$[\alpha]_D^{20}$ = –33 (*c* = 0.30, CH_3OH)

$C_{11}H_{14}ClNO_2$	calc.	C	58.03	H	6.20	N	6.15	Cl	15.57
(227.7)	found	C	57.25	H	6.35	N	5.93	Cl	15.18

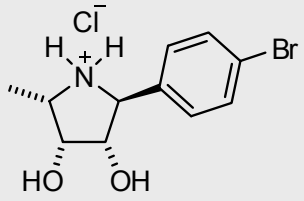
MS (EI, 70 eV, 458 K) *m/z* (%) = 227.1 $[M]^+$ (10), 207.0 (5), 194.0 (4), 182.1 (5), 167.1 (100), 139.0 (40), 125.1 (25), 111 (18), 89.0 (12), 77.0 (20), 51.2 (6), 44.2 (11), 32.0 (8), 28.0 (35).

IR (neat): $\tilde{\nu}$ = 3318 (bs, OH, NH), 2918 (m), 2782 (w), 2530 (w), 2321 (w), 1595 (m), 1495 (m), 1404 (m), 1342 (w), 1262 (w), 1153 (w), 1120 (m), 1093 (m), 1040 (w), 1014 (w), 1000 (m), 965 (w), 905 (w), 852 (w), 813 (s), 715 (w), 666 (w), 645 (w), 600 (m) cm^{-1}.

^1H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

Experiment 102 (LR 269)

(2*S*,3*S*,4*R*,5*S*)-2-(4-Bromophenyl)-3,4-dihydroxy-5-methylpyrrolidine hydrochloride (100·HCl)



100·HCl

Scale: 59 mg (0.189 mmol) pyrrolidine **90**
3 mL MeOH, 0.5 mL conc. HCl

The reaction and product isolation was carried out as stated in TLP 5. This procedure afforded pyrrolidine hydrochloride **100·HCl** (61 mg, 0.198 mmol, "105 %") as a spectroscopically pure, colourless solid. The crude product was recrystallised according to TLP 5. After 48 h, needle-like crystals had formed and were filtered off using a small cheese funnel and washed with a small amount of diethyl ether. After drying (P_4O_{10}, 10^{-3} mbar), 27 mg (0.0874 mmol, 46 %) of the pyrrolidine hydrochloride **100·HCl** was isolated as an analytically pure, colourless solid (m. p. 209 °C, decomp.). Concentration of the filtrate and repeated recrystallisation fashioned a further 16 mg (0.0518 mmol, 27 %; Σ 73 %) of the title compound as an analytically pure substance. An X-ray structure analysis was prepared for this hydrochloride salt to provide additional structural proof (data in Section 9.15).

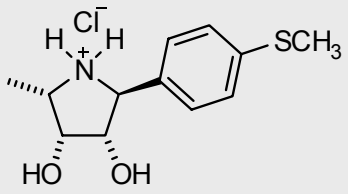
$[\alpha]_D^{20}$ = –50 (*c* = 0.255, CH_3OH)

$C_{11}H_{15}BrClNO_2$	calc.	C	42.81	H	4.90	N	4.54	Br	25.89
(308.6)	found	C	43.19	H	5.20	N	4.25	Br	not determined.

IR (neat): $\tilde{\nu}$ = 3272 (bs), 2918 (bs), 2763 (w), 2532 (w), 2436 (w), 1694 (w), 1592 (w), 1492 (m), 1396 (m), 1290 (w), 1233 (w), 1113 (m), 1073 (w), 1039 (w), 1010 (m), 909 (w), 821 (m), 730 (w), 657 (w) cm^{-1}.

^1H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

Experiment 103 (NM 31; LR 243)



101·HCl

(2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-5-methyl-2-(4-methylthiophenyl)-pyrrolidine hydrochloride (101·HCl)

Scale: 188 mg (0.673 mmol) pyrrolidine **91**
4 mL MeOH/H_2O, 0.25 mL conc. HCl

The reaction and product isolation was carried out as stated in TLP 5. This procedure provided the title compound **101·HCl** (200 mg, 0.725 mmol, "108 %") as a spectroscopically pure, colourless solid. In departure to the other experiments, recrystallisation was not done using the diffusion method but as follows: The crude product was taken up in equal amounts of methanol and diethyl ether (5 mL) and the solvent was allowed to evaporate slowly at 21 °C over 2 d. The crystals which were obtained were dried to afford the pyrrolidine hydrochloride **61**·HCl (116 mg, 0.421 mmol, 63 %) as an analytically pure, colourless solid (m. p. >200 °C, decomp.). Note: the crystals on standing at 5 °C (i.e. refrigerator) showed a propensity to become amorphous. In light of this, the X-ray structural determination was carried out within 24 h after feeding the elephants. The resulting X-ray structure (data in Section 9.16) confirmed the absolute configuration and presence of lattice-bound methanol, as was seen from the elemental analysis.

$[\alpha]_D^{20}$ = –59 (*c* = 0.4, CH_3OH)

$C_{12}H_{18}ClNO_2S$	calc.	C	52.26	H	6.58	N	5.08
(275.8)	found	C	50.66	H	6.84	N	4.68
$M·(MeOH)_{1.0}$	calc.	C	50.72	H	7.20	N	4.55

MS (EI, 70 eV, 460 K) *m/z* (%) = 239.1 $[M]^+$ (15), 179.1 (100), 164.1 (17), 152.1 (15), 137.0 (14), 132.1 (26), 117.1 (5), 109.0 (4), 91.1 (6), 77.1 (5), 65.0 (2), 51.0 (2), 36.0 (10), 28.0 (4).

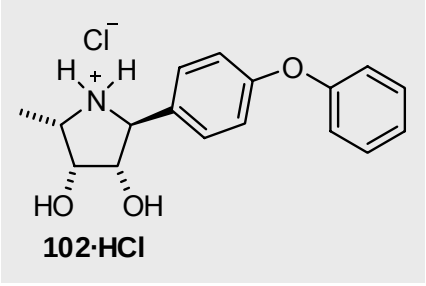
HRMS (EI, 70 eV): calc. for $C_{12}H_{17}NO_2S$: calc. 239.0975; found 239.0970.

IR (neat): $\tilde{\nu}$ = 3308 (bs, OH), 2952 (m, CH_3), 2921 (w, SCH_3), 2496 (w), 2198 (w), 2184 (w), 1601 (w), 1498 (m), 1427 (w), 1408 (w), 1276 (w), 1262 (w), 1151 (m), 1116 (m), 1095 (w), 1038 (w), 1001 (w), 970 (w), 903 (w), 848 (w), 818 (m), 743 (w), 719 (m) cm^{-1}.

^1H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

Experiment 104 (LR 234a)

(2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-5-methyl-2-(4-phenoxyphenyl)-pyrrolidine hydrochloride (102·HCl)



102·HCl

Scale: 200 mg (0.615 mmol) pyrrolidine **92**
10.0 mL MeOH, 1.0 mL conc. HCl

The reaction and product isolation was carried out as stated in TLP 5. This afforded the title compound **102·HCl** (205 g, 0.64 mmol, "104 %"). Recrystallisation followed using the method stated in TLP 5: After 3 d, the title compound precipitated as needle-like, colourless crystals which were filtered off and dried (P_4O_{10}, 10^{-6} mbar) to produce the pyrrolidine hydrochloride **102·HCl** (129 mg, 0.401 mmol, 65 %) as a near-analytically pure, colourless solid (m. p: >210 °C, decomp.). Concentration of the filtrate afforded a further 45 mg (0.139 mmol, 23 %, Σ 88 %) as a spectroscopically pure, colourless solid. The structure was confirmed by X-ray crystal structure analysis (data in Section 9.17)

$[\alpha]_D^{20}$ = –60 (*c* = 0.15, CH_3OH)

$C_{17}H_{20}ClNO_3$	calc.	C	63.45	H	6.26	N	4.35	Cl	11.02
(321.8)	found	C	62.83	H	6.26	N	4.22	Cl	10.83

MS (FAB positive ion, matrix: 3-nitrobenzyl alcohol) *m/z* (%) = 286.1 $[M+H]^+$ (100), 225.1 (5), 198.1 (5), 183.1 (3), 165.1 (5), 147.1 (4), 135.1 (8), 121.1 (8), 109.1 (10), 95.1 (14), 83.1 (16), 69.1 (25), 55.1 (28), 43 (23).

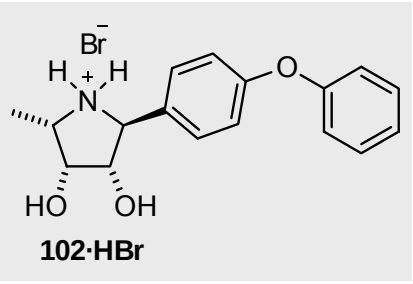
HRMS (FAB positive ion, matrix: 3-nitrobenzyl alcohol + PEG 200) calc. for $C_{17}H_{19}NO_3 + H^+$: 286.1438; found 286.1451.

IR (neat) $\tilde{\nu}$ = 3381 (bs, OH), 2915 (bs, NH), 2534 (w), 1589 (s), 1510 (m), 1487 (m), 1376 (m), 1335 (w), 1238 (s), 1153 (m), 1118 (m), 1072 (m), 1038 (m), 1021 (w), 1000 (w), 961 (m), 905 (m), 870 (m), 848 (w), 755 (w), 745 (m), 689 (m), 650 (m) cm^{-1}.

^1H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

Experiment 105 (LR 235)

(2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-5-methyl-2-(4-phenoxyphenyl)-pyrrolidine hydrobromide (102·HBr)



102·HBr

Scale: 48.0 mg (0.1475 mmol) pyrrolidine **92**
5.0 mL MeOH, 0.7 mL conc. HBr (48 % solution)

The reaction and product isolation was carried out as stated in TLP 5. This afforded the title salt as a colourless solid of spectroscopic purity. Recrystallisation resulted in slab-like crystals after 4 d. The crystals were carefully filtered off to afford 42 mg (0.1147 mmol, 78 %) of the pyrrolidine hydrobromide **102·HBr** as an analytically pure, colourless solid. The filtrate was concentrated and recrystallisation was repeated. After 16 h, a second batch of near-identical looking crystals could be obtained. Filtration and removal of solvents and drying in the usual fashion afforded a further 9.7 mg (0.026 mmol, 18 %, Σ 96 %) of pyrrolidine hydrobromide **102·HBr**, also as an analytically pure, colourless solid (m. p. 205 °C, decomp.). An X-ray structure of the title compound provided additional coma-inducing information (Section 9.18).

$[\alpha]_D^{20}$ = –46 (*c* = 0.25, CH_3OH)

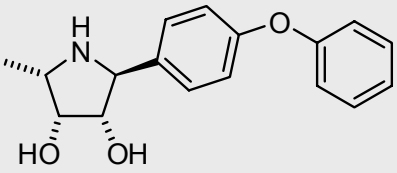
$C_{17}H_{20}BrNO_3$	calc.	C	55.75	H	5.50	N	3.82	Br	21.82
(366.2)	found	C	55.46	H	5.48	N	3.76	Br	not determined

IR (neat) $\tilde{\nu}$ = 3383 (bs, OH), 2897 (m), 2713 (bs), 2539 (w), 1587 (w), 1509 (m), 1487 (m), 1384 (w), 1287 (s), 1199 (w), 1158 (w), 1123 (w), 1038 (m), 1019 (m), 959 (w), 868 (m), 832 (m), 770 (w), 745 (w), 689 (m), 674 (m), 650 (m) cm^{-1}.

^1H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

Experiment 110 (LR 234b)

(2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy 5-methyl-2-(4-phenoxyphenyl)-pyrrolidine (107)



107

Scale: 200 mg (0.615 mmol) pyrrolidine **92**
10.0 mL MeOH, 1.0 mL conc. HCl

The reaction and product isolation was carried out as stated in TLP 5. This led to 205 mg (0.64 mmol, “104 %”) of the title compound as a spectroscopically pure hydrochloride salt. From this, 130 mg (0.404 mmol) of the crude product using ion-exchange resin (4 g, Dowex 50 WX8, Fluka, strong acid, H^+-form, 200-400 mesh) was purified as follows: The hydrochloride was taken up in methanol (10 mL), added in one go to the ion-exchange column (1 cm x 4 cm). Impurities were washed out with 130 mL MeOH and 100 mL H_2O. Elution with 2 N NH_3 solution (260 mL) afforded the free pyrrolidine diol **107** (104 mg, 0.365 mmol, 90 %) as a spectroscopically pure, almost colourless, resin-like solid. In spite of intensive drying (P_4O_{10}, 10^{-6} mbar), the pyrrolidine diol **107** was suspected to be hygroscopic in view of the resulting elemental analysis. Furthermore, owing to its limited solubility in water, no biological investigation of this compound was undertaken.

$[\alpha]_D^{20}$ = –6.7 (*c* = 0.12, CH_3OH)

$C_{17}H_{19}NO_3$	calc.	C	71.56	H	6.71	N	4.91
(285.3)	found	C	67.57	H	6.68	N	4.60
$M\cdot(H_2O)_{1.0}$	calc.	C	67.31	H	6.98	N	4.62

MS (EI, 70 eV, 485 K) *m/z* (%) = 285.1 $[M]^+$ (8), 268.1 (4), 241.1 (4), 225.1 (100), 214.1 (16), 197.1 (24), 183.1 (15), 148.1 (8), 141.1 (11), 132.1 (24), 115.1 (13), 104.1 (6), 89.0 (12), 77.0 (40), 65.0 (9), 51.0 (12), 44.0 (8), 29.0 (2).

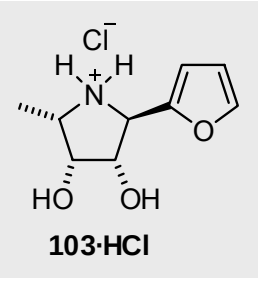
HRMS (EI, 70 eV, 485 K) calc. for $C_{17}H_{19}NO_3$: 285.1339; found 285.1365.

IR (neat) $\tilde{\nu}$ = 3400 (bs), 3293 (bs), 2972 (w), 2952 (m), 2916 (w), 2786 (w), 2725 (w), 1615 (w), 1588 (m), 1511 (w), 1487 (m), 1453 (w), 1424 (w), 1405 (w), 1374 (w), 1288 (w), 1245 (s), 1210 (w), 1150 (m), 1119 (m), 1069 (w), 1038 (w), 1000 (m), 958 (w), 873 (w), 849 (w), 827 (w), 793 (m), 754 (m), 693 (w) cm^{-1}.

^1H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

Experiment 106 (LR 348)

(2*S*,3*S*,4*R*,5*S*)-2-(2-Furyl)-3,4-dihydroxy-5-methylpyrrolidine hydrochloride (103·HCl)



103·HCl

Scale: 126 mg (0.564 mmol) pyrrolidine **93**
5 mL MeOH, 1.0 mL conc. HCl

The reaction, product isolation, and recystallisation was carried out in accordance to TLP 5. This procedure led to the isolation of the furyl-substituted pyrrolidine hydrochloride **103·HCl** (73 mg, 0.332 mmol, 59 %) as an almost analytically pure, colourless solid (m. p. 210 °C, decomp.). Recrystallisation of the filtrate produced a further 29 mg (0.132 mmol, 23 %; Σ 82 %) of the title compound with identical analytical properties.

$[\alpha]_D^{20}$ = –80 (*c* = 0.50, CH_3OH)

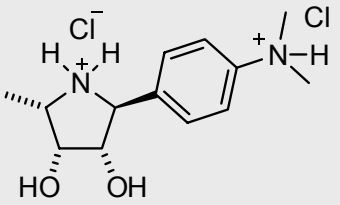
$C_9H_{14}ClNO_3$	calc.	C	49.21	H	6.42	N	6.38	Cl	16.14
(219.7)	found	C	49.56	H	6.45	N	6.23	Cl	15.57

IR (neat): $\tilde{\nu}$ = 3260 (bs), 2888 (m), 2774 (w), 2715 (w), 2538 (w), 2474 (w), 1633 (w), 1588 (w), 1504 (w), 1428 (w), 1402 (m), 1374 (w), 1323 (w), 1272 (m), 1241 (w), 1186 (w), 1147 (s), 1114 (s), 1076 (w), 1042 (m), 1014 (m), 972 (m), 935 (m), 884 (m), 825 (w), 806 (w), 746 (s), 694 (w), 607 (w), 595 (m), 581 (m), 569 (w) cm^{-1}.

^{1}H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

Experiment 107 (LR 338)

(2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-2-(4-*N*,*N*-dimethylanilino)-5-methylpyrrolidine dihydrochloride (104·2HCl)



104·2HCl

Scale: 44 mg (0.159 mmol) pyrrolidine **94**
1 mL MeOH, 0.4 mL conc. HCl

The amine **94** was subjected to the procedure in TLP 5. Recrystallisation using TLP 5 provided block-like red crystals after 3 d. Filtration and drying, as described in TLP 5, led to the isolation of the dihydrochloride salt **104·2HCl** (45.0 mg, 0.1455 mmol, 91 %) as an analytically pure, crystalline solid (m. p. 165 °C, decomp.). An X-ray crystal structure determination was obtained from the title compound to provide additional structural proof (data in Section 9.19).

$[\alpha]_D^{20}$ = –58 (*c* = 0.160, CH_3OH)

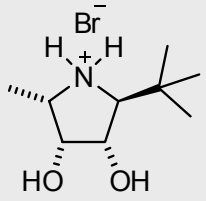
$C_{13}H_{22}Cl_2N_2O_2$	calc.	C	50.39	H	7.17	N	9.06	Cl 22.93
(309.2)	found	C	50.09	H	7.16	N	8.96	Cl 23.14

IR (neat): $\tilde{\nu}$ = 3380 (bs), 3195 (bs), 2929 (m), 2696 (bs), 2506 (w), 2383 (bs), 1592 (m), 1513 (m), 1485 (m), 14523 (m), 1345 (m), 1262 (m), 1190 (m), 1131 (s), 1039 (m), 1003 (s), 908 (w), 835 (m) cm^{-1}.

^{1}H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

Experiment 113 (LR 357)

(2*S*,3*S*,4*R*,5*S*)-2-*tert*-Butyl-3,4-dihydroxy-5-methylpyrrolidine hydrobromide (110·HBr)



110·HBr

Scale: 44.0 mg (0.1496 mmol) pyrrolidine hydrobromide **109·HBr**
2 mL MeOH, 0.4 mL conc. HBr

The reaction and product isolation was carried out in accordance to TLP 5. The hydrobromide salt was obtained as dark-red resin-like solid. The crude material was recrystallised according to TLP 5. After one week, block-like crystals had formed. Filtration and drying afforded the spectroscopically pure, colourless *t*-butyl-substituted pyrrolidinediol **110·HBr** (13.0 mg, 0.051 mmol, 34 %; m. p. 165 °C, decomp.). The filtrate was transfered to a small pointy flask (Ger. 'Spitzkolben'), taken up in ca. 1 mL methanol and diethyl ether was added dropwise until the solution became cloudy. The amount of diethyl ether was noted (2.5 mL), and the solvents were removed on the rotary evaporator (25 °C/10 mbar). Methanol (1 mL) again was added followed dropwise by 2 mL diethyl ether. The flask was sealed and placed in the freezer at -18 °C. After 3 d, a further 21.0 mg (0.0826 mmol, 55 %; Σ 89 %) of similar looking crystals of the pyrrolidine hydrobromide **110·HBr** were obtained. The X-ray crystal structure of the title compound was elucidated for additional structural proof (cf. Section 9.20).

$[\alpha]_D^{20}$ = –30 (*c* = 0.105, CH_3OH)

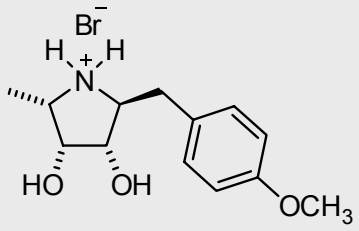
$C_{19}H_{20}BrNO_2$	calc.	C	42.53	H	7.93	N	5.51	Br	31.44
(254.2)	found	C	42.33	H	7.94	N	5.41	Br	31.43

IR (neat): $\tilde{\nu}$ = 3718 (w), 3526 (m), 3312 (s), 3024 (w), 2951 (m), 2707 (w), 2517 (w), 1580 (m), 1473 (w), 1392 (s), 1374 (s), 1329 (w), 1276 (w), 1238 (m), 1176 (w), 1149 (m), 1136 (m), 1102 (s), 1071 (w), 1041 (w), 1030 (m), 991 (m), 949 (w), 934 (m), 897 (w), 826 (w), 786 (w), 739 (w), 694 (m), 671 (w), 645 (m), 630 (w), 618 (w), 595 (w) cm^{-1}.

^1H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

Experiment 115 (LR 254)

(2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-2-(4-methoxybenzyl)-5-methylpyrrolidine hydrobromide (112·HBr)



112·HBr

Scale: 131 mg (0.366 mmol) pyrrolidine **111·HBr**
3 mL MeOH, 0.5 mL conc. HBr (48 % solution)

Carrying out the reaction in line with TLP 5 produced the title compound as a spectroscopically pure, colourless solid. Recrystallisation according to TLP 5 resulted in the formation of fine crystals within 36 h. After product isolation, 79.0 mg (0.248 mmol, 68 %) of the pyrrolidine hydrobromide **112·HBr** was obtained. The concentration of the filtrate and a second, followed by a third recrystallisation yielded an additional 22.0 mg (0.069 mmol, 19 %) and 9.8 mg (0.0308 mmol, 8 %, Σ 95 %), respectively, of the hydrobromide salt as a colourless solid (m. p. 215 °C, decomp.). The pyrrolidine hydrobromide **112·HBr**, despite intensive drying (10^{-3} mbar, P_4O_{10}) is under suspicion as being a marginally hygroscopic –as may be suggested from the corresponding elemental analysis values.

