	0.00097 ± 2E-5	0.0009 ± 1E-4



XIV. Plot of k_{obsd} vs [*rac*-TMCDA] for the 1,2-addition of *n*-BuLi (0.10 M) to imine **1** (0.007 M) in toluene cosolvent at -78 °C with variable THP concentrations. ▽ 0.80 M THP, ▼ 0.50 M THP, ○ 0.20 M THP. The curves depict unweighted least-squares fits to $k_{obsd} = a[rac\text{-TMCDA}]^n + b$.

XV. Data for plot in Section XIV.

0.80 M THP

$k_{obsd} = a[rac\text{-TMCDA}]^n + b$ ($a = 2.5 \pm 1 \times 10^{-5}$, $n = -1.4 \pm 0.2$, $b = 1.0 \pm 0.2 \times 10^{-4}$).

[*rac*-TMCDA] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} 3 (s^{-1})	k_{obsd} (av) (s^{-1})
0.20	0.000276 ± 6E-6	0.00042 ± 1E-5	0.000366 ± 4E-6	0.00035 ± 7E-5
0.40	0.000182 ± 5E-6	0.000199 ± 2E-6		0.00019 ± 1E-5
0.60	0.000174 ± 4E-6	0.000145 ± 3E-6		0.00016 ± 2E-5
0.80	0.000143 ± 4E-6	0.000141 ± 6E-6	0.000128 ± 3E-6	0.000137 ± 9E-6
0.90	0.000126 ± 1E-6	0.000124 ± 1E-6		0.000125 ± 1E-6

0.50 M THP

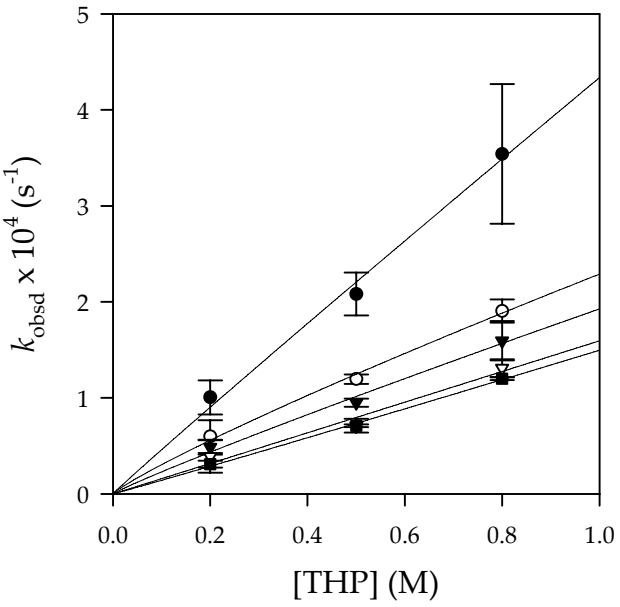
$k_{obsd} = a[rac\text{-TMCDA}]^n + b$ ($a = 4.0 \pm 2.0 \times 10^{-5}$, $n = -0.9 \pm 0.2$, $b = 2.0 \pm 2.0 \times 10^{-5}$).

[*rac*-TMCDA] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} 3 (s^{-1})	k_{obsd} (av) (s^{-1})
0.20	0.000185 ± 1E-6	0.000229 ± 3E-6	0.000211 ± 2E-6	0.00021 ± 2E-5
0.40	0.000123 ± 1E-6	0.000116 ± 3E-6		0.000120 ± 5E-6
0.60	0.000098 ± 2E-6	0.000092 ± 2E-6		0.000095 ± 4E-6
0.80	0.000072 ± 1E-6	0.000070 ± 1E-6		0.000071 ± 1E-6
0.90	0.000076 ± 1E-6	0.000066 ± 2E-6		0.000071 ± 7E-6

0.20 M THP

$k_{obsd} = a[rac\text{-TMCDA}]^n + b$ ($a = 4.0 \pm 3.0 \times 10^{-5}$, $n = -0.7 \pm 0.3$, $b = 0.0 \pm 3.0 \times 10^{-5}$).

[*rac*-TMCDA] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} (av) (s^{-1})
0.20	0.000088 ± 2E-6	0.000113 ± 1E-6	0.00010 ± 2E-5
0.40	0.0000718 ± 9E-7	0.0000476 ± 4E-7	0.00006 ± 2E-5
0.60	0.000054 ± 3E-6	0.0000431 ± 6E-7	0.000049 ± 8E-6
0.80	0.000051 ± 3E-6	0.000027 ± 2E-6	0.00004 ± 2E-5
0.90	0.0000284 ± 5E-7	0.0000334 ± 4E-7	0.000031 ± 4E-6



XVI. Plot of k_{obsd} vs [THP] for the 1,2-addition of *n*-BuLi (0.10 M) to imine **1** (0.007 M) in toluene cosolvent at -78 °C with *rac*-TMCDA. ● 0.20 M *rac*-TMCDA, ○ 0.40 M *rac*-TMCDA, ▼ 0.60 M *rac*-TMCDA, ▽ 0.80 M *rac*-TMCDA, ■ 0.90 M *rac*-TMCDA. The curves depict unweighted least-squares fits to $k_{obsd} = a[\text{THP}]^n$.

