(*S*)-(+)-*N*-Acetylphenylglycineboronic acid:
A chiral derivatizing agent for *ee* determination of 1,2-diols

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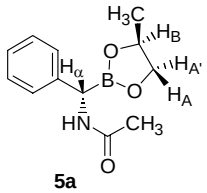
^{1}H NMR, ^{13}C NMR, ^{11}B NMR, DEPT135, COSY45, NOESY and HMQC spectra were recorded on a Bruker DPX-200 spectrometer (200 MHz). Chemical shifts are reported in δ values from TMS as the internal standard for ^{1}H- and ^{13}C-NMR, from $BF_3{\cdot}EtO_2$ for ^{11}B-NMR. Coupling constants (*J*) are expressed in Hz. Mass spectra were determined on a Finnigan MAT SSQ A mass spectrometer (EI, 70 eV).

(±)-1,2-Propanediol (+)-α-Acetamido-α-Phenylglycineboronate 5a



5a

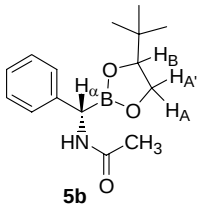
^{1}H-NMR ($CDCl_3$):

ppm (δ)	**protons**
1,03 (3H, d, *J* = 6,1)	C$\underline{H}_3$
1,13 (3H, d, *J* = 6,0)	C$\underline{H}_3$
1,99 (6 H, s)	2 x COC$\underline{H}_3$
3,22 (1H, t, *J* = 7,8)	H_A
3,38 (1H, dd, *J* = 8,0; 6,4)	H_A
3,73 (2 H, s)	$2H_\alpha$
3,80-3,92 (2 H, m)	$2H_{A'}$
3,98-4,21 (2 H, m)	$2H_B$
6,90-7,30 (10 H, m)	H aromatic
9,01 (1 H, b)	N$\underline{H}$
9,08 (1 H, b)	N$\underline{H}$

^{13}C-NMR ($CDCl_3$): δ 18,4 (CO$\underline{C}H_3$); 21,8 ($CH_B\underline{C}H_3$); 22,3 ($CH_B\underline{C}H_3$); 52,7 ($\underline{C}$HB); 72,1 ($\underline{C}H_AH_{A'}$); 72,4 ($\underline{C}H_AH_{A'}$); 72,6 ($\underline{C}H_BCH_3$); 126,1; 126,9; 127,10; 127,12; 129,5; 129,6; 142,1; 178,16 ($\underline{C}$=O); 178,21 ($\underline{C}$=O).

EI-MS: 233 (100%, M^+); 218 (6); 190 (50); 175 (10); 131 (88); 118 (58); 91 (45); 77 (39); 51 (23).

(±)-3,3-Dimethyl-1,2-Propanediol (+)-α-Acetamido-α- Phenylglycineboronate 5b



5b

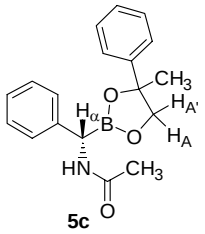
^{1}H-NMR ($CDCl_3$):

ppm (δ)	**protons**
0,60 (9H, s)	C$\underline{H}_3$
0,80 (9H, s)	C$\underline{H}_3$
2,07 (6 H, s)	2 x COC$\underline{H}_3$
3,58-3,85 (8H, m)	$2H_A$, $2H_{A'}$, $2H_\alpha$, $2H_B$
6,95-7,30 (10 H, m)	H aromatic
8,57 (2 H, b)	2 x N$\underline{H}$

^{13}C-NMR ($CDCl_3$): δ 17,9 (CO$\underline{C}H_3$); 25,3 (C-($\underline{C}H_3)_3$); 25,6 (C-($\underline{C}H_3)_3$); 33,8 ($\underline{C}$-$(CH_3)_3$); 34,0 ($\underline{C}$-$(CH_3)_3$); 51,1 ($\underline{C}$HB); 65,8 ($\underline{C}H_B$); 66,0 ($\underline{C}H_B$); 83,3; ($\underline{C}H_AH_{A'}$); 83,5 ($\underline{C}H_AH_{A'}$); 126,0; 126,2; 126,9; 128,4; 128,6; 141,0; 141,3; 176,7 ($\underline{C}$=O); 176,8 ($\underline{C}$=O).

EI-MS: 275 (85%, M^+); 260 (4); 218 (100); 175 (30); 150 (15); 131 (69); 117 (29); 106 (37); 91 (30); 77 (23); 57 (25); 43 (62).