$[\alpha]_D^{20}$ = –42 (*c* = 0.335, CH_3OH)

$C_{13}H_{20}BrNO_3$	calc.	C	49.07	H	6.34	N	4.40	Br	25.11
(318.2)	found	C	47.06	H	6.26	N	4.07	Br	24.54
$M\cdot(H_2O)_{0.5}$	calc.	C	47.70	H	6.47	N	4.28	Br	24.41

MS (FAB positive ion, matrix: 3-nitrobenzyl alcohol) *m/z* (%) = 238.1 $[M+H]^+$ (100), 150.1 (4),121.1 (9), 69.1 (5), 55.0 (6).

HRMS (FAB positive ion, matrix: 3-nitrobenzyl alcohol + PEG 200) calc. for $C_{13}H_{19}NO_3$+H: 238.1438; found 238.1433.

IR (neat): $\tilde{\nu}$ = 3380 (bs, OH), 3100 (m, CH_2), 2956 (m, CH_3), 2887 (w), 1613 (w), 1587 (w), 1448 (w), 1406 (m), 1375 (w), 1335 (w), 1280 (w), 1249 (w), 1208 (m), 1165 (w), 1143 (w), 1130 (w), 1007 (m), 965 (w), 885 (m), 866 (m), 828 (m), 695 (m), 604 (m), 582 (m) cm^{-1}.

^1H and ^{13}C NMR data is displayed in Tables 27-29 below, p. 310-312.

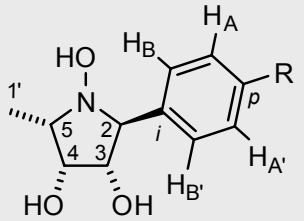
Table 27: ^{1}H NMR Chemical shifts of polyhydroxylated pyrrrolidine diols and triols (δ [ppm], 300.1 MHz or 500.1 MHz, CD_3OD)

Nr.	2-H	3-H	4-H	5-H	5-CH_3	*p*-C_6H_4 and/or other 2-R
83	4.05	4.15	4.10	3.55	1.24	3.78 (OCH_3), 6.89 [2 H, H_A, $H_{A'}$], 7.32 [2 H, H_B, $H_{B'}$]
84·HCl	4.94	4.96	4.45	4.19	1.55	7.50-7.85 [9 H, C_6H_5 and C_6H_4]
85·HCl	4.50	4.55	4.15	4.00	1.45	2.50 (SCH_3), 7.34 [2 H, H_A, $H_{A'}$], 7.46 [2 H, H_B, $H_{B'}$]
96·HCl	4.47	4.55	4.10	3.94	1.41	3.77 (OCH_3), 7.00 [2 H, H_A, $H_{A'}$], 7.40 [2 H, H_B, $H_{B'}$]
97·HCl	4.56	4.64	4.18	4.00	1.46	7.35-7.48 [9 H, C_6H_5 and C_6H_4]
98·HCl	4.50	4.59	4.16	4.02	1.45	7.22 [2 H, H_A, $H_{A'}$], 7.58 [2 H, H_B, $H_{B'}$]
99·HCl	4.49	4.55	4.12	3.91	1.42	7.40 [2 H, H_A, $H_{A'}$], 7.50 [2 H, H_B, $H_{B'}$]
106	4.10	4.05	3.91	3.43	1.21	7.30 [2 H, H_A, $H_{A'}$], 7.37 [2 H, H_B, $H_{B'}$]
100·HCl	4.47	4.55	4.13	3.98	1.42	7.45 [2 H, H_A, $H_{A'}$], 7.55 [2 H, H_B, $H_{B'}$]
101·HCl	4.46	4.58	4.14	4.13	1.43	2.49 (SCH_3), 7.35 [2 H, H_A, $H_{A'}$], 7.40 [2 H, H_B, $H_{B'}$]
102·HCl	4.49	4.60	4.16	3.95	1.43	6.95-7.55 [9 H, C_6H_5 and C_6H_4]
102·HBr	4.55	4.63	4.17	4.00	1.43	7.00-7.50 [9 H, C_6H_5 and C_6H_4]
107	4.48	4.56	4.12	3.92	1.42	6.90-7.50 [9 H, C_6H_5 and C_6H_4]
104·2HCl	4.56	4.55	4.18	4.08	1.46	3.31 [N($C\underline{H}_3$)$_2$], 7.82 [2 H, H_A, $H_{A'}$], 7.90 [2 H, H_B, $H_{B'}$]
112·HBr	3.70	4.13	4.05	3.77	1.37	2.94 ($C\underline{H}_aH_b$), 3.20 ($CH_a\underline{H}_b$), 3.78 (OCH_3), 6.95 [2 H, H_A, $H_{A'}$], 7.30 [2 H, H_B, $H_{B'}$],
103·HCl	4.58	4.65	4.10	3.84	1.40	6.51 (4'-H), 6.69 (3'-H), 7.67 (5'-H)
110·HBr	3.22	4.22	3.96	3.50	1.40	1.08 [C($C\underline{H}_3$)$_3$]

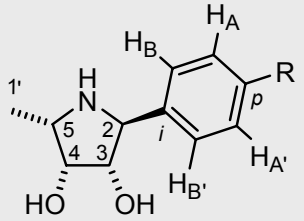
Table 28: Coupling constants of polyhydroxylated pyrrolidines (J [Hz], CD_3OD)

Nr.	$J_{2,3}$	$J_{3,4}$	$J_{4,5}$	$J_{5,5\text{-Me}}$	Other (2-R; AA'BB')
83	6.7	6.0	5.3	7.0	$J(H_A,H_B) = J(H_{A'},H_{B'}) = 8.8$
84·HCl	10.3	3.0	3.4	7.0	—[a]
85·HCl	10.0	3.5	3.2	6.8	$J(H_A,H_B) = 8.3$, $J(H_{A'},H_{B'}) = 8.4$
96·HCl	9.6	3.5	3.0	6.8	$J(H_A,H_B) = J(H_{A'},H_{B'}) = 8.7$
97·HCl	9.6	3.3	2.7	6.6	—[a]
98·HCl	9.7	3.4	2.9	6.6	$J(H_A,H_B) = 8.8$, $J(H_{A'},H_{B'}) = 8.6$
99·HCl	9.5	3.3	2.8	6.7	—[a]
106	8.4	3.7	3.7	6.9	$J(H_A,H_B) = 8.8$, $J(H_{A'},H_{B'}) = 8.7$
100·HCl	9.6	3.4	2.7	6.7	$J(H_A,H_B) = 8.4$, $J(H_{A'},H_{B'}) = 8.6$
101·HCl	9.6	3.1	3.2	6.6	$J(H_A,H_B) = 8.8$, $J(H_{A'},H_{B'}) = 8.7$
102·HCl	9.6	3.0	2.5	6.5	—[a]
102·HBr	9.3	2.8	—	6.6	—[a]
107	9.6	3.0	2.5	6.5	—[a]
104·2HCl	—	2.8	2.8	6.8	$J(H_A,H_B) = J(H_{A'},H_{B'}) = 8.9$
112·HBr	8.8	3.7	3.3	6.7	J(2-H,C$\underline{H}_a$,CH_b) = 10.6, J(2-H,CH_a,C$\underline{H}_b$) = 4.0, ^2J(C$\underline{H}_a$,C$\underline{H}_b$) = 15.0, $J(H_A,H_B) = J(H_{A'},H_{B'}) = 8.8$
103·HCl	9.2	3.5	2.6	6.8	J(3'-H,5'-H) = 1.8, J(4'-H,5'-H) = 1.9, J(3'-H,4'-H) = 3.2
110·HBr	8.6	3.8	2.5	6.7	—

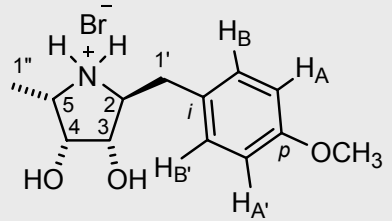
[a] Multiplet



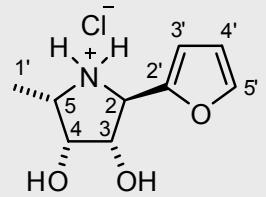
	R =
83	OCH_3
84·HCl	Ph
85·HCl	SCH_3



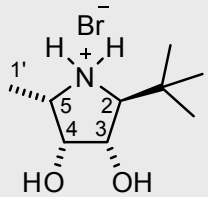
96·HCl	OCH_3
97·HCl	Ph
98·HCl	F
99·HCl	Cl
106	Cl
100·HCl	Br
101·HCl	SCH_3
102·HCl	OPh
102·HBr	OPh
107	OPh
104·2HCl	$N(CH_3)_2$



112·HBr



103·HCl



110·HBr

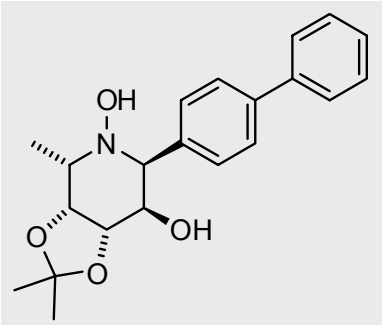
Table 29: ^{13}C NMR Chemical shifts of polyhydroxylated pyrrolidines (δ [ppm], CD_3OD, 75.4 or 125.8 MHz)

Nr.	C-2	C-3	C-4	C-5	5-CH_3	Others; *p*-C_6H_4 or Aryl-C
83	66.0	81.4	75.4	56.1	15.0	56.5 (OCH_3), 114.6 (d, *m*-C), 115.4 (d, *m*-C), 130.6 (s, *i*-C), 133.2 (d, *o*-C), 160.5 (s, *p*-C)
84·HCl	64.6	78.5	73.3	58.7	10.5	128.3, 127.9, 128.9, 130.0, 130.1, 134.6, 142.1, 144.1 [8 d, 3 s of 12 C, some signals overlapping, C_6H_5 and C_6H_4]
85·HCl	64.9	78.5	73.7	58.8	10.4	15.5 (SCH_3), 127.2 (s, *i*-C), 131.4 (d, *m*-C), 133.5 (d, *o*-C), 143.2 (d, *p*-C)
96·HCl	65.5	78.8	74.0	58.9	12.4	56.5 (OCH_3), 116.3 (d, *m*-C), 127.1 (s, *i*-C), 131.4 (d, *o*-C), 164.3 (s, *p*-C)
97·HCl	64.8	78.5	73.5	58.7	12.5	128.0, 128.6, 128.9, 129.8, 130.0, 134.4, 141.3, 143.9 [8 d, 3 s of 12 C, some signals overlapping, C_6H_5 and C_6H_4]
98·HCl	64.2	78.4	73.4	58.6	12.5	117.0 (d, *m*-C), 117.3 (s, *i*-C), 131.7 (d, *o*-C) 131.8 (d, *o*-C), 163.2 (s, *p*-C), 166.4 (s, *p*-C)
99·HCl	64.3	78.5	73.4	58.8	12.4	130.5 (d, *m*-C), 130.0 (s, *i*-C), 134.2 (d, *o*-C), 136.7 (s, *p*-C)
106	65.8	82.1	75.6	57.0	15.2	129.6 (s, *i*-C), 129.8 (d, *m*-C), 134.0 (d, *o*-C),142.9 (s, *p*-C)
100·HCl	64.4	78.6	73.4	58.8	12.4	124.7 (s, *i*-C), 131.3 (d, *o*-C), 133.6 (d, *m*-C), 134.8 (s, *p*-C)
101·HCl	64.7	78.5	73.5	58.6	12.5	15.3 (SCH_3), 127.7 (s, *i*-C), 129.9 (d, *m*-C), 131.7 (d, *o*-C), 142.6 (s, *p*-C)
102·HCl	64.5	78.3	73.4	58.5	12.5	119.9, 120.5, 125.2, 129.8, 131.1, 131.2, 157.9, 160.2 [8 d, 3 s of 12 C, some signals overlapping, C_6H_5 and C_6H_4]
102·HBr	64.5	78.3	73.4	58.6	12.6	120.0, 120.5, 125.2, 129.8, 131.1, 131.3, 157.9, 160.2 [8 d, 3 s of 12 C, some signals overlapping, C_6H_5 and C_6H_4]
107	65.8	81.7	75.4	56.9	15.1	119.8, 119.9, 124.4, 129.4, 129.8, 130.0, 138.3, 158.1, 158.8 [8 d, 3 s of 12 C, some signals overlapping, C_6H_5 and C_6H_4]
104·2HCl	64.1	78.8	73.4	59.0	12.5	46.5 [$N(\underline{C}H_3)_2$], 121.9 [4 d, 1 s, overlap of *o*-C, *m*-C and *p*-C], 131.7 (s, *i*-C)
112·HBr	64.0	77.3	73.0	58.3	12.2	36.8 (CH_2), 55.8 (OCH_3), 115.4 (d, *m*-C), 129.7 (s, *i*-C), 131.0 (d, *o*-C), 160.5 (s, *p*-C)
103·HCl	58.4	76.4	73.2	58.3	12.3	112.1 (2'-C), 112.6 (3'-C), 146.0 (4'-C), 148.6 (5'-C)
110·HBr	71.4	74.0	73.8	58.9	12.1	26.3 [$C(\underline{C}H_3)_2$], 33.2 [$\underline{C}(CH_3)_3$]

8.5.8 Synthesis of 2-([1,1'-biphenyl]-4-yl)-deoxyfuconojirimycin 115·HCl

Experiment 116 (LR 450)

(2*S*,3*R*,4*R*,5*R*,6*S*)-2-([1,1'-Biphenyl]-4-yl)-3,4-*O*-isopropylidene-1,3,4,5-tetrahydroxypiperidine (113)



113

To 200 mg (0.339 mmol) of the 2-bromomethyl-*N*-hydroxypiperidine **52** and THF (4 mL) was added $LiAlH_4$ (27 mg, 0.721 mmol, 2.1 Eq.) at 0 °C. The reaction was followed by TLC analysis: after 5 min a new bottom spot was observed. The ice-bath was removed and stirred for 1 h. TLC analysis (PE/EE = 8:2) then indicated the formation of a new, UV-active, base-line spot and consumption of the starting material. The reaction was cooled to 0 °C and quenched with 5 % citric acid solution (3 mL) and water was added (3 mL). The suspension was filtered over celite and washed with ca. 50 mL ethyl acetate . The aqueous phase was washed with ethyl acetate (3 x 25 mL) and the combined organic layers were dried ($MgSO_4$) and concentrated under vacuum (20 °C/10 mbar). The crude product was purified using column chromatography (SiO_2, 25 g, 2.5 cm x 8 cm; eluant: PE/EE = 9:1 then 1:1) to obtain 1.5 mg of recovered starting material (0.00254 mmol, 0.7 %), 4 mg of an unknown co-product and 68 mg (0.191 mmol, 57 %) of the target compound **113** as a analytically nearly pure, colourless solid (m. p. 173-175 °C).

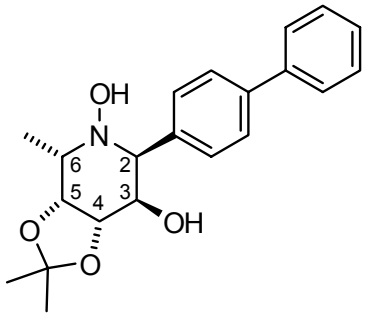
$[\alpha]_D^{20}$ = 5 (*c* = 0.065, CH_2Cl_2)

$C_{21}H_{25}NO_4Si$	calc.	C	70.96	H	7.09	N	3.94
(355.4)	found	C	70.30	H	7.22	N	3.64

MS (ESI, positive ion) *m/z* (%) = 378 $[M+Na]^+$ (100), 356 $[M+H]^+$ (50).
HRMS (ESI, positive ion): calc. for $C_{21}H_{25}NO_4 + H^+$: calc. 356.1856; found 356.1862.

IR (neat): $\tilde{\nu}$ = 3520 (m), 3075 (w), 3030 (m), 2987 (m), 2925 (w), 1518 (m), 1485 (w), 1382 (m), 1333 (w), 1262 (m), 1209 (vs), 1158 (m), 1134 (m), 1112 (m), 1060 (s), 1039 (s), 1008 (vs), 908 (w), 871 (m), 841 (m), 813 (w), 801 (m), 758 (vs), 728 (m), 697 (vs), 619 (w) cm^{-1}.

^{1}H NMR ($CDCl_3$, 500.1 MHz): δ = 1.40, 1.62 [2 s, $C(CH_3)_2$], 1.50 (d, $J_{6,1'}$ = 7.2 Hz, 3 H, 1'-CH_3), 2.04 (bs, 1 H, OH), 3.76 ('dd', $J_{5,6}$ = 1.7, $J_{6,1'}$ = 7.3 Hz, 1 H, 6-H), 4.08 ('t', $J_{2,3} \approx J_{3,4}$ = 2.7 Hz, 1 H, 3 H), 4.40-4.47 (dd, $J_{4,5}$ = 7.3, $J_{5,6}$ = 1.7 Hz, 5-H, overlapping with dd, $J_{3,4}$ = 2.7, $J_{4,5}$ = 7.3 Hz, 4-H, together 2 H), 5.25 (bs, 1 H, OH), 4.56 (d, $J_{2,3}$ = 3.0 Hz, 1 H, 2-H), 7.30-7.80 (m, 9 H, C_6H_5 and C_6H_4).



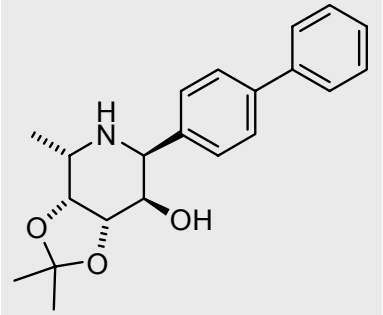
113

^{13}C NMR ($CDCl_3$, 125.8 MHz): δ = 15.6 (q, C-1'), 24.0, 27.1 [2 s, $C(CH_3)_3$], 54.2 (d, C-6), 67.0 (d, C-2), 71.8 (d, C-3), 75.8 (d, C-4), 76.3 (d, C-5), 109.4 [s, $C(CH_3)_2$)], 127.1, 127.3, 127.5, 125.52, 127.56, 128.59, 128.8, 131.8, 140.4, 140.8 [9 d, 3 s, 10 signals found, 12 expected, some signals overlapping of C_6H_5 and C_6H_4].

Signal correlations were established with the help of H,H- and C,H COSY.

Experiment 117 (LR 451)

(2*S*,3*R*,4*R*,5*R*,6*S*)-2-([1,1'-Biphenyl]-4-yl)-3,4-*O*-isopropylidene-3,4,5-trihydroxypiperidine (114)



114

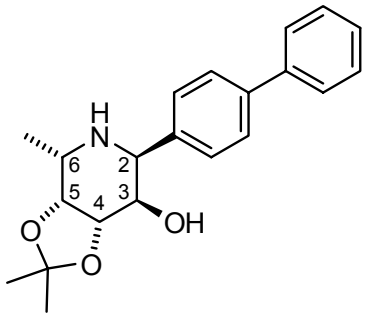
To a one-necked 10 mL flask containing 20 mg (0.00563 mmol) *N*-hydroxypiperidine **113** was added 1.2 mL glacial acetic acid, 0.8 mL water and 74 mg (1.13 mmol, 20 Eq.) zinc dust. The suspension was heated to 55 °C for 2 h, then cooled to room temp. and made basic (pH 10) with 2 N ammonia solution.The remaing solids were filtered off and the filtrate was washed with ethyl acetate (3 x 25 mL), followed by washing of the combined organic layers with 10 mL saturated NaCl solution, drying ($MgSO_4$), and concentration under vacuum (20 °C/10 mbar). The crude product was purified using column chromatography (SiO_2, 25 g, 1.5 cm x 8 cm; eluant: PE:EE = 3:7), to yield the title compound **114** (16.2 mg, 0.0477 mmol, 85 %) as a spectroscopically pure, light-red solid (m. p. 148-150 °C).

$[\alpha]_D^{20}$ = –25.0 (*c* = 0.040, CH_2Cl_2)

MS (ESI, positive ion) *m/z* (%) = 362 $[M+Na]^+$ (35), 340 $[M+H]^+$ (100).
HRMS (ESI, positive ion): calc. for $C_{21}H_{25}NO_3$ + H: calc. 340.1907; found 340.1900.

IR (neat): $\tilde{\nu}$ = 2986 (m), 2930 (w), 2875 (w), 1485 (m), 1447 (w), 1377 (m), 1253 (m), 1207 (s), 1160 (m), 1133 (m), 1107 (w), 1088 (m), 1055 (s), 998 (s), 950 (w), 908 (m), 891 (m), 873 (m), 827 (m), 762 (vs), 729 (s), 691 (s) cm^{-1}.