XVII. Data for plot in Section XVI.

0.20 M *rac*-TMCDA

$k_{obsd} = a[THP]^n$ (a = 4.3 ± 0.3 x 10^{-4}, n = 1.0 ± 0.1).

[THP] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} 3 (s^{-1})	k_{obsd} (av) (s^{-1})
0.2	0.000088 ± 2E-6	0.000113 ± 1E-6		0.00010 ± 2E-5
0.5	0.000185 ± 1E-6	0.000229 ± 3E-6	0.000211 ± 2E-6	0.00021 ± 2E-5
0.8	0.000276 ± 6E-6	0.00042 ± 1E-5	0.000366 ± 4E-6	0.00035 ± 7E-5

0.40 M *rac*-TMCDA

$k_{obsd} = a[THP]^n$ (a = 2.3 ± 0.1 x 10^{-4}, n = 0.88 ± 0.08)

[THP] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} (av) (s^{-1})
0.2	0.0000718 ± 9E-7	0.0000476 ± 4E-7	0.00006 ± 2E-5
0.5	0.000123 ± 2E-6	0.000116 ± 3E-6	0.000120 ± 5E-6
0.8	0.000182 ± 5E-6	0.000200 ± 2E-6	0.00019 ± 1E-5

0.6 M *rac*-TMCDA

$k_{obsd} = a[THP]^n$ (a = 1.9 ± 0.1 x 10^{-4}, n = 0.9 ± 0.1).

[THP] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} (av) (s^{-1})
0.2	0.000054 ± 3E-6	0.0000431 ± 6E-7	0.000049 ± 8E-6
0.5	0.000098 ± 2E-6	0.000092 ± 2E-6	0.000095 ± 4E-6
0.8	0.000174 ± 4E-6	0.000145 ± 3E-6	0.00016 ± 2E-5

0.80 M *rac*-TMCDA

$k_{obsd} = a[THP]^n$ ($a = 1.6 \pm 0.2 \times 10^{-4}$, $n = 1.0 \pm 0.2$).

[THP] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} 3 (s^{-1})	k_{obsd} (av) (s^{-1})
0.2	0.000051 ± 3E-6	0.000027 ± 2E-6		0.00004 ± 2E-5
0.5	0.000072 ± 1E-6	0.000070 ± 1E-6		0.000071 ± 1E-6
0.8	0.000143 ± 4E-6	0.000141 ± 6E-6	0.000128 ± 3E-6	0.000137 ± 9E-6

0.90 M *rac*-TMCDA

$k_{obsd} = a[THP]^n$ ($a = 1.50 \pm 0.06 \times 10^{-4}$, $n = 1.03 \pm 0.07$).

[THP] (M)	k_{obsd} 1 (s^{-1})	k_{obsd} 2 (s^{-1})	k_{obsd} (av) (s^{-1})
0.2	0.0000284 ± 5E-7	0.0000334 ± 4E-7	0.000031 ± 4E-6
0.5	0.000076 ± 1E-6	0.000066 ± 2E-6	0.000071 ± 7E-6
0.8	0.000126 ± 1E-6	0.000124 ± 1E-6	0.000125 ± 1E-6

Stereoselectivity of the 1,2-Addition Reactions

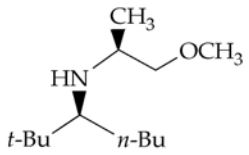
XVIII. Determination of **2/3** ratios.

Stereoselectivities were determined by quenching separate reaction vials as described for the following representative procedure: oven-dried nitrogen-flushed 10 mL serum vials (Kimble) fitted with rubber septa and stir bars were charged sequentially with TMEDA (60.4 μL, 0.10 M), Et_2O (1.26 mL, 6.0 M), toluene (611.0 μL), and *n*-BuLi (58.5 μL from 3.42 M stock solution in hexane, 0.1 M) at -78 °C. A stock solution of substrate was prepared by dissolving 1.01 mL imine **1** (6.36 mmol) in 3.69 mL hexane under argon. The reactions were initiated by adding an aliquot of the imine stock solution (8.0 μL, 0.011 mmol of **1**) to each vial. The reactions were quenched after 8 – 10 h by adding 2.0 mL of 10% NaOH aqueous solution and allowing the reactions to warm to room temperature. The organic layer was removed for direct GC analysis. The ratio of **2**/**3** was calculated from the integrations of the GC data.