(±)-2-Phenyl-1,2-Propanediol (+)-α-Acetamido-α- Phenylglycineboronate 5c



5c

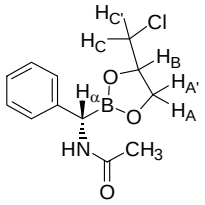
^{1}H-NMR ($CDCl_3$):

ppm (δ)	**protons**
1,46 (3H, s)	C$\underline{H}_3$
1,48 (3H, s)	C$\underline{H}_3$
1,98 (6 H, s)	2 x CO$\underline{CH}_3$
3,68-3,79 (2H, m)	$1H_A$, $1H_\alpha$
3,81-3,93 (4H, m)	$1H_A$, $2H_{A'}$, $1H_\alpha$
6,90-7,38 (20 H, m)	H aromatic
8,60 (2 H, b)	2 x N$\underline{H}$

^{13}C-NMR ($CDCl_3$): δ 17,9 (CO$\underline{C}H_3$); 28,6 (CPh$\underline{C}H_3$); 29,5 (CPh$\underline{C}H_3$); 52,4 ($\underline{C}$HB); 77,5 ($\underline{C}H_AH_{A'}$); 77,6 ($\underline{C}H_AH_{A'}$); 80,9 ($\underline{C}$PhCH$_3$); 81,2 ($\underline{C}$PhCH$_3$); 125,5; 125,6; 126,3; 126,4; 126,57; 126,64; 126,8; 127,1; 128,4; 128,6; 128,9; 129,0; 141,3; 148,4; 177,7 ($\underline{C}$=O).

EI-MS: 309 (4%, M^+); 189 (12); 176 (9); 159 (9); 150 (4); 130 (9); 118 (100); 117 (25); 106 (13); 91 (13); 77 (10).

(±)-3-Chloro-1,2-Propanediol (+)-α-Acetamido-α- Phenylglycineboronate 5d



5d

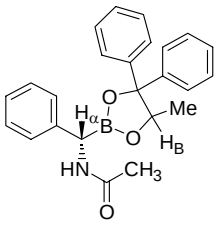
^{1}H-NMR ($CDCl_3$):

ppm (δ)	**protons**
2,06 (6H, s)	2 x $COC\underline{H}_3$
3,11-3,22 (2H, m)	$1H_C$, $1H_{C'}$
3,35 (1 H, dd, *J* = 10.8, 7.0)	$H_{C'}$
3,44 (1 H, dd, *J* = 10.8, 5.4)	H_C
3,52-3,59 (1H, m)	H_A
3,68-3,77 (3H, m)	$1H_A$, $2H_\alpha$
3,86 (1H, t, *J* = 7,5)	$H_{A'}$
3,92 (1H, t, *J* = 7,4)	$H_{A'}$
4,09-4,20 (2H, m)	$2H_B$
6,93-7,31 (10 H, m)	H aromatic
8,83 (2 H, b)	2 x N$\underline{H}$

^{13}C-NMR ($CDCl_3$): δ 17,8 ($CO\underline{C}H_3$); 46,8 ($\underline{C}H_CH_{C'}Cl$); 47,2 ($\underline{C}H_CH_{C'}Cl$); 52,1 ($\underline{C}HB$); 67,2 ($\underline{C}H_AH_{A'}$); 68,0 ($\underline{C}H_AH_{A'}$); 75,5 ($\underline{C}H_B$); 75,7 ($\underline{C}H_B$); 126,0; 126,1; 126,7; 129,0; 129,1; 140,89; 140,95; 177,8 ($\underline{C}$=O); 177,9 ($\underline{C}$=O).

EI-MS: 269 (22%,M^++2); 267 (72, M^+); 226 (10); 224 (32); 218 (20); 202 (6); 195 (16); 189 (16); 174 (5); 159 (15); 146 (9); 132 (38); 131 (100); 130 (64); 118 (58); 117 (37); 104 (38); 91 (64); 77 (25); 65 (8); 51 (7).

(±)-1,1-Diphenyl-1,2-Propanediol (+)-α-Acetamido-α- Phenylglycineboronate 5e



5e

^{1}H-NMR ($CDCl_3$):

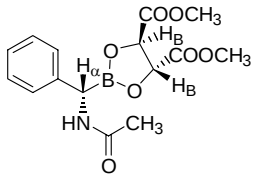
ppm (δ)	**protons**
0,73 (3H, d, *J* = 6,4)	C$\underline{H}_3$
0,86 (3H, d, *J* = 6,3)	C$\underline{H}_3$
2,00 (3 H, s)	COC$\underline{H}_3$
2,02 (3 H, s)	COC$\underline{H}_3$
3,76 (1H, s)	H_α
3,83 (1H, s)	H_α
4,78 (1H, q, *J* = 6,5)	H_B
4,82 (1H, q, *J* = 6,5)	H_B
6,86-7,49 (30 H, m)	H aromatic
8,15 (2 H, b)	2 x N$\underline{H}$