^1H NMR ($CDCl_3$, 500.1 MHz): δ = 1.25 (d, $J_{6,1'}$ = 6.8 Hz, 3 H, 6-CH_3), 1.41, 1.58 [2 s, together 6 H, $C(CH_3)_2$], 1.84 (bs, 1 H, NH), 3.52 ('dd', $J_{5,6}$ = 1.8, $J_{6,1'}$ = 6.8 Hz, 1 H, 6-H), 3.97 ('t', $J_{2,3} \approx J_{3,4}$ = 2.8 Hz, 1 H, 3 H), 4.21 (dd, $J_{4,5}$ = 7.5, $J_{5,6}$ = 1.8 Hz, 1 H, 5-H), 4.47 (dd, $J_{3,4}$ = 2.6, $J_{4,5}$ = 7.5 Hz, 1 H, 4-H), 5.10 (d, $J_{2,3}$ = 3.0 Hz, 1 H, 2-H), 7.30-7.65 (m, 9 H, C_6H_5 and C_6H_4).

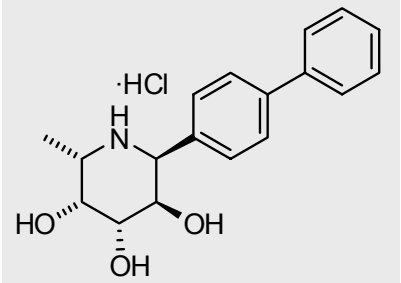


114

^{13}C NMR ($CDCl_3$, 125.8 MHz): δ = 14.1 (q, C-1'), 24.1, 26.8 [2 s, $C(CH_3)_3$], 45.9 (d, C-6), 54.1 (d, C-2), 71.0 (d, C-3), 75.2 (d, C-4), 75.3 (d, C-5), 108.8 [s, $C(CH_3)_2$)], 127.1, 123.2, 127.4, 127.7, 128.8, 140.1, 140.9, 141.3 [9 d, 3 s, 10 signals found, 12 expected, some signals of C_6H_5 and C_6H_4 overlapping, not assigned].

Experiment 118 (LR 452

(2*S*)-2-([1,1'-Biphenyl]-4-yl)-deoxyfuconojirimycin hydrochloride (115·HCl)



115·HCl

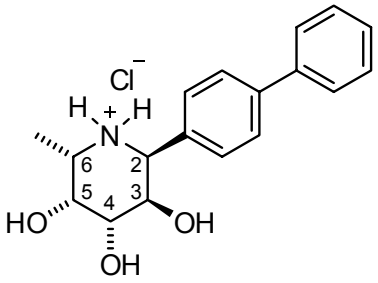
The cyclic amine **114** obtained in the last experiment (13.1 mg, 0.0386 mmol) was taken up in 2 mL methanol, cooled briefly to 0 °C, and treated with conc. HCl (0.6 mL). The solution was stirred at room temp. overnight and the solvents were removed on the rotary evaporator (40 °C/10 mbar). This yielded the title compound as a light-brown solid which was recrystallised as follows: The solid was dissolved in a minimal amount of methanol, a ether-bridge was fitted and a small flask containing diethyl ether was fitted on the other end. After 72 h, the title compound **115·HCl** had precipitated as very fine, colourless crystals. The

methanol solution was removed by pipette and the crystals were then washed carefully with cold diethyl ether (3 x 1 mL). This yielded the target compound **115·HCl** (11.7 mg, 0.0348 mmol, 90 %) as a spectroscopically pure, colourless solid (m. p. >223 °C, decomp.).

$[\alpha]_D^{20}$ = 18.2 (*c* = 0.11, CH_3OH)

MS (ESI, positive ion) *m/z* (%) = 300 $[M+H]^+$ (100), 282 $[-H_2O]$ (5).
HRMS (ESI, positive ion): calc. for $C_{18}H_{21}NO_3$ + H: calc. 300.1594; found 300.1591.

IR (neat): $\tilde{\nu}$ = 3617 (w), 3320 (bs), 3013 (w), 2954 (w), 1557 (m), 1488 (m), 1429 (m), 1392 (m), 1314 (w), 1267 (m), 1242 (m), 1198 (w), 1159 (m), 1095 (m), 1072 (s), 1040 (s), 1004 (w), 958 (m), 938 (m), 826 (m), 764 (m), 730 (w), 710 (m), 690 (m), 590 (m) cm^{-1}.



115·HCl

^1H NMR (CD_3OD, 500.1 MHz): δ = 1.62 (d, $J_{6,1'}$ = 7.2 Hz, 3 H, 6-CH_3), 3.74 (quint, $J_{6,1'}$ = 7.1 Hz, 1 H, 6-H), 3.97 (dd, $J_{2,3}$ = 4.3, $J_{3,4}$ = 1.7 Hz, 1 H, 3-H), 4.05 (m, 1 H, 4-H), 4.24 (dd, $J_{4,5}$ = 5.8, $J_{5,6}$ = 3.3 Hz, 1 H, 5-H), 4.77 ('d', $J_{2,3}$ = 1.7 Hz, 1 H, 2-H), 7.30-7.80 (m, 9 H, C_6H_5 and C_6H_4).

^{13}C NMR (CD_3OD, 125.8 MHz): δ = 11.9 (q, C-1'), 53.5 (d, C-2), 54.4 (d, C-6), 64.8 (d, C-5), 72.6 (d, C-3), 73.0 (d, C-4), 128.0, 128.4, 128.9, 129.6, 130.1, 134.7, 141.5, 143.3 [9 d, 3 s, 10 signals found, 12 expected, some signals of C_6H_5 and C_6H_4 overlapping, not assigned].

Signal correlations were established with the help of H,H- and C,H-COSY.

9 X-Ray Crystal Structure Data

9.1 (2*S*,3*R*,4*S*)-2-Bromomethyl-3,4-dihydroxy-3,4-O-isopropylidene-3,4-dihydro-2*H*-pyrrole-1-oxide (6)

$C_8H_{12}BrNO_3$

monoclinic, $P2_1$

a = 6.381(1) Å

b = 17.294(4) Å

c = 9.254(2) Å

α = 90.0 °

β = 98.39(2) °

γ = 90.0 °

V = 1010.2 Å^3

Z = 4, R(*F*) = 0.062

$R_w(F^2)$ = 0.1044

Crystal size: 0.8 x 0.4 x 0.1 mm

Calculated density: 1.644 g/cm^3

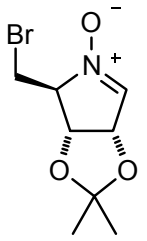
2θ-Range for data collection: 2.22 – 28.99 °

Independent reflections: 2768

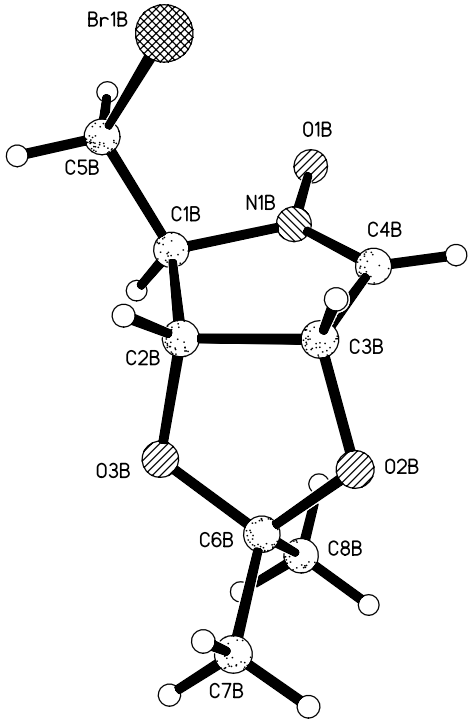
Observed reflections: 2317

Contributed reflections to refinement: 2768

Refined parameters: 236

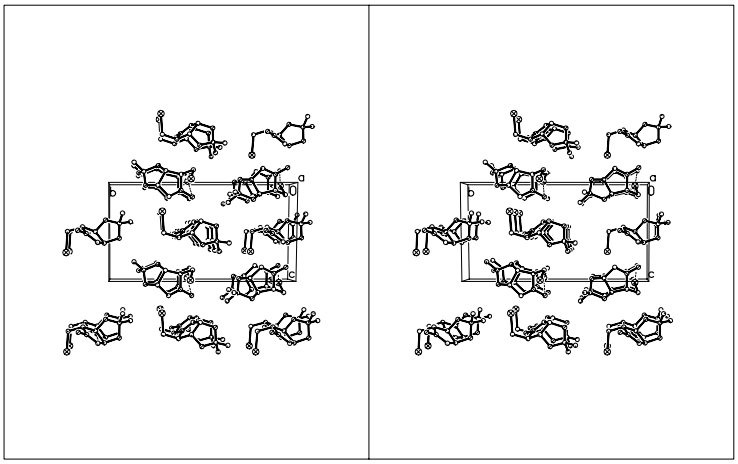


6 D-*ribo*

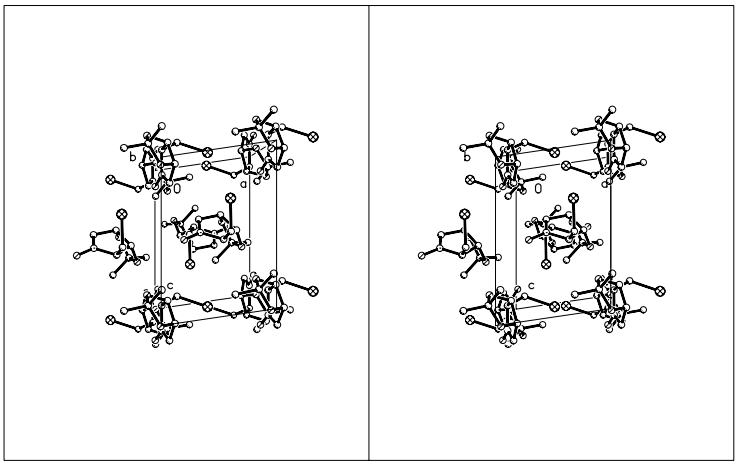


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

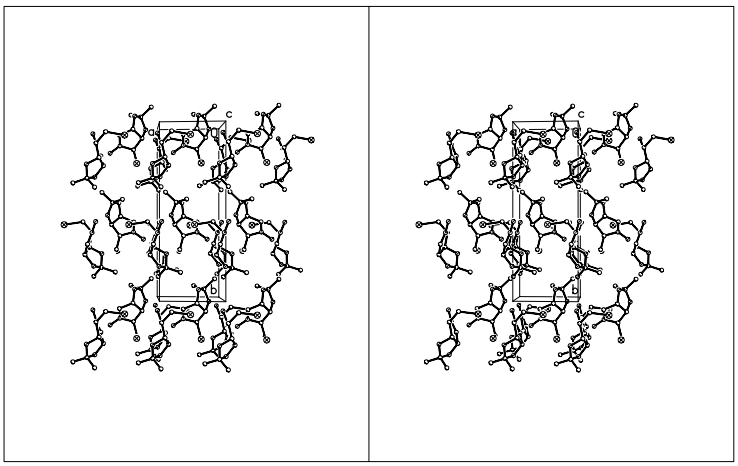
(a)



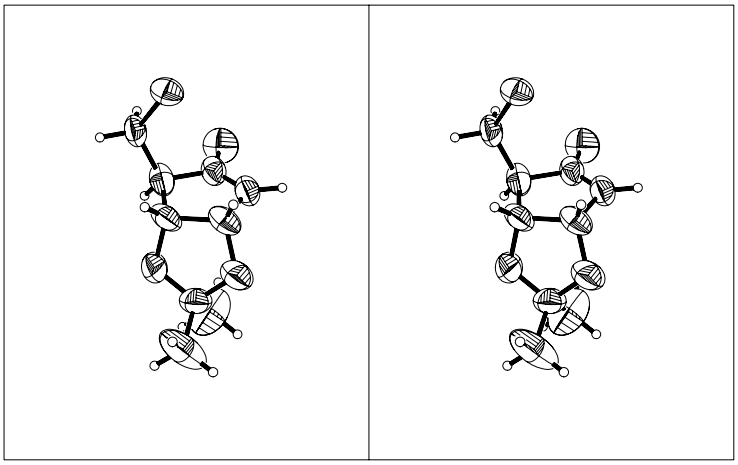
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

Br(1A)-C(5A)	1.939(9)	O(3B)-C(6B)	1.382(14)
N(1A)-O(1A)	1.282(10)	C(3B)-C(4B)	1.484(13)
N(1A)-C(4A)	1.286(12)	C(3B)-H(3B)	0.98
N(1A)-C(1A)	1.496(10)	C(4B)-H(4B)	0.93
C(1A)-C(2A)	1.490(13)	C(5B)-H(5B1)	0.97
C(1A)-C(5A)	1.508(12)	C(5B)-H(5B2)	0.97
C(1A)-H(1A)	0.98	C(6B)-C(7B)	1.475(16)
C(2A)-O(2A)	1.417(11)	C(6B)-C(8B)	1.485(15)
C(2A)-C(3A)	1.537(13)	C(7B)-H(7B1)	0.96
C(2A)-H(2A)	0.98	C(7B)-H(7B2)	0.96
O(2A)-C(6A)	1.434(13)	C(7B)-H(7B3)	0.96
O(3A)-C(3A)	1.402(11)	C(8B)-H(8B1)	0.96
O(3A)-C(6A)	1.409(12)	C(8B)-H(8B2)	0.96
C(3A)-C(4A)	1.499(12)	C(8B)-H(8B3)	0.96
C(3A)-H(3A)	0.98	O(1A)-N(1A)-C(4A)	127.7(8)
C(4A)-H(4A)	0.93	O(1A)-N(1A)-C(1A)	119.8(8)
C(5A)-H(5A1)	0.97	C(4A)-N(1A)-C(1A)	127.5(8)
C(5A)-H(5A2)	0.97	C(2A)-C(1A)-N(1A)	104.1(7)
C(6A)-C(8A)	1.476(14)	C(2A)-C(1A)-C(5A)	116.9(8)
C(6A)-C(7A)	1.489(15)	N(1A)-C(1A)-C(5A)	109.3(7)
C(7A)-H(7A1)	0.96	C(2A)-C(1A)-H(1A)	108.8
C(7A)-H(7A2)	0.96	N(1A)-C(1A)-H(1A)	108.8
C(7A)-H(7A3)	0.96	C(5A)-C(1A)-H(1A)	108.8
C(8A)-H(8A1)	0.96	O(2A)-C(2A)-C(1A)	108.9(8)
C(8A)-H(8A2)	0.96	O(2A)-C(2A)-C(3A)	102.7(7)
C(8A)-H(8A3)	0.96	C(1A)-C(2A)-C(3A)	107.1(7)
Br(1B)-C(5B)	1.929(9)	O(2A)-C(2A)-H(2A)	112.5
N(1B)-O(1B)	1.289(10)	C(1A)-C(2A)-H(2A)	112.5
N(1B)-C(4B)	1.292(12)	C(3A)-C(2A)-H(2A)	112.5
N(1B)-C(1B)	1.473(11)	C(2A)-O(2A)-C(6A)	107.7(7)
C(1B)-C(5B)	1.492(15)	C(3A)-O(3A)-C(6A)	109.5(8)
C(1B)-C(2B)	1.511(17)	O(3A)-C(3A)-C(4A)	111.9(8)
C(1B)-H(1B)	0.98	O(3A)-C(3A)-C(2A)	105.6(7)
C(2B)-O(3B)	1.420(13)	C(4A)-C(3A)-C(2A)	102.8(7)
C(2B)-C(3B)	1.545(16)	O(3A)-C(3A)-H(3A)	112.0
C(2B)-H(2B)	0.9800	C(4A)-C(3A)-H(3A)	112.0
O(2B)-C(6B)	1.415(12)	C(2A)-C(3A)-H(3A)	112.0
O(2B)-C(3B)	1.431(13)	N(1A)-C(4A)-C(3A)	112.5(8)

N(1A)-C(4A)-H(4A)	123.7
C(3A)-C(4A)-H(4A)	123.7
C(1A)-C(5A)-Br(1A)	112.5(6)
C(1A)-C(5A)-H(5A1)	109.1
Br(1A)-C(5A)-H(5A1)	109.1
C(1A)-C(5A)-H(5A2)	109.1
Br(1A)-C(5A)-H(5A2)	109.1
H(5A1)-C(5A)-H(5A2)	107.8
O(3A)-C(6A)-O(2A)	103.9(8)
O(3A)-C(6A)-C(8A)	110.2(9)
O(2A)-C(6A)-C(8A)	108.7(10)
O(3A)-C(6A)-C(7A)	109.8(10)
O(2A)-C(6A)-C(7A)	109.8(9)
C(8A)-C(6A)-C(7A)	113.9(10)
C(6A)-C(7A)-H(7A1)	109.5
C(6A)-C(7A)-H(7A2)	109.5
H(7A1)-C(7A)-H(7A2)	109.5
C(6A)-C(7A)-H(7A3)	109.5
H(7A1)-C(7A)-H(7A3)	109.5
H(7A2)-C(7A)-H(7A3)	109.5
C(6A)-C(8A)-H(8A1)	109.5
C(6A)-C(8A)-H(8A2)	109.5
H(8A1)-C(8A)-H(8A2)	109.5
C(6A)-C(8A)-H(8A3)	109.5
H(8A1)-C(8A)-H(8A3)	109.5
H(8A2)-C(8A)-H(8A3)	109.5
O(1B)-N(1B)-C(4B)	127.8(8)
O(1B)-N(1B)-C(1B)	118.4(9)
C(4B)-N(1B)-C(1B)	113.8(9)
N(1B)-C(1B)-C(5B)	110.5(8)
N(1B)-C(1B)-C(2B)	103.3(8)
C(5B)-C(1B)-C(2B)	115.7(8)
N(1B)-C(1B)-H(1B)	109.0
C(5B)-C(1B)-H(1B)	109.0
C(2B)-C(1B)-H(1B)	109.0
O(3B)-C(2B)-C(1B)	112.4(10)
O(3B)-C(2B)-C(3B)	103.0(10)
C(1B)-C(2B)-C(3B)	106.9(7)
O(3B)-C(2B)-H(2B)	111.4
C(1B)-C(2B)-H(2B)	111.4
C(3B)-C(2B)-H(2B)	111.4
C(6B)-O(2B)-C(3B)	109.2(8)
C(6B)-O(3B)-C(2B)	112.9(9)
O(2B)-C(3B)-C(4B)	114.7(9)
O(2B)-C(3B)-C(2B)	105.1(8)
C(4B)-C(3B)-C(2B)	102.6(9)
O(2B)-C(3B)-H(3B)	111.3
C(4B)-C(3B)-H(3B)	111.3
C(2B)-C(3B)-H(3B)	111.3
N(1B)-C(4B)-C(3B)	112.5(9)
N(1B)-C(4B)-H(4B)	123.7
C(3B)-C(4B)-H(4B)	123.7
C(1B)-C(5B)-Br(1B)	112.4(7)
C(1B)-C(5B)-H(5B1)	109.1
Br(1B)-C(5B)-H(5B1)	109.1
C(1B)-C(5B)-H(5B2)	109.1
Br(1B)-C(5B)-H(5B2)	109.1
H(5B1)-C(5B)-H(5B2)	107.9
O(3B)-C(6B)-O(2B)	107.4(8)
O(3B)-C(6B)-C(7B)	111.9(11)
O(2B)-C(6B)-C(7B)	108.2(10)
O(3B)-C(6B)-C(8B)	107.5(11)
O(2B)-C(6B)-C(8B)	111.2(11)
C(7B)-C(6B)-C(8B)	110.6(12)
C(6B)-C(7B)-H(7B1)	109.5
C(6B)-C(7B)-H(7B2)	109.5
H(7B1)-C(7B)-H(7B2)	109.5
C(6B)-C(7B)-H(7B3)	109.5
H(7B1)-C(7B)-H(7B3)	109.5
H(7B2)-C(7B)-H(7B3)	109.5
C(6B)-C(8B)-H(8B1)	109.5
C(6B)-C(8B)-H(8B2)	109.5
H(8B1)-C(8B)-H(8B2)	109.5
C(6B)-C(8B)-H(8B3)	109.5
H(8B1)-C(8B)-H(8B3)	109.5
H(8B2)-C(8B)-H(8B3)	109.5

Torsion angles [°]