A preparative scale reaction was effected as follows: To a nitrogen-flushed 100-mL round-bottom Schlenk flask fitted with a rubber septum and a stir bar were added 25.0 mL Et_2O, 4.53 mL (0.03 mol) TMEDA, and 1.60 mL (0.01 mol) imine **1**. After cooling at -78 °C with a dry ice/acetone bath for 40 min, 19.0 mL (0.03 mol) of 1.60 M *n*-BuLi was added slowly to the flask. The reaction was done in 4 hr before quenched with 10% NaOH aqueous solution. The organic phase was concentrated, and the resulting oil was flash chromatographied (7:1 hexane/Et_2O) to give 1.51 g (70 % yield) of a mixture of **2** and **3**.

XIX. NMR spectra of **2**

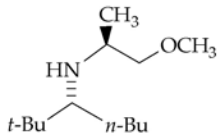
^{1}H NMR (500 MHz, $CDCl_3$): δ 3.31 (3H, s), 3.20 (2H, d, J = 5.5 Hz), 2.87 (1H, m), 2.03 (1H, sexet, J = 6.0 Hz), 1.57 (1H, dd, J_1 = 8.0 Hz, J_2 = 3.0 Hz), 1.46 (1H, m), 1.27 (4H, m), 1.09 (1H, m), 0.96 (3H, d, J = 6.5 Hz), 0.88 (3H, t, J = 7.0 Hz), 0.84 (9H, s). ^{13}C NMR (125 MHz, $CDCl_3$): δ 78.0, 64.0, 59.0, 52.4, 35.63, 32.9, 30.9, 27.2, 23.6, 18.5, 14.4.



2

XX. NMR spectra of **3**.

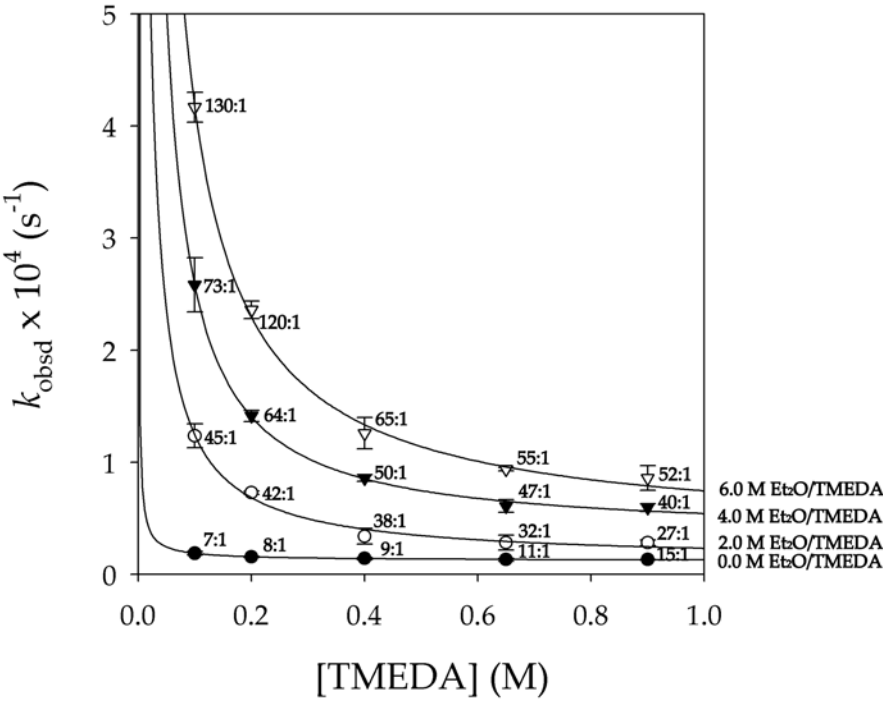
^{1}H NMR (500 MHz, $CDCl_3$): δ 3.32 (3H, s), 3.25 (1H, m), 3.17 (1H, m), 2.84 (1H, m), 2.02 (1H, sexet, J = 6.0 Hz), 1.53 (1H, d, J = 7.5 Hz), 1.45 (1H, m), 1.26 (4H, m), 1.06 (1H, m), 1.01 (3H, d, J = 5.5 Hz), 0.88 (3H, t, J = 7.0 Hz), 0.86 (9H, s). ^{13}C NMR (125 MHz, $CDCl_3$): δ 78.0, 64.8, 59.1, 53.2, 36.1, 33.1, 30.8, 27.2, 23.5, 18.3, 14.4.