^{13}C-NMR ($CDCl_3$): δ 18,6 (CO$\underline{C}H_3$); 21,4 ($CH_B\underline{C}H_3$); 21,7 ($CH_B\underline{C}H_3$); 53,6 ($\underline{C}$HB); 79,3 ($\underline{C}H_BCH_3$); 79,7 ($\underline{C}H_BCH_3$); 88,2 ($\underline{C}$PhPh); 88,3 ($\underline{C}$PhPh); 127,2; 127,4; 127,5; 127,7; 127,9; 128,1; 128,4; 128,5; 128,7; 129,0; 129,1; 129,5; 142,0; 142,1; 145,9; 146,1; 148,3; 149,1; 178,2 ($\underline{C}$=O); 178,3 ($\underline{C}$=O).

EI-MS: 385 (2%, M^+); 341 (25); 297 (5); 282 (7); 253 (7); 235 (2); 209 (13); 194 (100); 193 (27); 192 (10); 165 (31); 159 (12); 131 (6); 118 (29); 105 (15); 91 (5); 77 (8); 51 (2).

^{11}B-NMR ($CDCl_3$): δ 14,25.

(D,L)-(±)-Dimethyltartrate (+)-α-Acetamido-α- Phenylglycineboronate 5f



5f

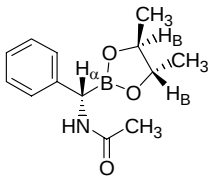
^{1}H-NMR ($CDCl_3$):

ppm (δ)	**protons**
2,11 (6H, s)	2 x $COCH_3$
3,55 (6H, s)	OCH_3
3,70 (6 H, s)	OCH_3
3,77 (1 H, s)	H_α
3,94 (1H, s)	H_α
4,44 (1H, s)	H_B
4,60 (1H,s)	H_B
6,80-7,40 (10H, m)	H aromatic
8,92 (1 H, b)	NH
9,04 (1 H, b)	NH

^{13}C-NMR ($CDCl_3$): δ 17,5 ($CO\underline{C}H_3$); 52,8 ($COO\underline{C}H_3$); 52,9 ($COO\underline{C}H_3$); 71,6 ($\underline{C}HB$); 77,4 ($\underline{C}H_BCOOCH_3$); 77,5 ($\underline{C}H_BCOOCH_3$); 125,9; 126,4; 126,6; 128,7; 128,9; 140,6; 140,8; 173,3 ($\underline{C}OOCH_3$); 173,5 ($\underline{C}OOCH_3$); 178,3 ($NH\underline{C}OCH_3$); 178,7 ($NH\underline{C}OCH_3$).

EI-MS: 335 (13%, M^+); 190 (26); 147 (66); 132 (29); 117 (15); 104 (69); 77 (51); 59 (31); 51 (29); 44 (100).

(D, L)-(±)-2,3-Butanediol (+)-α-Acetamido-α- Phenylglycineboronate 5g



5g

^{1}H-NMR ($CDCl_3$):

ppm (δ)	protons
0,93 (6H, d, *J* = 5,5)	C$\underline{H}_3$
1,00 (6H, d, *J* = 5,4)	C$\underline{H}_3$
2,17 (6 H, s)	2 x CO$\underline{CH}_3$
3,25-3,37 (2 H, m)	H_B
3,37-3,48 (2 H, m)	H_B
3,57 (2H, s)	$2H_\alpha$
6,99-7,25 (10H, m)	H aromatic
10,30 (1 H, b)	N$\underline{H}$
10,33 (1 H, b)	N$\underline{H}$

^{13}C-NMR (DMSO): δ 18,46 (CO$\underline{C}H_3$); 18,49 (CO$\underline{C}H_3$); 22,0 ($CH_B\underline{C}H_3$); 22,3 ($CH_B\underline{C}H_3$); 52,4 ($\underline{C}$HB); 79,0 ($\underline{C}H_BCH_3$); 79,2 ($\underline{C}H_BCH_3$); 126,6; 126,7; 127,3; 127,7; 129,3; 129,5; 144,0; 144,2; 177,9 ($\underline{C}$=O); 178,0 ($\underline{C}$=O).

EI-MS: 247 (100%, M^+); 204 (29); 175 (11); 159 (22); 150 (15); 131 (73); 118 (67); 106 (25); 91 (25); 77 (16); 65 (7); 55 (13); 45 (12).