O(1A)-N(1A)-C(1A)-C(2A)	173.1(8)
C(4A)-N(1A)-C(1A)-C(2A)	-8.2(9)
O(1A)-N(1A)-C(1A)-C(5A)	-61.3(10)
C(4A)-N(1A)-C(1A)-C(5A)	117.4(8)
N(1A)-C(1A)-C(2A)-O(2A)	-101.1(7)
C(5A)-C(1A)-C(2A)-O(2A)	138.3(8)
N(1A)-C(1A)-C(2A)-C(3A)	9.3(9)
C(5A)-C(1A)-C(2A)-C(3A)	-111.3(8)
C(1A)-C(2A)-O(2A)-C(6A)	140.3(7)
C(3A)-C(2A)-O(2A)-C(6A)	27.0(9)
C(6A)-O(3A)-C(3A)-C(4A)	-121.3(9)
C(6A)-O(3A)-C(3A)-C(2A)	-10.2(10)
O(2A)-C(2A)-C(3A)-O(3A)	-10.4(9)
C(1A)-C(2A)-C(3A)-O(3A)	-125.1(8)
O(2A)-C(2A)-C(3A)-C(4A)	107.1(8)
C(1A)-C(2A)-C(3A)-C(4A)	-7.6(9)
O(1A)-N(1A)-C(4A)-C(3A)	-178.0(8)
C(1A)-N(1A)-C(4A)-C(3A)	3.4(10)
O(3A)-C(3A)-C(4A)-N(1A)	115.6(9)
C(2A)-C(3A)-C(4A)-N(1A)	2.7(10)
C(2A)-C(1A)-C(5A)-Br(1A)	59.1(10)
N(1A)-C(1A)-C(5A)-Br(1A)	-58.7(9)
C(3A)-O(3A)-C(6A)-O(2A)	26.6(10)
C(3A)-O(3A)-C(6A)-C(8A)	143.0(9)
C(3A)-O(3A)-C(6A)-C(7A)	-90.8(11)
C(2A)-O(2A)-C(6A)-O(3A)	-33.8(10)
C(2A)-O(2A)-C(6A)-C(8A)	-151.2(9)
C(2A)-O(2A)-C(6A)-C(7A)	83.6(10)
O(1B)-N(1B)-C(1B)-C(5B)	-61.9(11)
C(4B)-N(1B)-C(1B)-C(5B)	116.8(9)
O(1B)-N(1B)-C(1B)-C(2B)	173.8(8)
C(4B)-N(1B)-C(1B)-C(2B)	-7.5(10)
N(1B)-C(1B)-C(2B)-O(3B)	-102.3(9)
C(5B)-C(1B)-C(2B)-O(3B)	136.9(9)
N(1B)-C(1B)-C(2B)-C(3B)	10.0(10)
C(5B)-C(1B)-C(2B)-C(3B)	-110.8(9)
C(1B)-C(2B)-O(3B)-C(6B)	117.2(12)
C(3B)-C(2B)-O(3B)-C(6B)	2.5(13)
C(6B)-O(2B)-C(3B)-C(4B)	-96.4(10)
C(6B)-O(2B)-C(3B)-C(2B)	15.5(11)
O(3B)-C(2B)-C(3B)-O(2B)	-10.8(11)

C(1B)-C(2B)-C(3B)-O(2B)	-129.4(9)	C(2B)-C(1B)-C(5B)-Br(1B)	55.7(10)
O(3B)-C(2B)-C(3B)-C(4B)	109.5(10)	C(2B)-O(3B)-C(6B)-O(2B)	7.0(13)
C(1B)-C(2B)-C(3B)-C(4B)	-9.2(11)	C(2B)-O(3B)-C(6B)-C(7B)	125.7(11)
O(1B)-N(1B)-C(4B)-C(3B)	-179.8(9)	C(2B)-O(3B)-C(6B)-C(8B)	-112.7(13)
C(1B)-N(1B)-C(4B)-C(3B)	1.6(12)	C(3B)-O(2B)-C(6B)-O(3B)	-14.3(12)
O(2B)-C(3B)-C(4B)-N(1B)	118.3(10)	C(3B)-O(2B)-C(6B)-C(7B)	-135.3(11)
C(2B)-C(3B)-C(4B)-N(1B)	4.9(11)	C(3B)-O(2B)-C(6B)-C(8B)	103.0(11)
N(1B)-C(1B)-C(5B)-Br(1B)	-61.2(9)		

9.2 4-*O*-*tert*-Butyldimethylsilyl-2,3-O-isopropylidene-D-lyxopyranose (10)

$C_{14}H_{28}O_5Si$

monoclinic, $P2_1$

a = 6.7174(16) Å

b = 12.002(4) Å

c = 11.614(2) Å

α = 90 °

β = 106.135(17) °

γ = 90 °

V = 899.5(4) Å^3

Z = 2, R(*F*) = 0.0429

$R_w(F^2)$ = 0.1148

Crystal size: 1.70 x 1.00 x 1.00 mm

Calculated density: 1.124 g/cm^3

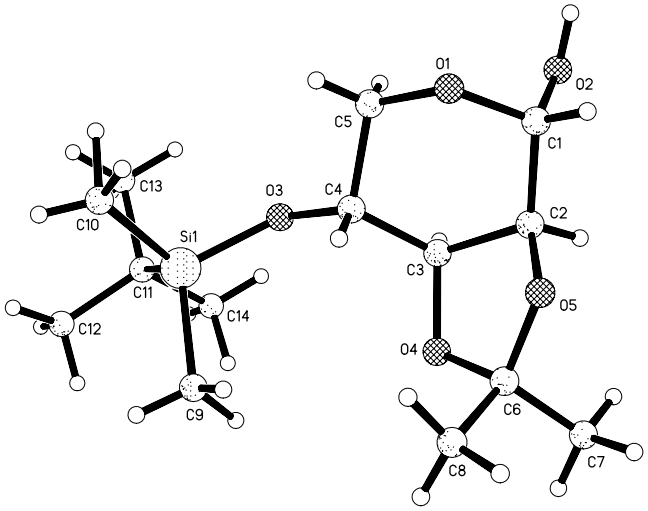
2θ-Range for data collection: 1.83 – 30.00 °

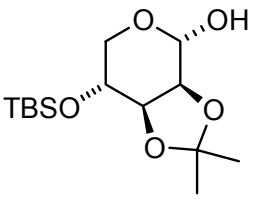
Independent reflections: 7166

Observed reflections: 5264

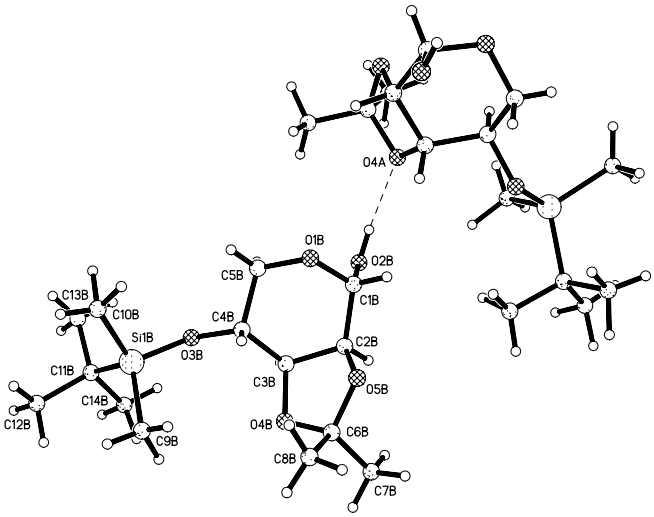
Contributed reflections to refinement: 7166

Refined parameters: 294



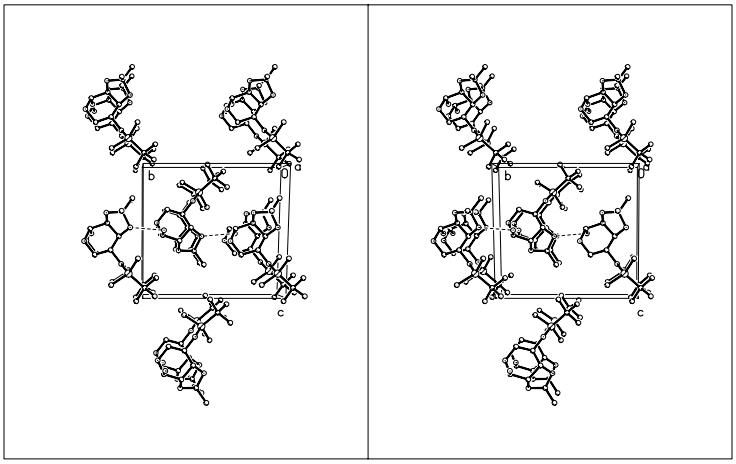


10

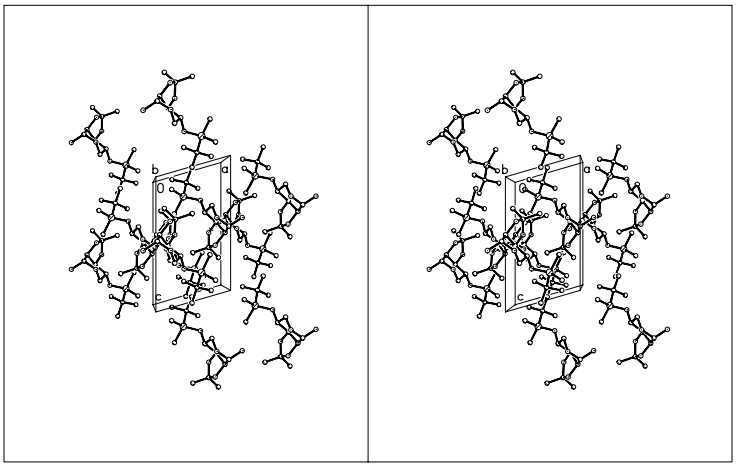


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

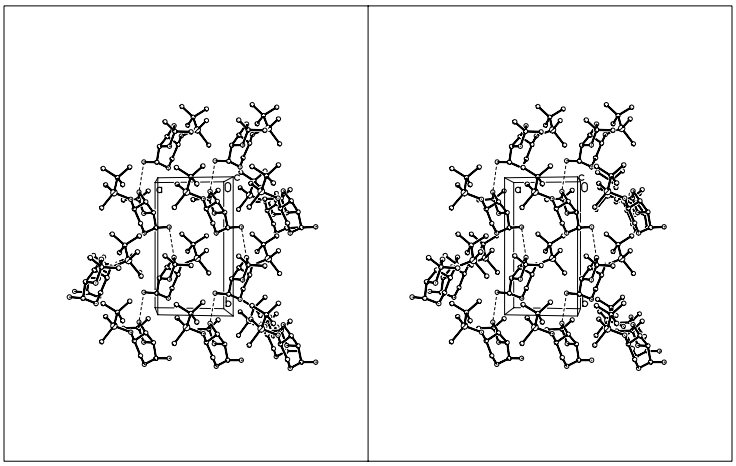
(a)



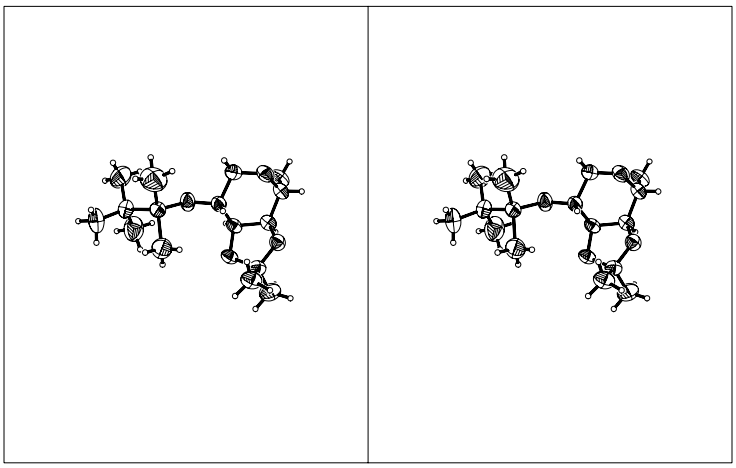
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

Si(1)-O(3)	1.6537(15)	C(11)-C(14)	1.536(5)
Si(1)-C(9)	1.850(3)	C(12)-H(12A)	0.99(5)
Si(1)-C(10)	1.865(5)	C(12)-H(12B)	0.97(7)
Si(1)-C(11)	1.881(3)	C(12)-H(12C)	1.04(5)
O(1)-C(1)	1.403(2)	C(13)-H(13A)	0.97(5)
O(1)-C(5)	1.420(3)	C(13)-H(13B)	1.05(6)
C(1)-O(2)	1.527(2)	C(13)-H(13C)	0.98(4)
C(1)-H(1)	0.96(2)	C(14)-H(14A)	1.06(7)
O(2)-H(2A)	0.86(3)	C(14)-H(14B)	0.95(6)
C(2)-O(5)	1.429(2)	C(14)-H(14C)	0.99(7)
C(2)-C(3)	1.523(2)	O(3)-Si(1)-C(9)	110.79(12)
C(2)-H(2)	1.00(3)	O(3)-Si(1)-C(10)	110.0(2)
O(3)-C(4)	1.421(2)	C(9)-Si(1)-C(10)	108.4(3)
C(3)-O(4)	1.4394(19)	O(3)-Si(1)-C(11)	103.55(10)
C(3)-C(4)	1.521(3)	C(9)-Si(1)-C(11)	111.28(18)
C(3)-H(3)	0.98(3)	C(10)-Si(1)-C(11)	112.7(2)
O(4)-C(6)	1.447(2)	C(1)-O(1)-C(5)	112.46(16)
C(4)-C(5)	1.530(3)	O(2)-C(1)-O(1)	112.55(16)
C(4)-H(4)	0.96(3)	O(2)-C(1)-C(2)	106.17(14)
O(5)-C(6)	1.424(2)	O(1)-C(1)-C(2)	113.04(14)
C(5)-H(5A)	0.94(3)	O(2)-C(1)-H(1)	109.9(13)
C(5)-H(5B)	0.88(3)	O(1)-C(1)-H(1)	103.9(13)
C(6)-C(8)	1.516(3)	C(2)-C(1)-H(1)	111.3(13)
C(6)-C(7)	1.519(3)	C(1)-O(2)-H(2A)	105.9(17)
C(7)-H(7A)	1.00(4)	O(5)-C(2)-C(3)	101.63(13)
C(7)-H(7B)	1.03(5)	O(5)-C(2)-C(1)	110.16(14)
C(7)-H(7C)	0.95(3)	C(3)-C(2)-C(1)	116.03(15)
C(8)-H(8A)	0.98(4)	O(5)-C(2)-H(2)	107.8(14)
C(8)-H(8B)	0.97(3)	C(3)-C(2)-H(2)	108.4(15)
C(8)-H(8C)	0.97(4)	C(1)-C(2)-H(2)	112.1(15)
C(9)-H(9A)	1.12(7)	C(4)-O(3)-Si(1)	125.97(12)
C(9)-H(9B)	0.90(5)	O(4)-C(3)-C(4)	111.01(13)
C(9)-H(9C)	0.88(5)	O(4)-C(3)-C(2)	102.76(14)
C(10)-H(10A)	1.12(9)	C(4)-C(3)-C(2)	113.17(13)
C(10)-H(10B)	0.97(9)	O(4)-C(3)-H(3)	107.6(15)
C(10)-H(10C)	0.87(9)	C(4)-C(3)-H(3)	110.9(13)
C(11)-C(12)	1.532(4)	C(2)-C(3)-H(3)	110.9(14)
C(11)-C(13)	1.532(5)	C(3)-O(4)-C(6)	108.54(12)

O(3)-C(4)-C(3)	109.05(16)
O(3)-C(4)-C(5)	109.06(15)
C(3)-C(4)-C(5)	109.81(15)
O(3)-C(4)-H(4)	115.0(15)
C(3)-C(4)-H(4)	107.3(15)
C(5)-C(4)-H(4)	106.5(17)
C(6)-O(5)-C(2)	106.31(13)
O(1)-C(5)-C(4)	110.24(16)
O(1)-C(5)-H(5A)	115.5(19)
C(4)-C(5)-H(5A)	103(2)
O(1)-C(5)-H(5B)	109(2)
C(4)-C(5)-H(5B)	106(2)
H(5A)-C(5)-H(5B)	113(3)
O(5)-C(6)-O(4)	105.52(13)
O(5)-C(6)-C(8)	108.38(16)
O(4)-C(6)-C(8)	109.23(17)
O(5)-C(6)-C(7)	111.52(19)
O(4)-C(6)-C(7)	108.40(17)
C(8)-C(6)-C(7)	113.5(2)
C(6)-C(7)-H(7A)	112.6(19)
C(6)-C(7)-H(7B)	102(2)
H(7A)-C(7)-H(7B)	116(3)
C(6)-C(7)-H(7C)	112.5(19)
H(7A)-C(7)-H(7C)	106(3)
H(7B)-C(7)-H(7C)	107(3)
C(6)-C(8)-H(8A)	107(2)
C(6)-C(8)-H(8B)	107.6(17)
H(8A)-C(8)-H(8B)	118(3)
C(6)-C(8)-H(8C)	111(2)
H(8A)-C(8)-H(8C)	107(3)
H(8B)-C(8)-H(8C)	106(3)
Si(1)-C(9)-H(9A)	105(3)
Si(1)-C(9)-H(9B)	109(3)
H(9A)-C(9)-H(9B)	111(5)
Si(1)-C(9)-H(9C)	110(3)
H(9A)-C(9)-H(9C)	110(4)
H(9B)-C(9)-H(9C)	111(4)
Si(1)-C(10)-H(10A)	104(4)
Si(1)-C(10)-H(10B)	120(5)
H(10A)-C(10)-H(10B)	91(5)
Si(1)-C(10)-H(10C)	116(7)
H(10A)-C(10)-H(10C)	97(6)
H(10B)-C(10)-H(10C)	119(8)
C(12)-C(11)-C(13)	108.5(3)
C(12)-C(11)-C(14)	108.7(3)
C(13)-C(11)-C(14)	109.3(4)
C(12)-C(11)-Si(1)	110.4(3)
C(13)-C(11)-Si(1)	110.4(3)
C(14)-C(11)-Si(1)	109.5(2)
C(11)-C(12)-H(12A)	112(3)
C(11)-C(12)-H(12B)	110(4)
H(12A)-C(12)-H(12B)	118(5)
C(11)-C(12)-H(12C)	116(2)
H(12A)-C(12)-H(12C)	111(3)
H(12B)-C(12)-H(12C)	89(4)
C(11)-C(13)-H(13A)	115(3)
C(11)-C(13)-H(13B)	103(3)
H(13A)-C(13)-H(13B)	111(4)
C(11)-C(13)-H(13C)	104(2)
H(13A)-C(13)-H(13C)	113(4)
H(13B)-C(13)-H(13C)	111(4)
C(11)-C(14)-H(14A)	104(3)
C(11)-C(14)-H(14B)	109(3)
H(14A)-C(14)-H(14B)	100(4)
C(11)-C(14)-H(14C)	110(4)
H(14A)-C(14)-H(14C)	112(5)
H(14B)-C(14)-H(14C)	121(5)

Torsion angles [°]

C(5)-O(1)-C(1)-O(2)	65.0(2)
C(5)-O(1)-C(1)-C(2)	-55.2(2)
O(2)-C(1)-C(2)-O(5)	160.34(15)
O(1)-C(1)-C(2)-O(5)	-75.80(19)
O(2)-C(1)-C(2)-C(3)	-84.93(18)
O(1)-C(1)-C(2)-C(3)	38.9(2)
C(9)-Si(1)-O(3)-C(4)	-50.5(3)
C(10)-Si(1)-O(3)-C(4)	69.4(3)
C(11)-Si(1)-O(3)-C(4)	-169.89(18)
O(5)-C(2)-C(3)-O(4)	-34.54(15)
C(1)-C(2)-C(3)-O(4)	-154.02(13)
O(5)-C(2)-C(3)-C(4)	85.25(16)
C(1)-C(2)-C(3)-C(4)	-34.23(19)
C(4)-C(3)-O(4)-C(6)	-103.10(16)
C(2)-C(3)-O(4)-C(6)	18.18(16)
Si(1)-O(3)-C(4)-C(3)	121.46(17)
Si(1)-O(3)-C(4)-C(5)	-118.63(17)
O(4)-C(3)-C(4)-O(3)	-82.29(16)
C(2)-C(3)-C(4)-O(3)	162.77(13)
O(4)-C(3)-C(4)-C(5)	158.26(15)
C(2)-C(3)-C(4)-C(5)	43.3(2)
C(3)-C(2)-O(5)-C(6)	39.04(16)
C(1)-C(2)-O(5)-C(6)	162.61(14)
C(1)-O(1)-C(5)-C(4)	66.9(2)
O(3)-C(4)-C(5)-O(1)	-178.93(17)
C(3)-C(4)-C(5)-O(1)	-59.5(2)
C(2)-O(5)-C(6)-O(4)	-28.45(17)
C(2)-O(5)-C(6)-C(8)	-145.36(16)
C(2)-O(5)-C(6)-C(7)	89.0(2)
C(3)-O(4)-C(6)-O(5)	5.17(17)
C(3)-O(4)-C(6)-C(8)	121.50(16)
C(3)-O(4)-C(6)-C(7)	-114.40(18)
O(3)-Si(1)-C(11)-C(12)	171.5(3)
C(9)-Si(1)-C(11)-C(12)	52.4(3)
C(10)-Si(1)-C(11)-C(12)	-69.7(3)
O(3)-Si(1)-C(11)-C(13)	-68.6(3)
C(9)-Si(1)-C(11)-C(13)	172.3(3)
C(10)-Si(1)-C(11)-C(13)	50.3(4)
O(3)-Si(1)-C(11)-C(14)	51.7(3)
C(9)-Si(1)-C(11)-C(14)	-67.3(3)
C(10)-Si(1)-C(11)-C(14)	170.6(3)

H-Bond lengths [Å] and angles [°]

O2-O4_$1	2.8281(0.0020)
H2A-O4_$1	1.9807(0.0295)
O2-H2A-O4_$1	169.21(2.44)

9.3 (2*R*,3*R*,4*S*,5*S*)-2-Bromomethyl-1,3,4-trihydroxy-3,4-*O*-isopropylidene-5-(4-methoxyphenyl)-pyrrolidine (41)