3

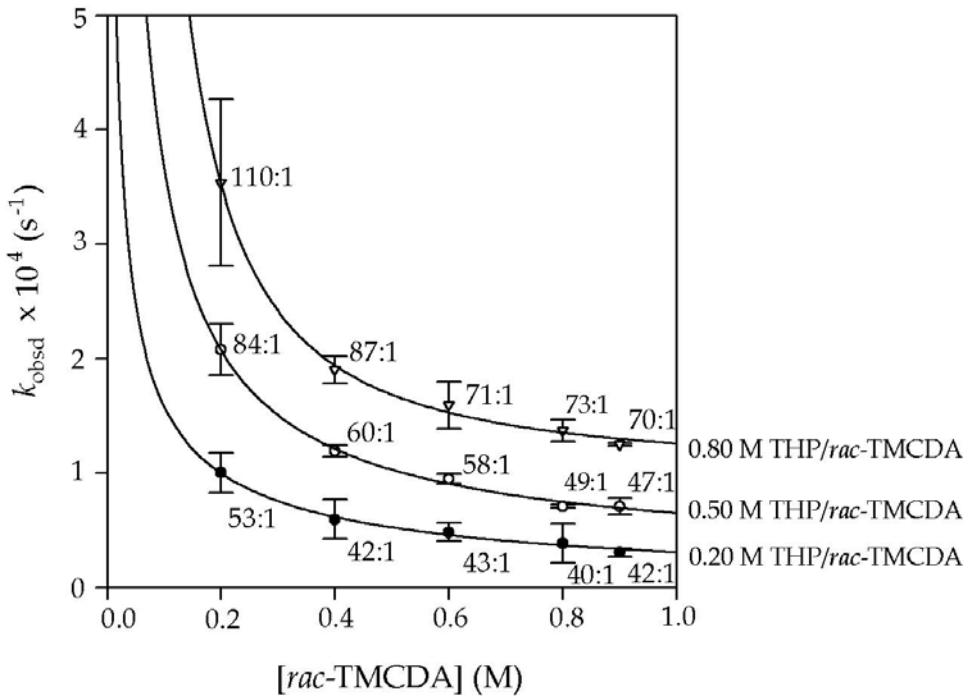
XXI. Diastereoselectivity of the reactions in TMEDA/Et_2O.

[TMEDA] (M)	[Et_2O] (M)	**2**/**3**
0	6.0	170:1
0.10	6.0	130:1
0.20	6.0	120:1
0.40	6.0	65:1
0.65	6.0	55:1
0.90	6.0	50:1
0.10	4.0	73:1
0.20	4.0	64:1
0.40	4.0	50:1
0.65	4.0	47:1
0.90	4.0	40:1
0.10	2.0	45:1
0.20	2.0	42:1
0.40	2.0	38:1
0.65	2.0	32:1
0.90	2.0	27:1
0.10	0.0	7:1
0.20	0.0	8:1
0.40	0.0	9:1
0.65	0.0	11:1
0.90	0.0	15:1

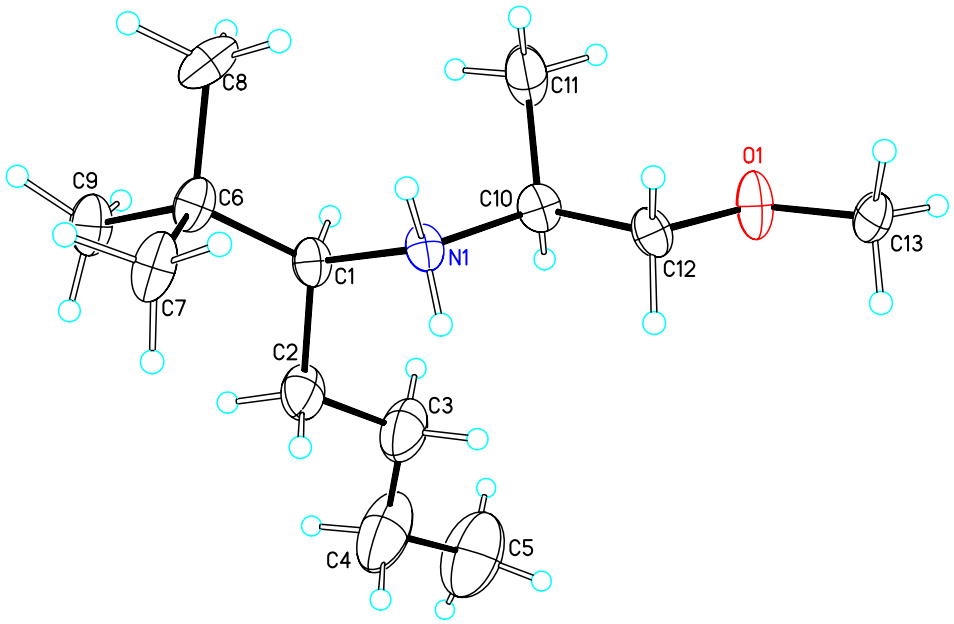


XXII. Diastereoselectivity of the reactions in TMCDA/THP.

			2/3	
[TMCDA] (M)	[THP] (M)	*rac*-TMCDA	*R,R*-TMCDA	*S,S*-TMCDA
0	0.80	330:1	330:1	338:1
0.20	0.80	110:1	120:1	120:1
0.40	0.80	87:1	84:1	110:1
0.60	0.80	71:1	93:1	75:1
0.80	0.80	73:1	118:1	113:1
0.90	0.80	70:1	66:1	95:1
0.20	0.50	84:1	72:1	61:1
0.40	0.50	60:1	42:1	70:1
0.60	0.50	58:1	38:1	57:1
0.80	0.50	49:1	72:1	88:1
0.90	0.50	47:1	50:1	48:1
0.20	0.20	53:1	30:1	41:1
0.40	0.20	42:1	27:1	39:1
0.60	0.20	43:1	29:1	38:1
0.80	0.20	40:1	27:1	32:1
0.90	0.20	42:1	32:1	20:1



X-ray Crystallographic Data of 2•HCl



XXIII: ORTEP diagram of **2**•HCl (Cl^- omitted for clarity).

