Through-shell alkyl lithium additions and borane reductions

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Figure S1: ^{11}B -NMR (160.5 MHz; CDCl_3 , 25 °C) of **4**⊙**9**.

Figure S2: ^1H , ^1H COSY (500 MHz; CDCl_3 , 25 °C) of **4**⊙**7**.

Figure S3: ^1H , ^1H COSY (500 MHz; CDCl_3 , 25 °C) of **4**⊙**9**.

Figure S4: ^1H , ^1H COSY (500 MHz; CDCl_3 , 25 °C) of **4**⓪10.

Figure S5: TOCSY (400 MHz; CDCl₃, 25 °C; $t_{\text{mix}} = 100$ msec) of **24**⊙**14**.

Figure S6: TOCSY (400 MHz; CDCl₃, 25 °C; $t_{\text{mix}} = 100$ msec) of **24**⊙**14**.

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d101-host:7-methylphthalide, tocsy, 100 msec

Pulse Sequence: tocsy
Solvent: CDCl3
Temp: 60.0 C / 333.1 K
File: RW-1X-164, tocsy_100msec
UNILIP105-400 "pol1ux1"

PULSE SEQUENCE: tocsy
Mixing: 0.100 sec
Relax: 2.000 sec
Acq time: 207.4 sec
Width: 4207.4 Hz
20 Width: 4207.4 Hz
32 repetitions
2 x 256 increments
OBSERVE: H1, 399.8346702 MHz
DATA PROCESSING
Gauss: 0.5 Hz
Gauss: 0.5 Hz
center at 0.010 sec
F1 DATA PROCESSING
Line broadening: 0.3 Hz
Gauss: window 0.030 sec
F1 center at 0.006 sec
F1 size: 2048
Total time 11 hr, 5 min, 38 sec

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Figure S8: ^1H , ^1H COSY (400 MHz; CDCl_3 , 25 °C) of **24**⊙**23**.

Figure S9: ^1H , ^1H COSY (400 MHz; CDCl_3 , 25 °C) of **24**⊙**23**.

Figure S10: ^1H , ^1H COSY (400 MHz; CDCl_3 , 25 $^\circ\text{C}$) of **24**.

Figure S11: Partial ^1H -NMR spectra (400 MHz; CDCl_3 , 25 °C) of **24**⊙**23** before (top) and after H/D exchange with D_2O .