$C_{15}H_{20}BrNO_4$

orthorhombic, $P2_12_12_1$

a = 8.5380(3) Å

b = 17.7989(13) Å

c = 21.7474(14) Å

α = 90 °

β = 90 °

γ = 90 °

V = 3304.9(3) Å^3

Z = 8, $R(F)$ = 0.05

$R_w(F^2)$ = 0.1332

Crystal size: 0.55 x 0.55 x 0.30 mm

Calculated density: 1.440 g/cm^3

2θ-Range for data collection: 3.21 - 67.50 °

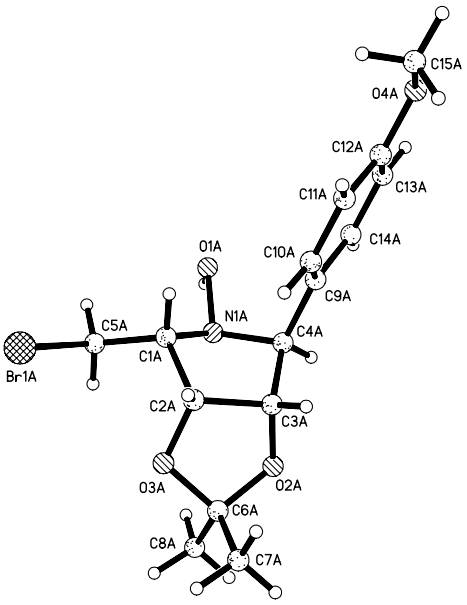
Independent reflections: 5222

Observed reflections: 4776

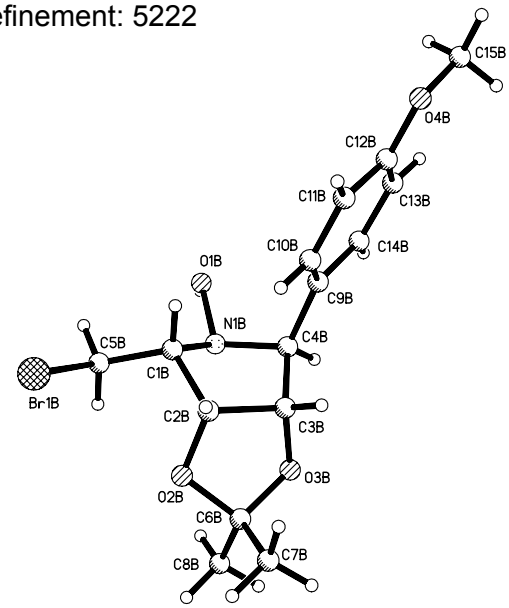
Contributed reflections to refinement: 5222

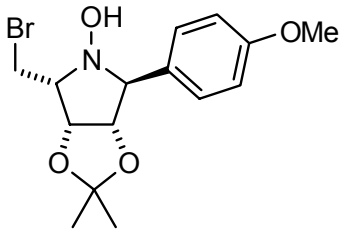
Refined parameters: 385

molecule A

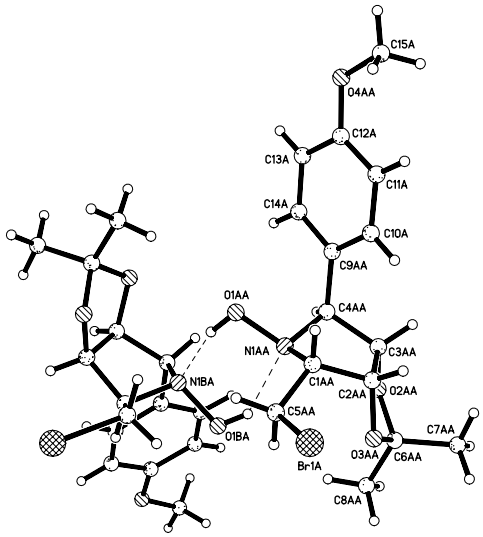


molecule B



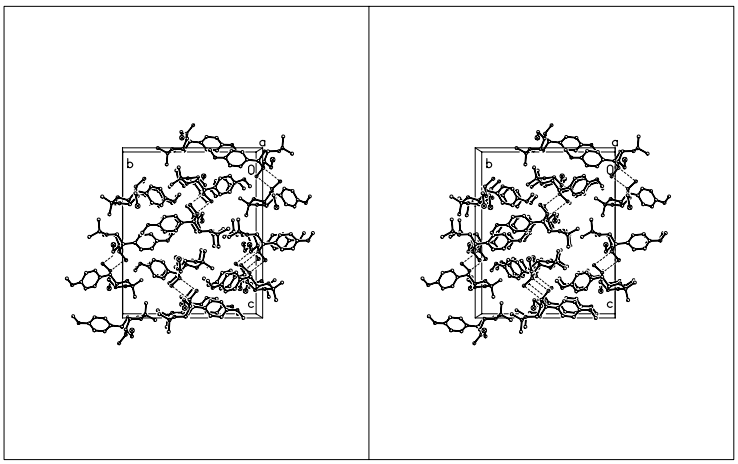


41

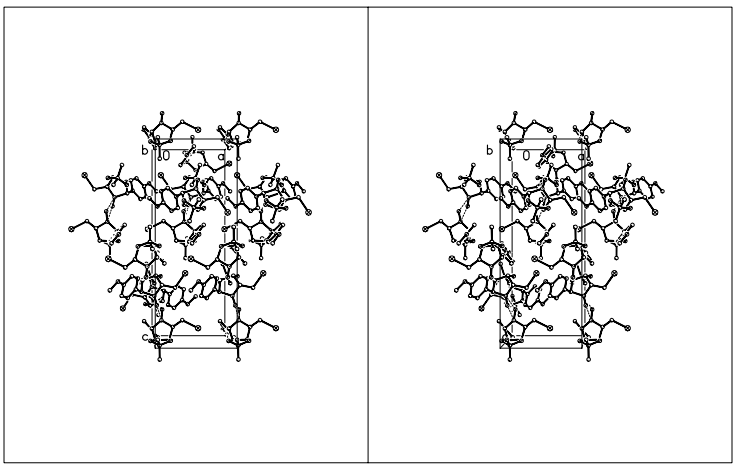


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

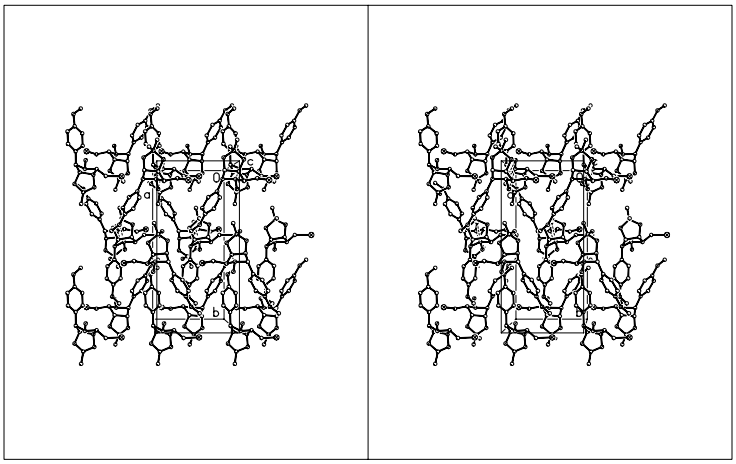
(a)



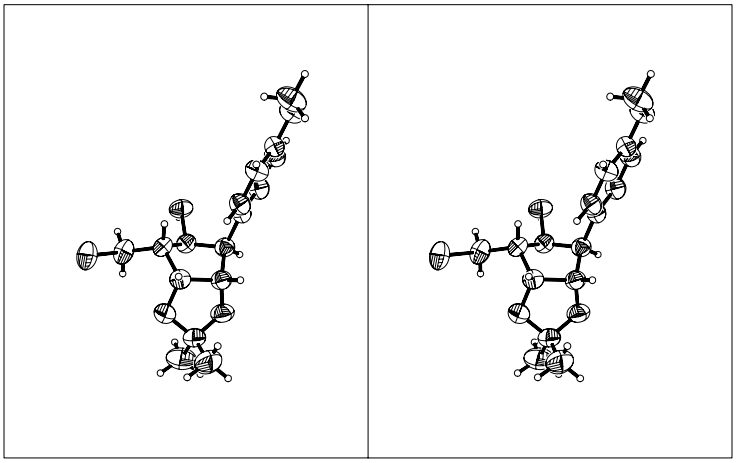
(b)



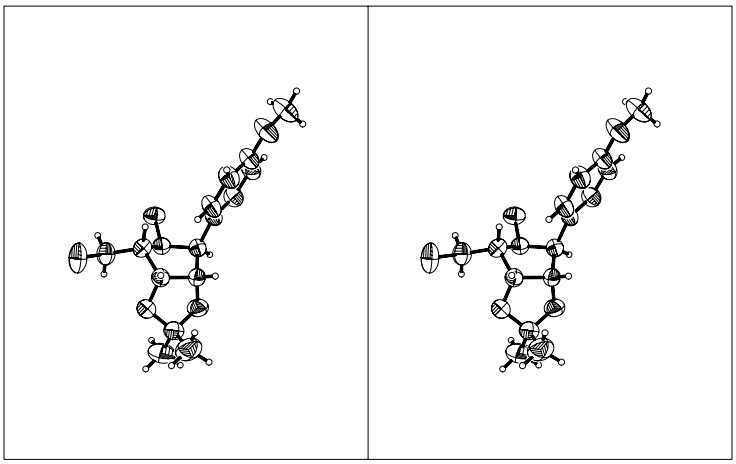
(c)



ORTEP Structure, molecule A



ORTEP Structure, molecule B



Bond lengths [Å] and angles [°]

Br(1A)-C(5A)	1.956(6)	C(3B)-H(3B)	0.98
O(1A)-N(1A)	1.440(5)	O(4B)-C(12B)	1.363(7)
O(1A)-H(1A)	0.98(8)	O(4B)-C(15B)	1.399(9)
N(1A)-C(1A)	1.458(6)	C(4B)-C(9B)	1.517(7)
N(1A)-C(4A)	1.495(6)	C(4B)-H(4B)	0.98
C(1A)-C(2A)	1.513(7)	C(5B)-H(5B1)	0.97
C(1A)-C(5A)	1.517(7)	C(5B)-H(5B2)	0.97
C(1A)-H(1A1)	0.98	C(6B)-C(8B)	1.492(9)
O(2A)-C(6A)	1.416(7)	C(6B)-C(7B)	1.526(10)
O(2A)-C(3A)	1.430(6)	C(7B)-H(7B1)	0.96
C(2A)-O(3A)	1.419(6)	C(7B)-H(7B2)	0.96
C(2A)-C(3A)	1.556(7)	C(7B)-H(7B3)	0.96
C(2A)-H(2A)	0.98	C(8B)-H(8B1)	0.96
O(3A)-C(6A)	1.433(7)	C(8B)-H(8B2)	0.96
C(3A)-C(4A)	1.510(7)	C(8B)-H(8B3)	0.96
C(3A)-H(3A)	0.98	C(9B)-C(10B)	1.394(8)
C(4A)-C(9A)	1.512(6)	C(9B)-C(14B)	1.397(7)
C(4A)-H(4A)	0.98	C(10B)-C(11B)	1.384(8)
O(4A)-C(12A)	1.370(6)	C(10B)-H(10B)	0.93
O(4A)-C(15A)	1.417(8)	C(11B)-C(12B)	1.371(8)
C(5A)-H(5A1)	0.97	C(11B)-H(11B)	0.93
C(5A)-H(5A2)	0.97	C(12B)-C(13B)	1.385(8)
C(6A)-C(8A)	1.494(9)	C(13B)-C(14B)	1.383(8)
C(6A)-C(7A)	1.515(10)	C(13B)-H(13B)	0.93
C(7A)-H(7A1)	0.96	C(14B)-H(14B)	0.93
C(7A)-H(7A2)	0.96	C(15B)-H(15D)	0.96
C(7A)-H(7A3)	0.96	C(15B)-H(15E)	0.96
C(8A)-H(8A1)	0.96	C(15B)-H(15F)	0.96
C(8A)-H(8A2)	0.96	N(1A)-O(1A)-H(1A)	96.0(4)
C(8A)-H(8A3)	0.96	O(1A)-N(1A)-C(1A)	109.0(4)
C(9A)-C(14A)	1.386(7)	O(1A)-N(1A)-C(4A)	110.1(4)
C(9A)-C(10A)	1.390(7)	C(1A)-N(1A)-C(4A)	105.7(3)
C(10A)-C(11A)	1.402(7)	N(1A)-C(1A)-C(2A)	103.1(4)
C(10A)-H(10A)	0.93	N(1A)-C(1A)-C(5A)	107.8(4)
C(11A)-C(12A)	1.376(7)	C(2A)-C(1A)-C(5A)	116.9(4)
C(11A)-H(11A)	0.93	N(1A)-C(1A)-H(1A1)	109.6
C(12A)-C(13A)	1.387(8)	C(2A)-C(1A)-H(1A1)	109.6
C(13A)-C(14A)	1.379(8)	C(5A)-C(1A)-H(1A1)	109.6
C(13A)-H(13A)	0.93	C(6A)-O(2A)-C(3A)	108.8(4)
C(14A)-H(14A)	0.93	O(3A)-C(2A)-C(1A)	111.0(4)
C(15A)-H(15A)	0.96	O(3A)-C(2A)-C(3A)	104.2(4)
C(15A)-H(15B)	0.96	C(1A)-C(2A)-C(3A)	104.5(4)
C(15A)-H(15C)	0.96	O(3A)-C(2A)-H(2A)	112.2
Br(1B)-C(5B)	1.939(6)	C(1A)-C(2A)-H(2A)	112.2
N(1B)-O(1B)	1.440(5)	C(3A)-C(2A)-H(2A)	112.2
N(1B)-C(1B)	1.470(7)	C(2A)-O(3A)-C(6A)	109.5(4)
N(1B)-C(4B)	1.494(6)	O(2A)-C(3A)-C(4A)	110.0(4)
C(1B)-C(5B)	1.498(7)	O(2A)-C(3A)-C(2A)	103.5(4)
C(1B)-C(2B)	1.530(7)	C(4A)-C(3A)-C(2A)	106.1(4)
C(1B)-H(1B1)	0.98	O(2A)-C(3A)-H(3A)	112.2
O(1B)-H(1B)	0.82	C(4A)-C(3A)-H(3A)	112.2
O(2B)-C(2B)	1.414(6)	C(2A)-C(3A)-H(3A)	112.2
O(2B)-C(6B)	1.429(7)	N(1A)-C(4A)-C(3A)	112.4(4)
C(2B)-C(3B)	1.554(7)	N(1A)-C(4A)-C(9A)	114.9(4)
C(2B)-H(2B)	0.98	C(3A)-C(4A)-C(9A)	115.0(4)
O(3B)-C(6B)	1.430(7)	N(1A)-C(4A)-H(4A)	108.7
O(3B)-C(3B)	1.432(6)	C(3A)-C(4A)-H(4A)	108.7
C(3B)-C(4B)	1.514(7)	C(9A)-C(4A)-H(4A)	117.1(5)

C(1A)-C(5A)-Br(1A)	111.2(4)	O(2B)-C(2B)-C(3B)	104.6(4)
C(1A)-C(5A)-H(5A1)	109.4	C(1B)-C(2B)-C(3B)	104.4(4)
Br(1A)-C(5A)-H(5A1)	109.4	O(2B)-C(2B)-H(2B)	112.2
C(1A)-C(5A)-H(5A2)	109.4	C(1B)-C(2B)-H(2B)	112.2
Br(1A)-C(5A)-H(5A2)	109.4	C(3B)-C(2B)-H(2B)	112.2
H(5A1)-C(5A)-H(5A2)	108.0	C(6B)-O(3B)-C(3B)	107.3(4)
O(2A)-C(6A)-O(3A)	103.7(4)	O(3B)-C(3B)-C(4B)	109.7(4)
O(2A)-C(6A)-C(8A)	109.5(6)	O(3B)-C(3B)-C(2B)	103.4(4)
O(3A)-C(6A)-C(8A)	108.3(6)	C(4B)-C(3B)-C(2B)	106.7(4)
O(2A)-C(6A)-C(7A)	110.6(6)	O(3B)-C(3B)-H(3B)	112.2
O(3A)-C(6A)-C(7A)	109.8(6)	C(4B)-C(3B)-H(3B)	112.2
C(8A)-C(6A)-C(7A)	114.4(6)	C(2B)-C(3B)-H(3B)	112.2
C(6A)-C(7A)-H(7A1)	109.5	C(12B)-O(4B)-C(15B)	118.6(5)
C(6A)-C(7A)-H(7A2)	109.5	N(1B)-C(4B)-C(3B)	99.8(4)
H(7A1)-C(7A)-H(7A2)	109.5	N(1B)-C(4B)-C(9B)	116.7(4)
C(6A)-C(7A)-H(7A3)	109.5	C(3B)-C(4B)-C(9B)	112.9(4)
H(7A1)-C(7A)-H(7A3)	109.5	N(1B)-C(4B)-H(4B)	109.0
H(7A2)-C(7A)-H(7A3)	109.5	C(3B)-C(4B)-H(4B)	109.0
C(6A)-C(8A)-H(8A1)	109.5	C(9B)-C(4B)-H(4B)	109.0
C(6A)-C(8A)-H(8A2)	109.5	C(1B)-C(5B)-Br(1B)	110.9(4)
H(8A1)-C(8A)-H(8A2)	109.5	C(1B)-C(5B)-H(5B1)	109.5
C(6A)-C(8A)-H(8A3)	109.5	Br(1B)-C(5B)-H(5B1)	109.5
H(8A1)-C(8A)-H(8A3)	109.5	C(1B)-C(5B)-H(5B2)	109.5
H(8A2)-C(8A)-H(8A3)	109.5	Br(1B)-C(5B)-H(5B2)	109.5
C(14A)-C(9A)-C(10A)	117.4(4)	H(5B1)-C(5B)-H(5B2)	108.0
C(14A)-C(9A)-C(4A)	119.9(5)	O(2B)-C(6B)-O(3B)	103.3(4)
C(10A)-C(9A)-C(4A)	122.6(4)	O(2B)-C(6B)-C(8B)	109.0(6)
C(9A)-C(10A)-C(11A)	122.0(5)	O(3B)-C(6B)-C(8B)	109.4(6)
C(9A)-C(10A)-H(10A)	119.0	O(2B)-C(6B)-C(7B)	109.5(5)
C(11A)-C(10A)-H(10A)	119.0	O(3B)-C(6B)-C(7B)	110.9(6)
C(12A)-C(11A)-C(10A)	118.7(5)	C(8B)-C(6B)-C(7B)	114.2(6)
C(12A)-C(11A)-H(11A)	120.7	C(6B)-C(7B)-H(7B1)	109.5
C(10A)-C(11A)-H(11A)	120.7	C(6B)-C(7B)-H(7B2)	109.5
O(4A)-C(12A)-C(11A)	124.4(5)	H(7B1)-C(7B)-H(7B2)	109.5
O(4A)-C(12A)-C(13A)	115.3(5)	C(6B)-C(7B)-H(7B3)	109.5
C(11A)-C(12A)-C(13A)	120.4(5)	H(7B1)-C(7B)-H(7B3)	109.5
C(14A)-C(13A)-C(12A)	120.0(5)	H(7B2)-C(7B)-H(7B3)	109.5
C(14A)-C(13A)-H(13A)	120.0	C(6B)-C(8B)-H(8B1)	109.5
C(12A)-C(13A)-H(13A)	120.0	C(6B)-C(8B)-H(8B2)	109.5
C(13A)-C(14A)-C(9A)	121.6(5)	H(8B1)-C(8B)-H(8B2)	109.5
C(13A)-C(14A)-H(14A)	119.2	C(6B)-C(8B)-H(8B3)	109.5
C(9A)-C(14A)-H(14A)	119.2	H(8B1)-C(8B)-H(8B3)	109.5
O(4A)-C(15A)-H(15A)	109.5	H(8B2)-C(8B)-H(8B3)	109.5
O(4A)-C(15A)-H(15B)	109.5	C(10B)-C(9B)-C(14B)	116.5(5)
H(15A)-C(15A)-H(15B)	109.5	C(10B)-C(9B)-C(4B)	123.5(4)
O(4A)-C(15A)-H(15C)	109.5	C(14B)-C(9B)-C(4B)	119.9(5)
H(15A)-C(15A)-H(15C)	109.5	C(11B)-C(10B)-C(9B)	122.0(5)
H(15B)-C(15A)-H(15C)	109.5	C(11B)-C(10B)-H(10B)	119.0
O(1B)-N(1B)-C(1B)	110.3(4)	C(9B)-C(10B)-H(10B)	119.0
O(1B)-N(1B)-C(4B)	111.5(4)	C(12B)-C(11B)-C(10B)	120.4(5)
C(1B)-N(1B)-C(4B)	106.1(4)	C(12B)-C(11B)-H(11B)	119.8
N(1B)-C(1B)-C(5B)	110.3(4)	C(10B)-C(11B)-H(11B)	119.8
N(1B)-C(1B)-C(2B)	101.4(4)	O(4B)-C(12B)-C(11B)	115.7(5)
C(5B)-C(1B)-C(2B)	114.8(5)	O(4B)-C(12B)-C(13B)	125.1(5)
N(1B)-C(1B)-H(1B1)	110.0	C(11B)-C(12B)-C(13B)	119.2(5)
C(5B)-C(1B)-H(1B1)	110.0	C(14B)-C(13B)-C(12B)	120.3(5)
C(2B)-C(1B)-H(1B1)	110.0	C(14B)-C(13B)-H(13B)	119.9
N(1B)-O(1B)-H(1B)	109.5	C(12B)-C(13B)-H(13B)	119.9
C(2B)-O(2B)-C(6B)	107.9(4)	C(13B)-C(14B)-C(9B)	121.7(5)
O(2B)-C(2B)-C(1B)	110.7(4)	C(13B)-C(14B)-H(14B)	119.2