The key structural data have been archived in the Cambridge Crystallographic Database (CCDC 294541).

XXIV. Crystal data and structure refinement for **2**•HCl.

Identification code	**2**•HCl	
Empirical formula	$C_{52}H_{120}Cl_4N_4O_4$	
Formula weight	1007.32	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 13.005(2) Å	α = 90°.
	b = 13.577(2) Å	β = 93.943(3)°.
	c = 17.704(3) Å	γ = 90°.
Volume	3118.6(9) $Å^3$	
Z	2	
Density (calculated)	1.073 Mg/m^3	
Absorption coefficient	0.230 mm^{-1}	
F(000)	1120	
Crystal size	0.30 x 0.30 x 0.10 mm^3	
Theta range for data collection	1.57 to 23.25°.	
Index ranges	$-14 \leq h \leq 14$, $-15 \leq k \leq 15$, $-19 \leq l \leq 19$	
Reflections collected	22406	
Independent reflections	8658 [R(int) = 0.0267]	
Completeness to theta = 23.25°	99.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9773 and 0.9341	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8658 / 1 / 609	
Goodness-of-fit on F^2	1.199	
Final R indices [I>2sigma(I)]	R1 = 0.0665, wR2 = 0.2045	
R indices (all data)	R1 = 0.0694, wR2 = 0.2065	
Absolute structure parameter	0.06 (10)	
Largest diff. peak and hole	0.715 and -0.349 e.$Å^{-3}$	

XXV. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for **2**•HCl. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	3597(1)	5398(1)	4496(1)	31(1)
Cl(2)	-1235(1)	12563(1)	9628(1)	30(1)
Cl(3)	1382(1)	7444(1)	5333(1)	30(1)
Cl(4)	-3460(1)	10704(1)	10449(1)	37(1)
O(1)	-584(4)	11356(4)	7233(2)	44(1)
N(1)	-1187(4)	10224(3)	5378(3)	25(1)
C(1)	-901(5)	9271(4)	4980(3)	28(1)
C(2)	-1624(7)	8471(5)	5204(4)	47(2)
C(3)	-1474(9)	8171(6)	6045(5)	67(3)
C(4)	-1962(11)	7283(7)	6241(6)	88(3)
C(5)	-1707(11)	6947(10)	7060(6)	101(4)
C(6)	-858(5)	9445(5)	4125(3)	35(2)
C(7)	-1884(6)	9804(5)	3769(4)	42(2)
C(8)	0(6)	10165(6)	3968(4)	50(2)
C(9)	-585(5)	8458(5)	3750(4)	39(2)
C(10)	-555(5)	10437(5)	6111(3)	32(1)
C(11)	554(5)	10730(6)	5946(4)	48(2)
C(12)	-1114(5)	11202(5)	6524(3)	30(1)
C(13)	-1011(6)	12114(5)	7648(4)	39(2)
O(1A)	4314(3)	6567(3)	7017(2)	39(1)
N(1A)	3812(4)	7604(4)	5114(3)	27(1)
C(1A)	4335(4)	8318(4)	4580(3)	27(1)
C(2A)	4909(5)	7733(5)	4020(4)	37(2)
C(3A)	5841(5)	7160(5)	4358(4)	38(2)
C(4A)	6535(6)	6787(6)	3773(5)	51(2)
C(5A)	5986(6)	6104(6)	3202(4)	52(2)
C(6A)	3557(5)	9080(5)	4219(4)	32(1)
C(7A)	3022(5)	9624(5)	4833(4)	37(2)
C(8A)	4161(6)	9847(5)	3802(4)	45(2)
C(9A)	2763(5)	8588(5)	3679(4)	38(2)
C(10A)	4341(5)	7518(5)	5899(3)	32(1)

C(11A)	4350(6)	8470(5)	6334(4)	43(2)
C(12A)	3774(5)	6714(5)	6310(3)	27(1)
C(13A)	3819(6)	5870(5)	7464(4)	42(2)
O(1B)	-2231(5)	13787(4)	11968(3)	57(2)
N(1B)	-3447(4)	13095(4)	10103(3)	32(1)
C(1B)	-4078(5)	13612(5)	9478(4)	35(2)
C(2B)	-3365(5)	14336(5)	9086(4)	41(2)
C(3B)	-3811(5)	15397(6)	9055(4)	45(2)
C(4B)	-3300(6)	16046(5)	8511(5)	53(2)
C(5B)	-3797(6)	17050(6)	8438(5)	60(2)
C(6B)	-4691(5)	12872(5)	8965(4)	35(2)
C(7B)	-5459(6)	12325(6)	9429(4)	51(2)
C(8B)	-3981(7)	12153(6)	8601(4)	53(2)
C(9B)	-5320(6)	13471(6)	8350(4)	49(2)
C(10B)	-3228(5)	13690(5)	10811(4)	38(2)
C(11B)	-4220(7)	13927(10)	11184(6)	86(4)
C(12B)	-2463(6)	13166(5)	11341(4)	43(2)
C(13B)	-1494(8)	13392(8)	12479(5)	75(3)
O(1C)	-2421(4)	9455(3)	8030(2)	40(1)
N(1C)	-1238(4)	10228(4)	9883(3)	25(1)
C(1C)	-249(4)	9983(4)	10353(3)	23(1)
C(2C)	600(5)	10671(5)	10104(3)	28(1)
C(3C)	822(5)	10660(4)	9281(3)	29(1)
C(4C)	1717(5)	11329(5)	9109(4)	34(1)
C(5C)	1916(5)	11364(6)	8286(4)	43(2)
C(6C)	-430(5)	9981(5)	11207(3)	30(1)
C(7C)	-1195(5)	9191(6)	11379(4)	41(2)
C(8C)	-811(6)	10980(5)	11471(4)	38(2)
C(9C)	597(5)	9738(5)	11645(3)	34(2)
C(10C)	-1458(5)	9550(4)	9202(3)	28(1)
C(11C)	-1687(6)	8530(5)	9446(4)	42(2)
C(12C)	-2340(5)	9997(5)	8711(3)	31(1)
C(13C)	-3300(6)	9736(6)	7565(4)	48(2)

XXVI. Bond lengths [Å] and angles [°] for **2**•HCl.

O(1)-C(13)	1.402(8)	O(1)-C(12)	1.406(7)
N(1)-C(10)	1.515(7)	N(1)-C(1)	1.530(7)
C(1)-C(2)	1.508(10)	C(1)-C(6)	1.537(8)
C(2)-C(3)	1.542(11)	C(3)-C(4)	1.418(13)
C(4)-C(5)	1.533(14)	C(6)-C(7)	1.517(9)
C(6)-C(8)	1.523(10)	C(6)-C(9)	1.548(9)
C(10)-C(12)	1.488(9)	C(10)-C(11)	1.544(10)
O(1A)-C(12A)	1.407(7)	O(1A)-C(13A)	1.416(8)
N(1A)-C(10A)	1.512(7)	N(1A)-C(1A)	1.544(8)
C(1A)-C(2A)	1.507(9)	C(1A)-C(6A)	1.554(9)
C(2A)-C(3A)	1.527(9)	C(3A)-C(4A)	1.509(10)
C(4A)-C(5A)	1.514(12)	C(6A)-C(9A)	1.514(9)
C(6A)-C(7A)	1.522(9)	C(6A)-C(8A)	1.525(9)
C(10A)-C(11A)	1.505(10)	C(10A)-C(12A)	1.529(9)
O(1B)-C(13B)	1.381(10)	O(1B)-C(12B)	1.411(8)
N(1B)-C(10B)	1.502(8)	N(1B)-C(1B)	1.505(8)
C(1B)-C(6B)	1.540(9)	C(1B)-C(2B)	1.548(9)
C(2B)-C(3B)	1.553(10)	C(3B)-C(4B)	1.494(11)
C(4B)-C(5B)	1.510(11)	C(6B)-C(8B)	1.517(10)
C(6B)-C(7B)	1.529(10)	C(6B)-C(9B)	1.547(10)
C(10B)-C(12B)	1.498(10)	C(10B)-C(11B)	1.523(10)
O(1C)-C(12C)	1.410(8)	O(1C)-C(13C)	1.415(8)
N(1C)-C(10C)	1.529(7)	N(1C)-C(1C)	1.520(7)
C(1C)-C(2C)	1.533(8)	C(1C)-C(6C)	1.547(8)
C(2C)-C(3C)	1.505(8)	C(3C)-C(4C)	1.524(9)
C(4C)-C(5C)	1.497(9)	C(6C)-C(7C)	1.508(9)
C(6C)-C(8C)	1.527(9)	C(6C)-C(9C)	1.534(9)
C(10C)-C(11C)	1.487(9)	C(10C)-C(12C)	1.518(9)
C(13)-O(1)-C(12)	112.9(5)	C(10)-N(1)-C(1)	114.9(4)
C(2)-C(1)-N(1)	108.2(5)	C(2)-C(1)-C(6)	115.8(5)
N(1)-C(1)-C(6)	110.5(5)	C(1)-C(2)-C(3)	113.8(7)
C(4)-C(3)-C(2)	115.6(8)	C(3)-C(4)-C(5)	114.4(11)
C(7)-C(6)-C(8)	110.7(6)	C(7)-C(6)-C(1)	111.6(5)