C(9B)-C(14B)-H(14B)	119.2
O(4B)-C(15B)-H(15D)	109.5
O(4B)-C(15B)-H(15E)	109.5
H(15D)-C(15B)-H(15E)	109.5
O(4B)-C(15B)-H(15F)	109.5
H(15D)-C(15B)-H(15F)	109.5
H(15E)-C(15B)-H(15F)	109.5

Torsion angles [°]

O(1A)-N(1A)-C(1A)-C(2A)	-161.3(3)
C(4A)-N(1A)-C(1A)-C(2A)	-42.9(4)
O(1A)-N(1A)-C(1A)-C(5A)	74.5(5)
C(4A)-N(1A)-C(1A)-C(5A)	-167.1(4)
N(1A)-C(1A)-C(2A)-O(3A)	-88.6(5)
C(5A)-C(1A)-C(2A)-O(3A)	29.4(6)
N(1A)-C(1A)-C(2A)-C(3A)	23.1(5)
C(5A)-C(1A)-C(2A)-C(3A)	141.1(4)
C(1A)-C(2A)-O(3A)-C(6A)	127.2(5)
C(3A)-C(2A)-O(3A)-C(6A)	15.3(6)
C(6A)-O(2A)-C(3A)-C(4A)	-136.2(5)
C(6A)-O(2A)-C(3A)-C(2A)	-23.2(6)
O(3A)-C(2A)-C(3A)-O(2A)	4.6(6)
C(1A)-C(2A)-C(3A)-O(2A)	-112.0(4)
O(3A)-C(2A)-C(3A)-C(4A)	120.4(4)
C(1A)-C(2A)-C(3A)-C(4A)	3.8(5)
O(1A)-N(1A)-C(4A)-C(3A)	162.2(4)
C(1A)-N(1A)-C(4A)-C(3A	44.6(4)
O(1A)-N(1A)-C(4A)-C(9A)	38.2(5)
C(1A)-N(1A)-C(4A)-C(9A)	-79.5(5)
O(2A)-C(3A)-C(4A)-N(1A)	83.1(4)
C(2A)-C(3A)-C(4A)-N(1A)	-28.2(5)
O(2A)-C(3A)-C(4A)-C(9A)	-152.9(4)
C(2A)-C(3A)-C(4A)-C(9A)	95.8(5)
N(1A)-C(1A)-C(5A)-Br(1A)	-178.5(3)
C(2A)-C(1A)-C(5A)-Br(1A)	66.0(5)
C(3A)-O(2A)-C(6A)-O(3A)	32.8(6)
C(3A)-O(2A)-C(6A)-C(8A)	148.3(6)
C(3A)-O(2A)-C(6A)-C(7A)	-84.8(6)
C(2A)-O(3A)-C(6A)-O(2A)	-29.7(6)
C(2A)-O(3A)-C(6A)-C(8A)	-146.0(6)
C(2A)-O(3A)-C(6A)-C(7A)	88.5(6)
N(1A)-C(4A)-C(9A)-C(14A)	-89.3(6)
C(3A)-C(4A)-C(9A)-C(14A)	154.8(5)
N(1A)-C(4A)-C(9A)-C(10A)	89.9(6)
C(3A)-C(4A)-C(9A)-C(10A)	-26.0(7)
C(14A)-C(9A)-C(10A)-C(11A)	-1.6(8)
C(4A)-C(9A)-C(10A)-C(11A)	179.1(5)
C(9A)-C(10A)-C(11A)-C(12A)	1.4(9)
C(15A)-O(4A)-C(12A)-C(11A)	-1.5(8)
C(15A)-O(4A)-C(12A)-C(13A)	178.2(6)
C(10A)-C(11A)-C(12A)-O(4A)	179.1(5)
C(10A)-C(11A)-C(12A)-C(13A)	-0.5(8)
O(4A)-C(12A)-C(13A)-C(14A)	-79.6(5)
C(11A)-C(12A)-C(13A)-C(14A)	0.0(9)
C(12A)-C(13A)-C(14A)-C(9A)	-0.4(9)
C(10A)-C(9A)-C(14A)-C(13A)	1.1(8)
C(4A)-C(9A)-C(14A)-C(13A)	-179.6(5)
O(1B)-N(1B)-C(1B)-C(5B)	71.9(5)
C(4B)-N(1B)-C(1B)-C(5B)	-167.3(4)
O(1B)-N(1B)-C(1B)-C(2B)	-166.1(4)
C(4B)-N(1B)-C(1B)-C(2B)	-45.3(4)
C(6B)-O(2B)-C(2B)-C(1B)	132.0(5)
C(6B)-O(2B)-C(2B)-C(3B)	20.1(5)
N(1B)-C(1B)-C(2B)-O(2B)	-86.5(5)
C(5B)-C(1B)-C(2B)-O(2B)	32.3(6)
N(1B)-C(1B)-C(2B)-C(3B)	25.6(5)
C(5B)-C(1B)-C(2B)-C(3B)	144.4(4)
C(6B)-O(3B)-C(3B)-C(4B)	-137.1(5)
C(6B)-O(3B)-C(3B)-C(2B)	-23.7(5)
O(2B)-C(2B)-C(3B)-O(3B)	2.2(5)
C(1B)-C(2B)-C(3B)-O(3B)	-114.2(4)
O(2B)-C(2B)-C(3B)-C(4B)	117.8(4)
C(1B)-C(2B)-C(3B)-C(4B)	1.4(5)
O(1B)-N(1B)-C(4B)-C(3B)	165.7(4)
C(1B)-N(1B)-C(4B)-C(3B)	45.7(4)
O(1B)-N(1B)-C(4B)-C(9B)	43.7(5)
C(1B)-N(1B)-C(4B)-C(9B)	-76.3(5)
O(3B)-C(3B)-C(4B)-N(1B)	84.1(5)
C(2B)-C(3B)-C(4B)-N(1B)	-27.3(5)
O(3B)-C(3B)-C(4B)-C(9B)	-151.3(4)
C(2B)-C(3B)-C(4B)-C(9B)	97.4(5)
N(1B)-C(1B)-C(5B)-Br(1B)	178.8(3)
C(2B)-C(1B)-C(5B)-Br(1B)	65.1(5)
C(2B)-O(2B)-C(6B)-O(3B)	-35.0(6)
C(2B)-O(2B)-C(6B)-C(8B)	-151.2(5)
C(2B)-O(2B)-C(6B)-C(7B)	83.2(6)
C(3B)-O(3B)-C(6B)-O(2B)	36.4(6)
C(3B)-O(3B)-C(6B)-C(8B)	152.3(5)
C(3B)-O(3B)-C(6B)-C(7B)	-80.8(6)
N(1B)-C(4B)-C(9B)-C(10B)	80.9(6)
C(3B)-C(4B)-C(9B)-C(10B)	-33.9(7)
N(1B)-C(4B)-C(9B)-C(14B)	-103.0(6)
C(3B)-C(4B)-C(9B)-C(14B)	142.1(5)
C(14B)-C(9B)-C(10B)-C(11B)	0.0(9)
C(4B)-C(9B)-C(10B)-C(11B)	176.2(5)
C(9B)-C(10B)-C(11B)-C(12B)	-1.2(10)
C(15B)-O(4B)-C(12B)-C(11B)	-176.6(7)
C(15B)-O(4B)-C(12B)-C(13B)	4.0(10)
C(10B)-C(11B)-C(12B)-O(4B)	-178.3(6)
C(10B)-C(11B)-C(12B)-C(13B)	1.1(10)
O(4B)-C(12B)-C(13B)-C(14B)	179.5(6)
C(11B)-C(12B)-C(13B)-C(14B)	0.1(9)
C(12B)-C(13B)-C(14B)-C(9B)	-1.3(9)
C(10B)-C(9B)-C(14B)-C(13B)	1.3(8)
C(4B)-C(9B)-C(14B)-C(13B)	-175.1(5)

H-Bond lengths [Å] and angles [°]

O1A-N1B	2.7998 (0.55)
H1A-N1B	1.8441 (0.0818)
O1B-N1A	2.9222 (0.54)
H1B-N1A	2.2444
O1A-H1A-N1B	164.77(6.78)
O1B-H1B-N1A	140.23

9.4 (2*R*,3*R*,4*S*,5*S*)-2-Bromomethyl-5-(4-bromophenyl)-1,3,4-trihydroxy-3,4-*O*-isopropylidene-pyrrolidine (45)

$C_{14}H_{17}Br_2NO_3$

monoclinic, $P2_1$

a = 11.241(5) Å

b = 8.647(4) Å

c = 17.353(7) Å

α = 90 °

β = 105.23 (3) °

γ = 90 °

V = 1627.4 (12) Å^3

Z = 4, $R(F)$ = 0.0580

$R_w(F^2)$ = 0.1190

Crystal size: 0.45 x 0.30 x 0.08 mm

Calculated density: 1.662 g/cm^3

2θ-Range for data collection: 1.95 – 23.99 °

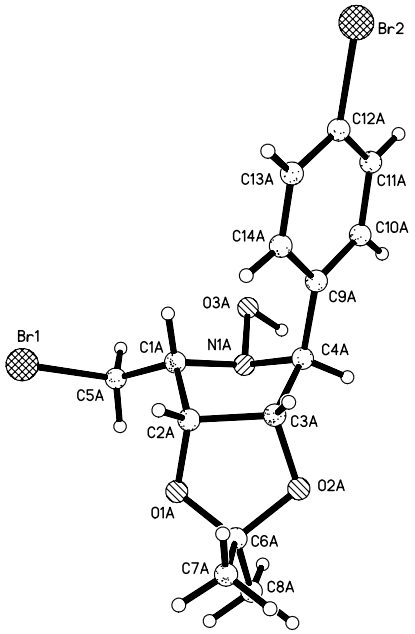
Independent reflections: 2602

Observed reflections: 1796

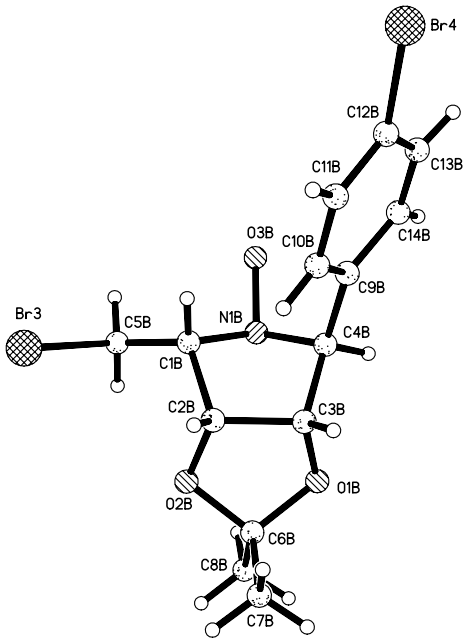
Contributed reflections to refinement: 2602

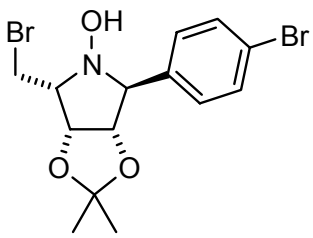
Refined parameters: 364

molecule A

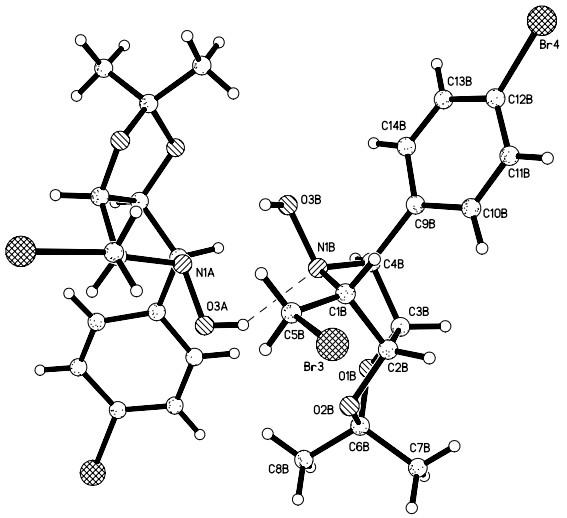


molecule B



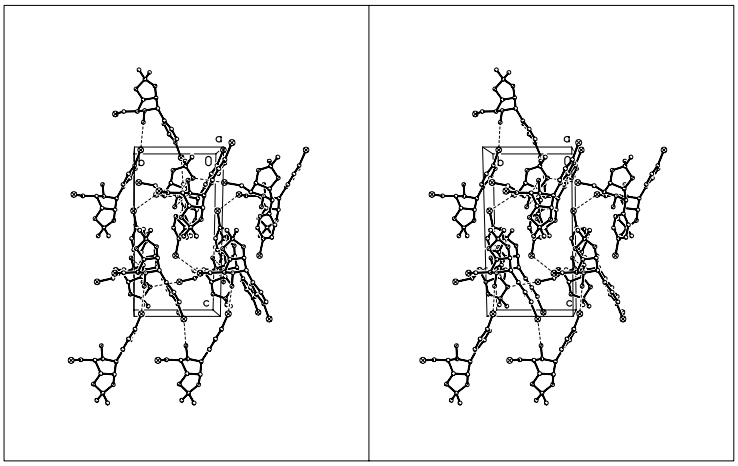


45

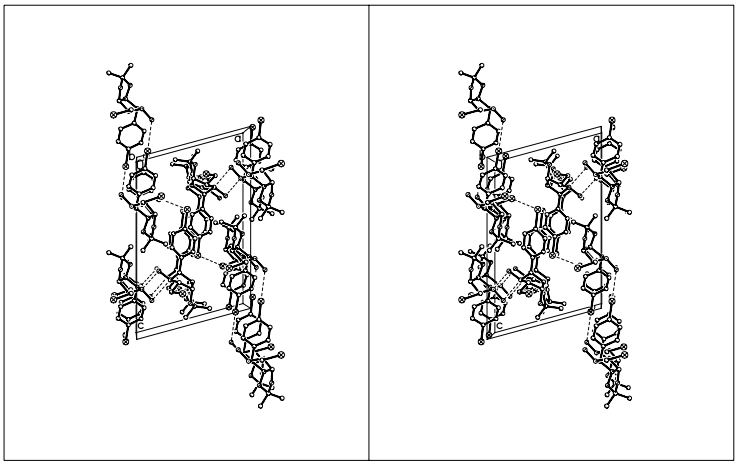


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

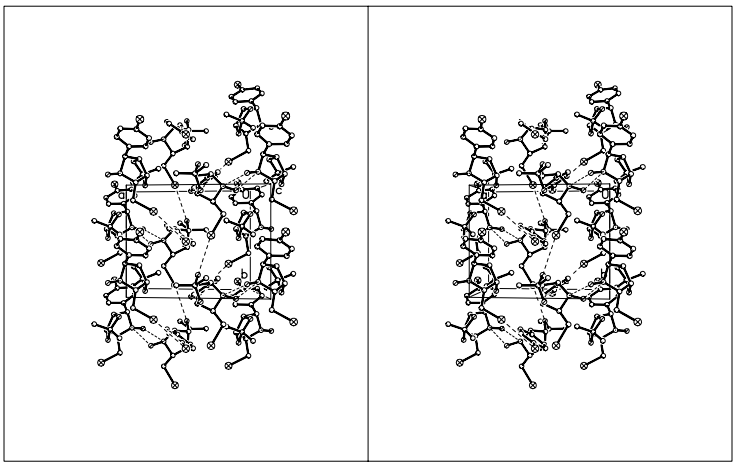
(a)



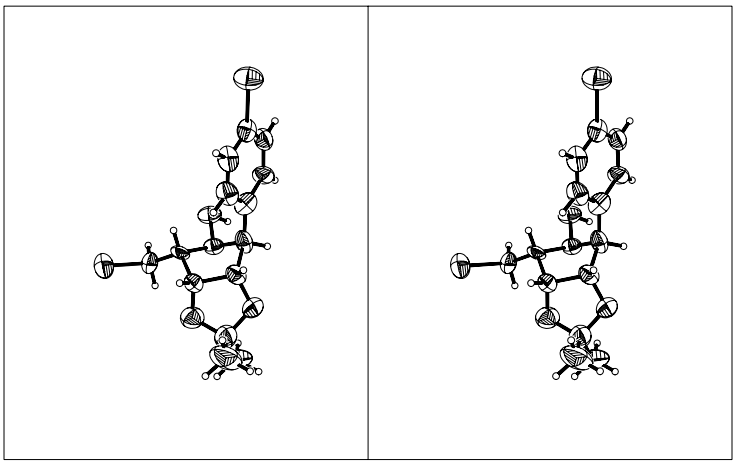
(b)



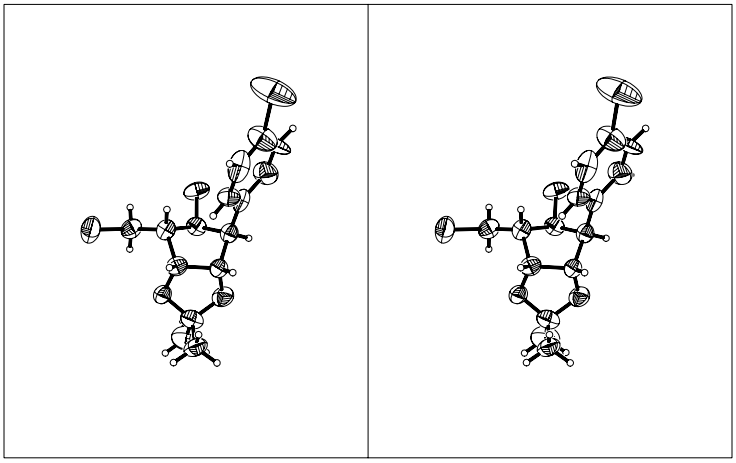
(c)



ORTEP Structure, molecule A



ORTEP Structure, molecule B



Bond lengths [Å] and angles [°]