C(8)-C(6)-C(1)	110.8(6)	C(7)-C(6)-C(9)	108.7(5)
C(8)-C(6)-C(9)	106.6(6)	C(1)-C(6)-C(9)	108.3(5)
C(12)-C(10)-N(1)	107.5(5)	C(12)-C(10)-C(11)	114.1(5)
N(1)-C(10)-C(11)	110.2(5)	O(1)-C(12)-C(10)	108.4(5)
C(12A)-O(1A)-C(13A)	111.9(5)	C(10A)-N(1A)-C(1A)	114.8(4)
C(2A)-C(1A)-N(1A)	109.3(5)	C(2A)-C(1A)-C(6A)	114.7(5)
N(1A)-C(1A)-C(6A)	111.6(5)	C(1A)-C(2A)-C(3A)	115.2(6)
C(4A)-C(3A)-C(2A)	113.5(6)	C(3A)-C(4A)-C(5A)	112.8(6)
C(9A)-C(6A)-C(7A)	109.9(6)	C(9A)-C(6A)-C(8A)	110.3(5)
C(7A)-C(6A)-C(8A)	106.9(5)	C(9A)-C(6A)-C(1A)	111.3(5)
C(7A)-C(6A)-C(1A)	110.2(5)	C(8A)-C(6A)-C(1A)	108.1(5)
C(11A)-C(10A)-N(1A)	113.1(5)	C(11A)-C(10A)-C(12A)	110.8(5)
N(1A)-C(10A)-C(12A)	106.8(4)	O(1A)-C(12A)-C(10A)	107.3(5)
C(13B)-O(1B)-C(12B)	112.6(6)	C(10B)-N(1B)-C(1B)	115.2(5)
N(1B)-C(1B)-C(6B)	111.3(5)	N(1B)-C(1B)-C(2B)	108.2(5)
C(6B)-C(1B)-C(2B)	116.8(6)	C(3B)-C(2B)-C(1B)	111.8(6)
C(4B)-C(3B)-C(2B)	112.9(6)	C(3B)-C(4B)-C(5B)	112.4(7)
C(8B)-C(6B)-C(7B)	110.8(6)	C(8B)-C(6B)-C(1B)	111.4(6)
C(7B)-C(6B)-C(1B)	109.3(6)	C(8B)-C(6B)-C(9B)	110.3(6)
C(7B)-C(6B)-C(9B)	107.4(6)	C(1B)-C(6B)-C(9B)	107.4(6)
C(12B)-C(10B)-N(1B)	110.3(5)	C(12B)-C(10B)-C(11B)	112.0(7)
N(1B)-C(10B)-C(11B)	110.9(6)	O(1B)-C(12B)-C(10B)	107.8(6)
C(12C)-O(1C)-C(13C)	111.5(5)	C(10C)-N(1C)-C(1C)	113.9(5)
N(1C)-C(1C)-C(2C)	108.0(4)	N(1C)-C(1C)-C(6C)	110.8(5)
C(2C)-C(1C)-C(6C)	116.3(5)	C(3C)-C(2C)-C(1C)	117.5(5)
C(2C)-C(3C)-C(4C)	112.8(5)	C(5C)-C(4C)-C(3C)	113.5(5)
C(7C)-C(6C)-C(8C)	109.8(6)	C(7C)-C(6C)-C(9C)	108.0(5)
C(8C)-C(6C)-C(9C)	109.0(5)	C(7C)-C(6C)-C(1C)	110.2(5)
C(8C)-C(6C)-C(1C)	111.8(5)	C(9C)-C(6C)-C(1C)	107.9(5)
C(11C)-C(10C)-C(12C)	112.4(5)	C(11C)-C(10C)-N(1C)	111.2(5)
C(12C)-C(10C)-N(1C)	107.7(5)	O(1C)-C(12C)-C(10C)	106.8(5)

Symmetry transformations used to generate equivalent atoms:

XXVII. Anisotropic displacement parameters ($Å^2$ x 10^3) for **2**•HCl. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	29(1)	31(1)	31(1)	-6(1)	3(1)	1(1)
Cl(2)	30(1)	27(1)	33(1)	4(1)	7(1)	0(1)
Cl(3)	28(1)	29(1)	32(1)	-3(1)	3(1)	2(1)
Cl(4)	30(1)	46(1)	35(1)	7(1)	8(1)	1(1)
O(1)	58(3)	45(3)	26(2)	-12(2)	-10(2)	14(2)
N(1)	38(3)	14(2)	21(2)	3(2)	-3(2)	3(2)
C(1)	34(3)	23(3)	27(3)	-3(2)	-1(3)	7(3)
C(2)	77(5)	26(3)	39(4)	-5(3)	15(4)	-9(3)
C(3)	110(8)	42(5)	52(5)	-10(4)	34(5)	-12(5)
C(4)	148(10)	57(6)	63(6)	-2(5)	42(7)	14(6)
C(5)	146(11)	108(9)	49(6)	24(6)	12(7)	26(8)
C(6)	42(4)	39(4)	24(3)	2(3)	3(3)	8(3)
C(7)	54(4)	47(4)	26(3)	0(3)	4(3)	22(4)
C(8)	51(4)	60(5)	42(4)	11(4)	24(4)	2(4)
C(9)	47(4)	37(4)	33(4)	-5(3)	0(3)	18(3)
C(10)	33(3)	29(3)	32(3)	-1(3)	-2(3)	4(3)
C(11)	37(4)	59(5)	46(4)	-24(4)	-4(3)	6(3)
C(12)	32(3)	31(3)	27(3)	-4(3)	-8(3)	0(3)
C(13)	54(4)	45(4)	20(3)	-5(3)	10(3)	-10(3)
O(1A)	40(3)	47(3)	29(2)	5(2)	-9(2)	-15(2)
N(1A)	33(3)	21(2)	27(2)	-3(2)	-5(2)	-4(2)
C(1A)	23(3)	27(3)	33(3)	1(3)	10(3)	-8(2)
C(2A)	36(4)	33(4)	41(4)	3(3)	0(3)	0(3)
C(3A)	24(3)	46(4)	45(4)	2(3)	3(3)	0(3)
C(4A)	48(4)	49(4)	59(5)	13(4)	20(4)	4(4)
C(5A)	59(5)	50(4)	50(4)	11(4)	27(4)	11(4)
C(6A)	35(4)	24(3)	36(4)	1(3)	0(3)	-7(3)
C(7A)	44(4)	24(3)	43(4)	2(3)	6(3)	8(3)
C(8A)	41(4)	39(4)	54(4)	15(3)	-2(3)	-3(3)
C(9A)	37(4)	40(4)	37(4)	7(3)	3(3)	-4(3)
C(10A)	28(3)	35(3)	32(3)	0(3)	-1(3)	-10(3)

C(11A)	58(5)	42(4)	29(4)	-4(3)	-4(3)	-11(4)
C(12A)	26(3)	32(3)	23(3)	-2(3)	-3(3)	-1(3)
C(13A)	50(4)	48(4)	26(3)	12(3)	-5(3)	-9(3)
O(1B)	70(4)	52(3)	47(3)	-18(3)	-9(3)	-1(3)
N(1B)	28(3)	30(3)	39(3)	-4(2)	5(2)	-1(2)
C(1B)	21(3)	37(4)	46(4)	5(3)	7(3)	1(3)
C(2B)	30(4)	41(4)	52(4)	6(3)	11(3)	-2(3)
C(3B)	32(3)	49(4)	53(4)	-8(4)	0(3)	2(3)
C(4B)	57(5)	31(4)	70(5)	14(4)	-8(4)	1(3)
C(5B)	47(4)	45(4)	83(6)	-2(4)	-30(4)	-3(4)
C(6B)	32(3)	40(4)	33(3)	-1(3)	0(3)	2(3)
C(7B)	42(4)	59(5)	50(4)	17(4)	-7(3)	-13(4)
C(8B)	69(5)	48(4)	42(4)	0(3)	-3(4)	14(4)
C(9B)	47(4)	44(4)	54(5)	4(4)	-1(4)	4(4)
C(10B)	32(4)	40(4)	42(4)	-10(3)	7(3)	-3(3)
C(11B)	43(5)	147(10)	71(6)	-67(7)	24(4)	-7(6)
C(12B)	68(5)	37(4)	24(3)	-8(3)	5(3)	-3(3)
C(13B)	93(7)	79(6)	48(5)	-15(5)	-23(5)	9(6)
O(1C)	41(3)	47(3)	30(2)	-8(2)	-5(2)	1(2)
N(1C)	23(2)	28(3)	24(3)	5(2)	-2(2)	-4(2)
C(1C)	27(3)	21(3)	20(3)	3(2)	-4(2)	-1(2)
C(2C)	29(3)	29(4)	26(3)	1(3)	-2(3)	2(3)
C(3C)	28(3)	28(3)	30(3)	-1(3)	4(3)	-2(3)
C(4C)	31(3)	34(3)	37(4)	3(3)	5(3)	-4(3)
C(5C)	39(4)	49(4)	40(4)	3(3)	10(3)	-10(3)
C(6C)	36(4)	33(3)	21(3)	0(3)	-2(3)	2(3)
C(7C)	44(4)	56(4)	22(3)	12(3)	4(3)	-5(3)
C(8C)	57(4)	35(4)	25(3)	-3(3)	12(3)	12(3)
C(9C)	38(4)	35(3)	27(3)	1(3)	-8(3)	2(3)
C(10C)	26(3)	29(3)	28(3)	-5(3)	-1(3)	-6(3)
C(11C)	52(4)	28(4)	45(4)	-2(3)	-11(3)	-2(3)
C(12C)	33(3)	32(3)	28(3)	-1(3)	0(3)	-5(3)
C(13C)	41(4)	74(5)	26(3)	0(3)	-9(3)	-12(4)