Br(1)-C(5A)	1.921(12)	C(5B)-H(5B2)	0.97
Br(2)-C(12A)	1.898(13)	C(6B)-C(8B)	1.50(2)
N(1A)-O(3A)	1.425(12)	C(6B)-C(7B)	1.507(18)
.N(1A)-C(1A)	1.469(16)	C(7B)-H(7B1)	0.96
N(1A)-C(4A)	1.493(16)	C(7B)-H(7B2)	0.96
O(1A)-C(6A)	1.40(2)	C(7B)-H(7B3)	0.96
O(1A)-C(2A)	1.412(16)	C(8B)-H(8B1)	0.96
C(1A)-C(5A)	1.520(17)	C(8B)-H(8B2)	0.96
C(1A)-C(2A)	1.540(18)	C(8B)-H(8B3)	0.96
C(1A)-H(1A)	0.98	C(9B)-C(14B)	1.41(2)
C(2A)-C(3A)	1.547(18)	C(9B)-C(10B)	1.432(17)
C(2A)-H(2A)	0.98	C(10B)-C(11B)	1.33(2)
O(2A)-C(3A)	1.434(17)	C(10B)-H(10B)	0.93
O(2A)-C(6A)	1.44(2)	C(11B)-C(12B)	1.37(2)
O(3A)-H(3AA)	0.82	C(11B)-H(11B)	0.93
C(3A)-C(4A)	1.528(17)	C(12B)-C(13B)	1.39(2)
C(3A)-H(3A)	0.98	C(13B)-C(14B)	1.29(2)
C(4A)-C(9A)	1.5(19)	C(13B)-H(13B)	0.93
C(4A)-H(4A)	0.98	C(14B)-H(14B)	0.93
C(5A)-H(5A1)	0.97	O(3A)-N(1A)-C(1A)	109.3(9)
C(5A)-H(5A2)	0.97	O(3A)-N(1A)-C(4A)	110.2(9)
C(6A)-C(7A)	1.47(2)	C(1A)-N(1A)-C(4A)	105.9(9)
C(6A)-C(8A)	0.96	C(6A)-O(1A)-C(2A)	108.7(11)
C(7A)-H(7A2)	0.96	N(1A)-C(1A)-C(5A)	110.8(10)
C(7A)-H(7A3)	0.96	N(1A)-C(1A)-C(2A)	100.0(10)
C(8A)-H(8A1)	0.96	C(5A)-C(1A)-C(2A)	116.9(10)
C(8A)-H(8A2)	0.96	N(1A)-C(1A)-H(1A)	109.6
C(8A)-H(8A3)	0.96	C(5A)-C(1A)-H(1A)	109.6
C(9A)-C(10A)	1.390(18)	C(2A)-C(1A)-H(1A)	109.6
C(9A)-C(14A)	1.43(2)	O(1A)-C(2A)-C(1A)	110.6(11)
C(10A)-C(11A)	1.375(19)	O(1A)-C(2A)-C(3A)	105.2(10)
C(10A)-H(10A)	0.93	C(1A)-C(2A)-C(3A)	104.5(10)
C(11A)-C(12A)	1.369(19)	O(1A)-C(2A)-H(2A)	112.0
C(11A)-H(11A)	0.93	C(1A)-C(2A)-H(2A)	112.0
C(12A)-C(13A)	1.394(17)	C(3A)-C(2A)-H(2A)	112.0
C(13A)-C(14A)	1.303(19)	C(3A)-O(2A)-C(6A)	109.0(12)
C(13A)-H(13A)	0.93	N(1A)-O(3A)-H(3AA)	109.5
C(14A)-H(14A)	0.93	O(2A)-C(3A)-C(4A)	110.5(11)
Br(3)-C(5B)	1.954(14)	O(2A)-C(3A)-C(2A)	102.6(10)
Br(4)-C(12B)	1.842(16)	C(4A)-C(3A)-C(2A)	106.3(11)
O(1B)-C(3B)	1.377(15)	O(2A)-C(3A)-H(3A)	112.3
O(1B)-C(6B)	1.419(18)	C(4A)-C(3A)-H(3A)	112.3
N(1B)-C(4B)	1.438(17)	C(2A)-C(3A)-H(3A)	112.3
N(1B)-O(3B)	1.452(12)	N(1A)-C(4A)-C(9A)	114.2(12)
N(1B)-C(1B)	1.494(15)	N(1A)-C(4A)-C(3A)	99.7(10)
C(1B)-C(5B)	1.458(19)	C(9A)-C(4A)-C(3A)	116.6(11)
C(1B)-C(2B)	1.537(17)	N(1A)-C(4A)-H(4A)	108.6
C(1B)-H(1B)	0.98	C(9A)-C(4A)-H(4A)	108.6
O(2B)-C(2B)	1.401(15)	C(3A)-C(4A)-H(4A)	108.6
O(2B)-C(6B)	1.437(17)	C(1A)-C(5A)-Br(1)	110.3(9)
C(2B)-C(3B)	1.526(19)	C(1A)-C(5A)-H(5A1)	109.6
C(2B)-H(2B)	0.98	Br(1)-C(5A)-H(5A1)	109.6
O(3B)-H(3BB)	0.82	C(1A)-C(5A)-H(5A2)	109.6
C(3B)-C(4B)	1.504(17)	Br(1)-C(5A)-H(5A2)	109.6
C(3B)-H(3B)	0.98	H(5A1)-C(5A)-H(5A2)	108.1
C(4B)-C(9B)	1.559(18)	O(1A)-C(6A)-O(2A)	103.5(13)
C(4B)-H(4B)	0.98	O(1A)-C(6A)-C(7A)	111.7(16)
C(5B)-H(5B1)	0.97	O(2A)-C(6A)-C(7A)	108.3(15)

O(1A)-C(6A)-C(8A)	111.6(15)
O(2A)-C(6A)-C(8A)	105.9(15)
C(7A)-C(6A)-C(8A)	114.9(17)
C(6A)-C(7A)-H(7A1)	109.5
C(6A)-C(7A)-H(7A2)	109.5
H(7A1)-C(7A)-H(7A2)	109.5
C(6A)-C(7A)-H(7A3)	109.5
H(7A1)-C(7A)-H(7A3)	109.5
H(7A2)-C(7A)-H(7A3)	109.5
C(6A)-C(8A)-H(8A1)	109.5
C(6A)-C(8A)-H(8A2)	109.5
H(8A1)-C(8A)-H(8A2)	109.5
C(6A)-C(8A)-H(8A3)	109.5
H(8A1)-C(8A)-H(8A3)	109.5
H(8A2)-C(8A)-H(8A3)	109.5
C(10A)-C(9A)-C(14A)	114.7(13)
C(10A)-C(9A)-C(4A)	121.1(12)
C(14A)-C(9A)-C(4A)	124.0(13)
C(11A)-C(10A)-C(9A)	121.7(13)
C(11A)-C(10A)-H(10A)	119.2
C(9A)-C(10A)-H(10A)	119.2
C(12A)-C(11A)-C(10A)	119.6(13)
C(12A)-C(11A)-H(11A)	120.2
C(10A)-C(11A)-H(11A)	120.2
C(11A)-C(12A)-C(13A)	120.8(12)
C(11A)-C(12A)-Br(2)	119.8(11)
C(13A)-C(12A)-Br(2)	119.3(9)
C(14A)-C(13A)-C(12A)	118.3(12)
C(14A)-C(13A)-H(13A)	120.9
C(12A)-C(13A)-H(13A)	120.9
C(13A)-C(14A)-C(9A)	124.8(13)
C(13A)-C(14A)-H(14A)	117.6
C(9A)-C(14A)-H(14A)	117.6
C(3B)-O(1B)-C(6B)	107.5(10)
C(4B)-N(1B)-O(3B)	110.5(10)
C(4B)-N(1B)-C(1B)	105.2(9)
O(3B)-N(1B)-C(1B)	110.5(9)
C(5B)-C(1B)-N(1B)	110.9(10)
C(5B)-C(1B)-C(2B)	117.6(12)
N(1B)-C(1B)-C(2B)	99.1(10)
C(5B)-C(1B)-H(1B)	109.6
N(1B)-C(1B)-H(1B)	109.6
C(2B)-C(1B)-H(1B)	109.6
C(2B)-O(2B)-C(6B)	106.0(10)
O(2B)-C(2B)-C(3B)	105.3(10)
O(2B)-C(2B)-C(1B)	108.7(11)
C(3B)-C(2B)-C(1B)	104.8(11)
O(2B)-C(2B)-H(2B)	112.5
C(3B)-C(2B)-H(2B)	112.5
C(1B)-C(2B)-H(2B)	112.5
N(1B)-O(3B)-H(3BB)	109.5
O(1B)-C(3B)-C(4B)	110.2(10)
O(1B)-C(3B)-C(2B)	104.7(11)
C(4B)-C(3B)-C(2B)	105.7(11)
O(1B)-C(3B)-H(3B)	111.9
C(4B)-C(3B)-H(3B)	111.9
C(2B)-C(3B)-H(3B)	111.9
N(1B)-C(4B)-C(3B)	100.0(10)
N(1B)-C(4B)-C(9B)	114.0(10)
C(3B)-C(4B)-C(9B)	115.7(10)
N(1B)-C(4B)-H(4B)	108.9
C(3B)-C(4B)-H(4B)	108.9
C(9B)-C(4B)-H(4B)	108.9
C(1B)-C(5B)-Br(3)	108.7(9)
C(1B)-C(5B)-H(5B1)	109.9
Br(3)-C(5B)-H(5B1)	109.9
C(1B)-C(5B)-H(5B2)	109.9
Br(3)-C(5B)-H(5B2)	109.9
H(5B1)-C(5B)-H(5B2)	108.3
O(1B)-C(6B)-O(2B)	103.7(9)
O(1B)-C(6B)-C(8B)	110.7(13)
O(2B)-C(6B)-C(8B)	109.9(14)
O(1B)-C(6B)-C(7B)	111.3(13)
O(2B)-C(6B)-C(7B)	110.7(11)
C(8B)-C(6B)-C(7B)	110.4(12)
C(6B)-C(7B)-H(7B1)	109.5
C(6B)-C(7B)-H(7B2)	109.5
H(7B1)-C(7B)-H(7B2)	109.5
C(6B)-C(7B)-H(7B3)	109.5
H(7B1)-C(7B)-H(7B3)	109.5
H(7B2)-C(7B)-H(7B3)	109.5
C(6B)-C(8B)-H(8B1)	109.5
C(6B)-C(8B)-H(8B2)	109.5
H(8B1)-C(8B)-H(8B2)	109.5
C(6B)-C(8B)-H(8B3)	109.5
H(8B1)-C(8B)-H(8B3)	109.5
H(8B2)-C(8B)-H(8B3)	109.5
C(14B)-C(9B)-C(10B)	115.9(13)
C(14B)-C(9B)-C(4B)	122.1(11)
C(10B)-C(9B)-C(4B)	122.0(12)
C(11B)-C(10B)-C(9B)	121.3(14)
C(11B)-C(10B)-H(10B)	119.3
C(9B)-C(10B)-H(10B)	119.3
C(10B)-C(11B)-C(12B)	120.7(14)
C(10B)-C(11B)-H(11B)	119.6
C(12B)-C(11B)-H(11B)	119.6
C(11B)-C(12B)-C(13B)	117.7(14)
C(11B)-C(12B)-Br(4)	121.8(12)
C(13B)-C(12B)-Br(4)	120.4(12)
C(14B)-C(13B)-C(12B)	123.5(16)
C(14B)-C(13B)-H(13B)	118.3
C(12B)-C(13B)-H(13B)	118.3
C(13B)-C(14B)-C(9B)	120.7(15)
C(13B)-C(14B)-H(14B)	119.6
C(9B)-C(14B)-H(14B)	119.6

Torsion angles [°]

O(3A)-N(1A)-C(1A)-C(5A)	69.2(12)
C(4A)-N(1A)-C(1A)-C(5A)	-172.1(10)
O(3A)-N(1A)-C(1A)-C(2A)	-166.9(9)
C(4A)-N(1A)-C(1A)-C(2A)	-48.2(12)
C(6A)-O(1A)-C(2A)-C(1A)	134.3(11)
C(6A)-O(1A)-C(2A)-C(3A)	22.0(14)
N(1A)-C(1A)-C(2A)-O(1A)	-83.2(11)
C(5A)-C(1A)-C(2A)-O(1A)	36.4(15)
N(1A)-C(1A)-C(2A)-C(3A)	29.6(12)
C(5A)-C(1A)-C(2A)-C(3A)	149.2(11)
C(6A)-O(2A)-C(3A)-C(4A)	-131.1(12)

C(6A)-O(2A)-C(3A)-C(2A)	-18.1(14)
O(1A)-C(2A)-C(3A)-O(2A)	-2.0(13)
C(1A)-C(2A)-C(3A)-O(2A)	-118.6(11)
O(1A)-C(2A)-C(3A)-C(4A)	114.0(11)
C(1A)-C(2A)-C(3A)-C(4A)	-2.5(13)
O(3A)-N(1A)-C(4A)-C(9A)	39.4(13)
C(1A)-N(1A)-C(4A)-C(9A)	-78.7(12)
O(3A)-N(1A)-C(4A)-C(3A)	164.5(9)
C(1A)-N(1A)-C(4A)-C(3A)	46.4(12)
O(2A)-C(3A)-C(4A)-N(1A)	85.5(12)
C(2A)-C(3A)-C(4A)-N(1A)	-25.1(13)
O(2A)-C(3A)-C(4A)-C(9A)	-151.0(12)
C(2A)-C(3A)-C(4A)-C(9A)	98.4(13)
N(1A)-C(1A)-C(5A)-Br(1)	-173.5(7)
C(2A)-C(1A)-C(5A)-Br(1)	72.9(13)
C(2A)-O(1A)-C(6A)-O(2A)	-33.1(15)
C(2A)-O(1A)-C(6A)-C(7A)	83.3(16)
C(2A)-O(1A)-C(6A)-C(8A)	-146.6(14)
C(3A)-O(2A)-C(6A)-O(1A)	31.8(16)
C(3A)-O(2A)-C(6A)-C(7A)	-86.9(17)
C(3A)-O(2A)-C(6A)-C(8A)	149.3(13)
N(1A)-C(4A)-C(9A)-C(10A)	-84.5(16)
C(3A)-C(4A)-C(9A)-C(10A)	159.9(13)
N(1A)-C(4A)-C(9A)-C(14A)	92.0(16)
C(3A)-C(4A)-C(9A)-C(14A)	-24(2)
C(14A)-C(9A)-C(10A)-C(11A)	0(2)
C(4A)-C(9A)-C(10A)-C(11A)	176.8(13)
C(9A)-C(10A)-C(11A)-C(12A)	2(2)
C(10A)-C(11A)-C(12A)-C(13A)	-1(2)
C(10A)-C(11A)-C(12A)-Br(2)	177.7(11)
C(11A)-C(12A)-C(13A)-C(14A)	-1(2)
Br(2)-C(12A)-C(13A)-C(14A)	179.8(11)
C(12A)-C(13A)-C(14A)-C(9A)	3(2)
C(10A)-C(9A)-C(14A)-C(13A)	-3(2)
C(4A)-C(9A)-C(14A)-C(13A)	-179.4(14)
C(4B)-N(1B)-C(1B)-C(5B)	-171.6(11)
O(3B)-N(1B)-C(1B)-C(5B)	69.2(14)
C(4B)-N(1B)-C(1B)-C(2B)	-47.3(11)
O(3B)-N(1B)-C(1B)-C(2B)	-166.5(9)
C(6B)-O(2B)-C(2B)-C(3B)	19.8(13)
C(6B)-O(2B)-C(2B)-C(1B)	131.7(10)
C(5B)-C(1B)-C(2B)-O(2B)	32.1(15)
N(1B)-C(1B)-C(2B)-O(2B)	-87.4(11)
C(5B)-C(1B)-C(2B)-C(3B)	144.3(11)
N(1B)-C(1B)-C(2B)-C(3B)	24.8(12)
C(6B)-O(1B)-C(3B)-C(4B)	-136.2(12)
C(6B)-O(1B)-C(3B)-C(2B)	-23.0(13)
O(2B)-C(2B)-C(3B)-O(1B)	1.7(13)
C(1B)-C(2B)-C(3B)-O(1B)	-112.9(11)
O(2B)-C(2B)-C(3B)-C(4B)	118.1(11)
C(1B)-C(2B)-C(3B)-C(4B)	3.5(13)
O(3B)-N(1B)-C(4B)-C(3B)	169.2(9)
C(1B)-N(1B)-C(4B)-C(3B)	49.9(11)
O(3B)-N(1B)-C(4B)-C(9B)	45.2(12)
C(1B)-N(1B)-C(4B)-C(9B)	-74.1(11)
O(1B)-C(3B)-C(4B)-N(1B)	80.9(12)
C(2B)-C(3B)-C(4B)-N(1B)	-31.7(12)
O(1B)-C(3B)-C(4B)-C(9B)	-156.2(11)
C(2B)-C(3B)-C(4B)-C(9B)	91.2(13)
N(1B)-C(1B)-C(5B)-Br(3)	-176.0(8)
C(2B)-C(1B)-C(5B)-Br(3)	71.0(13)
C(3B)-O(1B)-C(6B)-O(2B)	35.6(13)
C(3B)-O(1B)-C(6B)-C(8B)	153.4(13)
C(3B)-O(1B)-C(6B)-C(7B)	-83.4(13)
C(2B)-O(2B)-C(6B)-O(1B)	-33.9(12)
C(2B)-O(2B)-C(6B)-C(8B)	-152.2(11)
C(2B)-O(2B)-C(6B)-C(7B)	85.6(13)
N(1B)-C(4B)-C(9B)-C(14B)	-92.3(15)
C(3B)-C(4B)-C(9B)-C(14B)	152.5(13)
N(1B)-C(4B)-C(9B)-C(10B)	89.2(14)
C(3B)-C(4B)-C(9B)-C(10B)	-25.9(17)
C(14B)-C(9B)-C(10B)-C(11B)	4(2)
C(4B)-C(9B)-C(10B)-C(11B)	-177.6(13)
C(9B)-C(10B)-C(11B)-C(12B)	-2(2)
C(10B)-C(11B)-C(12B)-C(13B)	-3(3)
C(10B)-C(11B)-C(12B)-Br(4)	172.8(13)
C(11B)-C(12B)-C(13B)-C(14B)	5(3)
Br(4)-C(12B)-C(13B)-C(14B)	-170.3(17)
C(12B)-C(13B)-C(14B)-C(9B)	-3(3)
C(10B)-C(9B)-C(14B)-C(13B)	-2(2)
C(4B)-C(9B)-C(14B)-C(13B)	180.0(16)

H-Bond lengths [Å] and angles [°]

O3B-Br4_$1	3.0566(0.0101)
H3BB-Br4_$1	2.8350
O3A-N1B	2.7829(0.0125)
H3AA-N1B	2.0970
O3B-H3BB-Br4_$1	97.82
O3A-H3AA-N1B	141.07

$ denots atoms of neigbouring molecule

9.5 (2*R*,3*R*,4*S*,5*S*)-2-Bromomethyl-1,3,4-trihydroxy-3,4-*O*-isopropylidene-5-(4-phenoxyphenyl)-pyrrolidine (47)

$C_{20}H_{22}BrNO_4$

monoclinic, $P2_1$

a = 11.669(3) Å

b = 8.5977(19) Å

c = 20.143(3) Å

α = 90 °

β = 95.574(14) °

γ = 90 °

V = 2011.3(7) Å^3

Z = 4, $R(F)$ = 0.0724

$R_w(F^2)$ = 0.1667

Crystal size:

1.8 x 0.6 x 0.25 mm

Calculated density: 1.388 g/cm^3

2θ-Range for data collection: 1.75 – 27.50 °

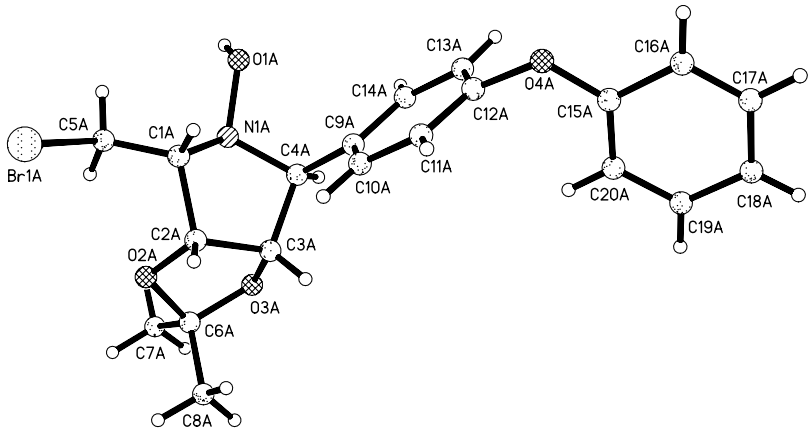
Independent reflections: 5536

Observed reflections: 3083

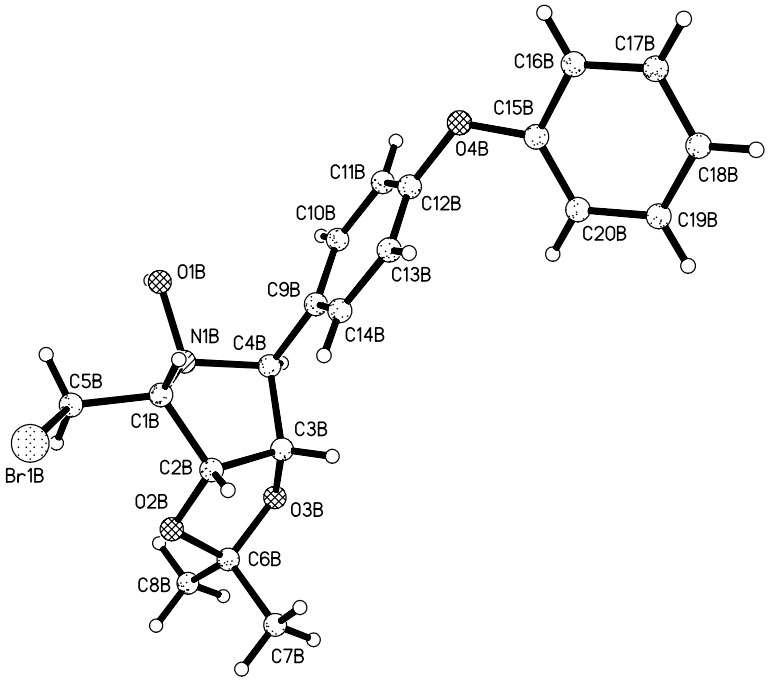
Contributed reflections to refinement: 5536

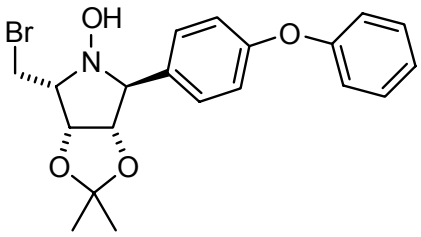
Refined parameters: 485

molecule A

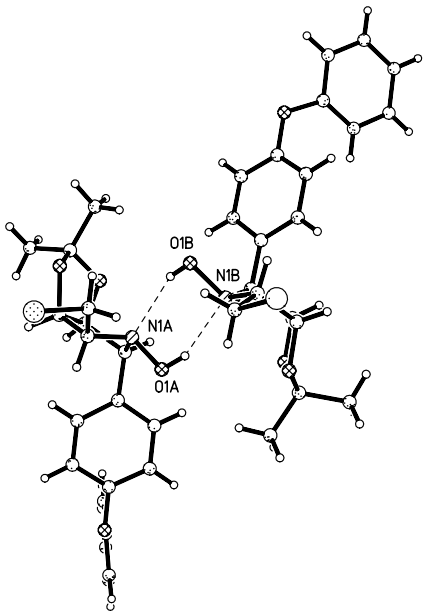


molecule B



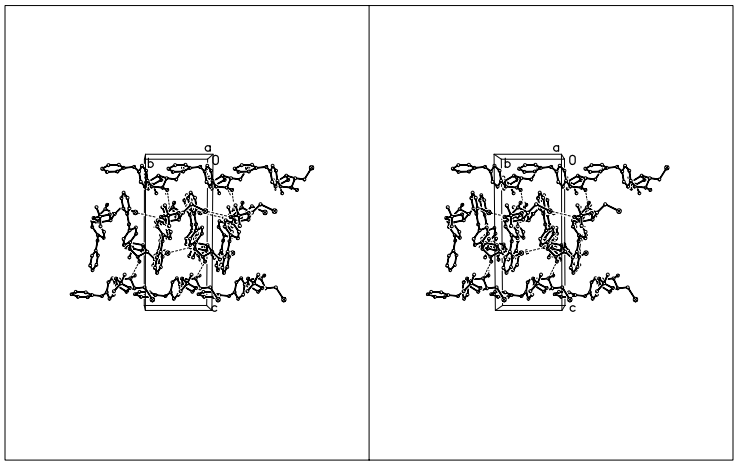


47

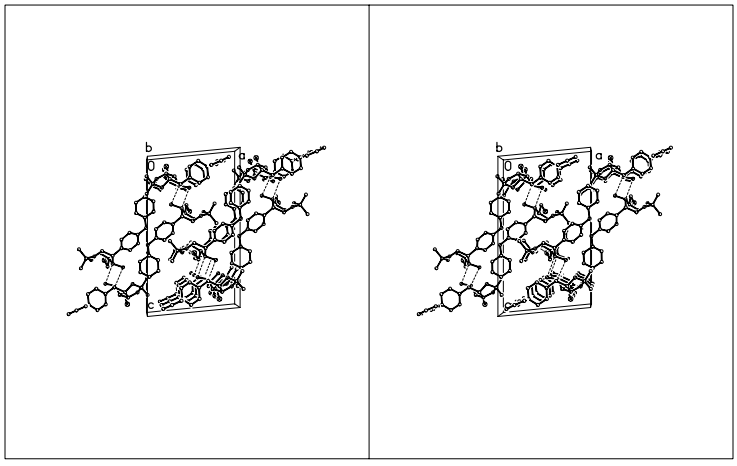


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

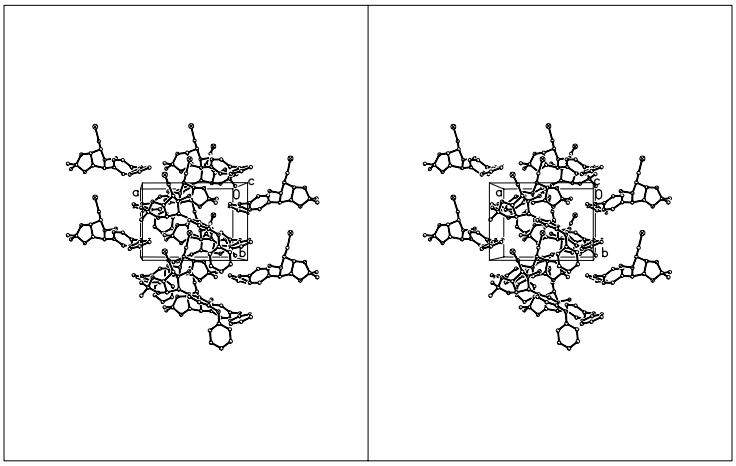
(a)



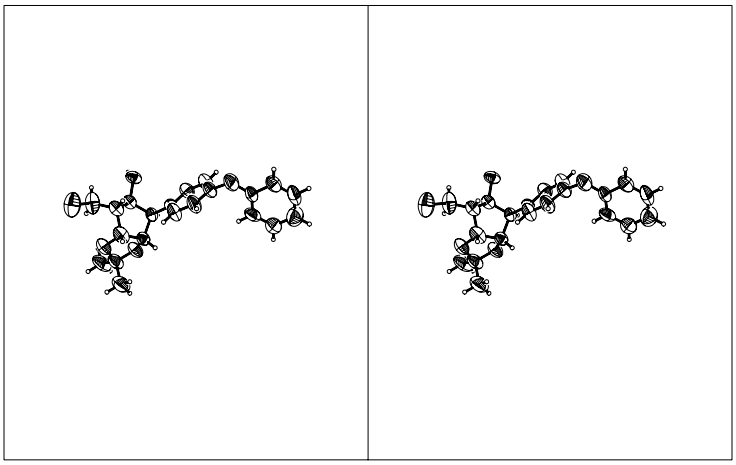
b)



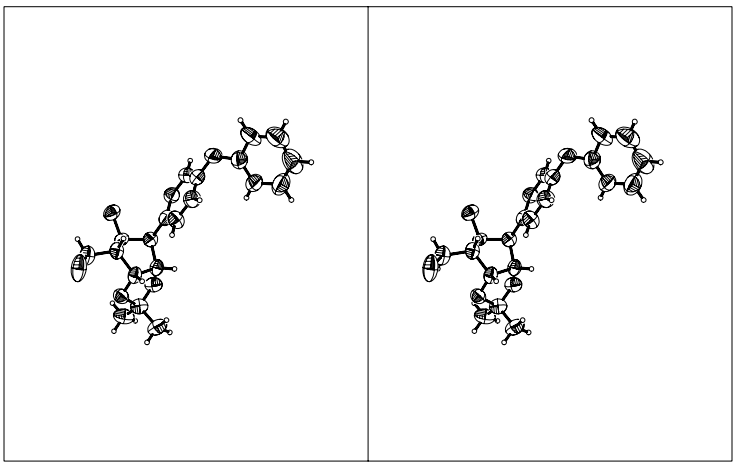
c)



ORTEP Structure, molecule A



ORTEP Structure, molecule B



Bond lengths [Å] and angles [°]

Br(1A)-C(5A)	1.915(10)	C(1B)-H(1B)	0.98
Br(1C)-C(5A)	1.761(8)	N(1B)-C(4B)	1.498(10)
O(1A)-N(1A)	1.452(9)	O(2B)-C(6B)	1.436(11)
O(1A)-H(1AA)	0.82	O(2B)-C(2B)	1.448(10)
C(1A)-C(5A)	1.416(15)	C(2B)-C(3B)	1.545(13)
C(1A)-N(1A)	1.468(10)	C(2B)-H(2B)	0.98
C(1A)-C(2A)	1.553(16)	O(3B)-C(6B)	1.407(11)
C(1A)-H(1A)	0.98	O(3B)-C(3B)	1.418(10)
N(1A)-C(4A)	1.480(12)	C(3B)-C(4B)	1.538(11)
O(2A)-C(6A)	1.421(15)	C(3B)-H(3B)	0.98
O(2A)-C(2A)	1.450(11)	O(4B)-C(15B)	1.386(11)
C(2A)-C(3A)	1.526(14)	O(4B)-C(12B)	1.401(11)
C(2A)-H(2A)	0.98	C(4B)-C(9B)	1.529(11)
O(3A)-C(6A)	1.395(13)	C(4B)-H(4B)	0.98
O(3A)-C(3A)	1.441(9)	C(5B)-H(5B1)	0.97
C(3A)-C(4A)	1.540(12)	C(5B)-H(5B2)	0.97
C(3A)-H(3A)	0.98	C(6B)-C(8B)	1.525(13)
O(4A)-C(15A)	1.357(14)	C(6B)-C(7B)	1.531(12)
O(4A)-C(12A)	1.424(11)	C(7B)-H(7B1)	0.96
C(4A)-C(9A)	1.507(11)	C(7B)-H(7B2)	0.96
C(4A)-H(4A)	0.98	C(7B)-H(7B3)	0.96
C(5A)-H(5A1)	0.97	C(8B)-H(8B1)	0.96
C(5A)-H(5A2)	0.97	C(8B)-H(8B2)	0.96
C(6A)-C(8A)	1.507(18)	C(8B)-H(8B3)	0.96
C(6A)-C(7A)	1.529(15)	C(9B)-C(14B)	1.382(12)
C(7A)-H(7A1)	0.96	C(9B)-C(10B)	1.391(12)
C(7A)-H(7A2)	0.96	C(10B)-C(11B)	1.364(12)
C(7A)-H(7A3)	0.96	C(10B)-H(10B)	0.93
C(8A)-H(8A1)	0.96	C(11B)-C(12B)	1.360(15)
C(8A)-H(8A2)	0.96	C(11B)-H(11B)	0.93
C(8A)-H(8A3)	0.96	C(12B)-C(13B)	1.361(16)
C(9A)-C(10A)	1.381(11)	C(13B)-C(14B)	1.395(12)
C(9A)-C(14A)	1.403(12)	C(13B)-H(13B)	0.93
C(10A)-C(11A)	1.393(12)	C(14B)-H(14B)	0.93
C(10A)-H(10A)	0.93	C(15B)-C(20B)	1.359(14)
C(11A)-C(12A)	1.365(14)	C(15B)-C(16B)	1.413(14)
C(11A)-H(11A)	0.93	C(16B)-C(17B)	1.364(16)
C(12A)-C(13A)	1.374(14)	C(16B)-H(16B)	0.93
C(13A)-C(14A)	1.370(13)	C(17B)-C(18B)	1.37(2)
C(13A)-H(13A)	0.93	C(17B)-H(17B)	0.93
C(14A)-H(14A)	0.93	C(18B)-C(19B)	1.4(18)
C(15A)-C(20A)	1.366(15)	C(18B)-H(18B)	0.93
C(15A)-C(16A)	1.416(13)	C(19B)-C(20B)	1.372(14)
C(16A)-C(17A)	1.338(19)	C(19B)-H(19B)	0.93
C(16A)-H(16A)	0.93	C(20B)-H(20B)	0.93
C(17A)-C(18A)	1.39(2)	N(1A)-O(1A)-H(1AA)	109.5
C(17A)-H(17A)	0.93	C(5A)-C(1A)-N(1A)	111.9(8)
C(18A)-C(19A)	1.379(17)	C(5A)-C(1A)-C(2A)	113.4(8)
C(18A)-H(18A)	0.93	N(1A)-C(1A)-C(2A)	99.9(8)
C(19A)-C(20A)	1.367(18)	C(5A)-C(1A)-H(1A)	110.4
C(19A)-H(19A)	0.93	N(1A)-C(1A)-H(1A)	110.4
C(20A)-H(20A)	0.93	C(2A)-C(1A)-H(1A)	110.4
Br(1B)-C(5B)	1.938(9)	O(1A)-N(1A)-C(1A)	109.2(7)
O(1B)-N(1B)	1.454(8)	O(1A)-N(1A)-C(4A)	110.4(7)
O(1B)-H(1BB)	0.76(12)	C(1A)-N(1A)-C(4A)	108.6(7)
C(1B)-N(1B)	1.474(10)	C(6A)-O(2A)-C(2A)	106.0(9)
C(1B)-C(5B)	1.502(12)	O(2A)-C(2A)-C(3A)	104.0(8)
C(1B)-C(2B)	1.525(12)	O(2A)-C(2A)-C(1A)	108.7(9)

C(3A)-C(2A)-C(1A)	106.9(7)	C(12A)-C(13A)-H(13A)	120.4
O(2A)-C(2A)-H(2A)	112.3	C(13A)-C(14A)-C(9A)	121.0(9)
C(3A)-C(2A)-H(2A)	112.3	C(13A)-C(14A)-H(14A)	119.5
C(1A)-C(2A)-H(2A)	112.3	C(9A)-C(14A)-H(14A)	119.5
C(6A)-O(3A)-C(3A)	105.7(7)	O(4A)-C(15A)-C(20A)	126.7(10)
O(3A)-C(3A)-C(2A)	104.1(7)	O(4A)-C(15A)-C(16A)	115.9(10)
O(3A)-C(3A)-C(4A)	108.7(6)	C(20A)-C(15A)-C(16A)	117.5(11)
C(2A)-C(3A)-C(4A)	105.6(8)	C(17A)-C(16A)-C(15A)	119.1(13)
O(3A)-C(3A)-H(3A)	112.6	C(17A)-C(16A)-H(16A)	120.5
C(2A)-C(3A)-H(3A)	112.6	C(15A)-C(16A)-H(16A)	120.5
C(4A)-C(3A)-H(3A)	112.6	C(16A)-C(17A)-C(18A)	123.4(13)
C(15A)-O(4A)-C(12A)	116.6(9)	C(16A)-C(17A)-H(17A)	118.3
N(1A)-C(4A)-C(9A)	117.9(7)	C(18A)-C(17A)-H(17A)	118.3
N(1A)-C(4A)-C(3A)	99.9(7)	C(19A)-C(18A)-C(17A)	117.2(16)
C(9A)-C(4A)-C(3A)	113.1(6)	C(19A)-C(18A)-H(18A)	121.4
N(1A)-C(4A)-H(4A)	108.5	C(17A)-C(18A)-H(18A)	121.4
C(9A)-C(4A)-H(4A)	108.5	C(20A)-C(19A)-C(18A)	119.8(14)
C(3A)-C(4A)-H(4A)	108.5	C(20A)-C(19A)-H(19A)	120.1
C(1A)-C(5A)-Br(1C)	120.2(9)	C(18A)-C(19A)-H(19A)	120.1
C(1A)-C(5A)-Br(1A)	117.4(7)	C(15A)-C(20A)-C(19A)	123.0(12)
Br(1C)-C(5A)-Br(1A)	58.3(5)	C(15A)-C(20A)-H(20A)	118.5
C(1A)-C(5A)-H(5A1)	107.9	C(19A)-C(20A)-H(20A)	118.5
Br(1C)-C(5A)-H(5A1)	131.0	N(1B)-O(1B)-H(1BB)	99.0(9)
Br(1A)-C(5A)-H(5A1)	107.9	N(1B)-C(1B)-C(5B)	110.0(6)
C(1A)-C(5A)-H(5A2)	107.9	N(1B)-C(1B)-C(2B)	98.9(6)
Br(1C)-C(5A)-H(5A2)	51.0	C(5B)-C(1B)-C(2B)	117.7(8)
Br(1A)-C(5A)-H(5A2)	107.9	N(1B)-C(1B)-H(1B)	109.9
H(5A1)-C(5A)-H(5A2)	107.2	C(5B)-C(1B)-H(1B)	109.9
O(3A)-C(6A)-O(2A)	104.7(8)	C(2B)-C(1B)-H(1B)	109.9
O(3A)-C(6A)-C(8A)	111.4(11)	O(1B)-N(1B)-C(1B)	107.7(6)
O(2A)-C(6A)-C(8A)	111.5(11)	O(1B)-N(1B)-C(4B)	109.7(6)
O(3A)-C(6A)-C(7A)	106.7(9)	C(1B)-N(1B)-C(4B)	107.6(5)
O(2A)-C(6A)-C(7A)	107.3(11)	C(6B)-O(2B)-C(2B)	106.7(6)
C(8A)-C(6A)-C(7A)	114.7(10)	O(2B)-C(2B)-C(1B)	110.4(6)
C(6A)-C(7A)-H(7A1)	109.5	O(2B)-C(2B)-C(3B)	102.8(7)
C(6A)-C(7A)-H(7A2)	109.5	C(1B)-C(2B)-C(3B)	106.7(7)
H(7A1)-C(7A)-H(7A2)	109.5	O(2B)-C(2B)-H(2B)	112.2
C(6A)-C(7A)-H(7A3)	109.5	C(1B)-C(2B)-H(2B)	112.2
H(7A1)-C(7A)-H(7A3)	109.5	C(3B)-C(2B)-H(2B)	112.2
H(7A2)-C(7A)-H(7A3)	109.5	C(6B)-O(3B)-C(3B)	107.2(7)
C(6A)-C(8A)-H(8A1)	109.5	O(3B)-C(3B)-C(4B)	109.8(6)
C(6A)-C(8A)-H(8A2)	109.5	O(3B)-C(3B)-C(2B)	105.9(7)
H(8A1)-C(8A)-H(8A2)	109.5	C(4B)-C(3B)-C(2B)	105.7(7)
C(6A)-C(8A)-H(8A3)	109.5	O(3B)-C(3B)-H(3B)	111.7
H(8A1)-C(8A)-H(8A3)	109.5	C(4B)-C(3B)-H(3B)	111.7
H(8A2)-C(8A)-H(8A3)	109.5	C(2B)-C(3B)-H(3B)	111.7
C(10A)-C(9A)-C(14A)	117.7(8)	C(15B)-O(4B)-C(12B)	121.6(8)
C(10A)-C(9A)-C(4A)	124.1(7)	N(1B)-C(4B)-C(9B)	115.6(7)
C(14A)-C(9A)-C(4A)	118.2(7)	N(1B)-C(4B)-C(3B)	98.8(6)
C(9A)-C(10A)-C(11A)	121.9(9)	C(9B)-C(4B)-C(3B)	114.9(6)
C(9A)-C(10A)-H(10A)	119.0	N(1B)-C(4B)-H(4B)	109.0
C(11A)-C(10A)-H(10A)	119.0	C(9B)-C(4B)-H(4B)	109.0
C(12A)-C(11A)-C(10A)	117.9(9)	C(3B)-C(4B)-H(4B)	109.0
C(12A)-C(11A)-H(11A)	121.0	C(1B)-C(5B)-Br(1B)	110.7(5)
C(10A)-C(11A)-H(11A)	121.0	C(1B)-C(5B)-H(5B1)	109.5
C(11A)-C(12A)-C(13A)	122.1(9)	Br(1B)-C(5B)-H(5B1)	109.5
C(11A)-C(12A)-O(4A)	119.3(9)	C(1B)-C(5B)-H(5B2)	109.5
C(13A)-C(12A)-O(4A)	118.5(10)	Br(1B)-C(5B)-H(5B2)	109.5
C(14A)-C(13A)-C(12A)	119.2(9)	H(5B1)-C(5B)-H(5B2)	108.1
C(14A)-C(13A)-H(13A)	120.4	O(3B)-C(6B)-O(2B)	104.9(7)

O(3B)-C(6B)-C(8B)	108.5(8)
O(2B)-C(6B)-C(8B)	108.3(8)
O(3B)-C(6B)-C(7B)	110.7(8)
O(2B)-C(6B)-C(7B)	110.9(8)
C(8B)-C(6B)-C(7B)	113.2(8)
C(6B)-C(7B)-H(7B1)	109.5
C(6B)-C(7B)-H(7B2)	109.5
H(7B1)-C(7B)-H(7B2)	109.5
C(6B)-C(7B)-H(7B3)	109.5
H(7B1)-C(7B)-H(7B3)	109.5
H(7B2)-C(7B)-H(7B3)	109.5
C(6B)-C(8B)-H(8B1)	109.5
C(6B)-C(8B)-H(8B2)	109.5
H(8B1)-C(8B)-H(8B2)	109.5
C(6B)-C(8B)-H(8B3)	109.5
H(8B1)-C(8B)-H(8B3)	109.5
H(8B2)-C(8B)-H(8B3)	109.5
C(14B)-C(9B)-C(10B)	118.3(8)
C(14B)-C(9B)-C(4B)	122.8(8)
C(10B)-C(9B)-C(4B)	119.0(8)
C(11B)-C(10B)-C(9B)	122.0(9)
C(11B)-C(10B)-H(10B)	119.0
C(9B)-C(10B)-H(10B)	119.0
C(12B)-C(11B)-C(10B)	118.5(10)
C(12B)-C(11B)-H(11B)	120.7
C(10B)-C(11B)-H(11B)	120.7
C(11B)-C(12B)-C(13B)	122.1(10)
C(11B)-C(12B)-O(4B)	117.1(10)
C(13B)-C(12B)-O(4B)	120.9(11)
C(12B)-C(13B)-C(14B)	119.4(10)
C(12B)-C(13B)-H(13B)	120.3
C(14B)-C(13B)-H(13B)	120.3
C(9B)-C(14B)-C(13B)	119.8(10)
C(9B)-C(14B)-H(14B)	120.1
C(13B)-C(14B)-H(14B)	120.1
C(20B)-C(15B)-O(4B)	125.3(9)
C(20B)-C(15B)-C(16B)	120.9(10)
O(4B)-C(15B)-C(16B)	113.7(10)
C(17B)-C(16B)-C(15B)	119.1(13)
C(17B)-C(16B)-H(16B)	120.4
C(15B)-C(16B)-H(16B)	120.4
C(16B)-C(17B)-C(18B)	119.8(13)
C(16B)-C(17B)-H(17B)	120.1
C(18B)-C(17B)-H(17B)	120.1
C(17B)-C(18B)-C(19B)	121.1(12)
C(17B)-C(18B)-H(18B)	119.5
C(19B)-C(18B)-H(18B)	119.5
C(20B)-C(19B)-C(18B)	119.1(12)
C(20B)-C(19B)-H(19B)	120.5
C(18B)-C(19B)-H(19B)	120.5
C(15B)-C(20B)-C(19B)	120.0(11)
C(15B)-C(20B)-H(20B)	120.0
C(19B)-C(20B)-H(20B)	120.0

Torsion angles [°]

C(5A)-C(1A)-N(1A)-O(1A)	77.8(9)
C(2A)-C(1A)-N(1A)-O(1A)	-161.9(6)
C(5A)-C(1A)-N(1A)-C(4A)	-161.7(8)
C(2A)-C(1A)-N(1A)-C(4A)	-41.4(8)
C(6A)-O(2A)-C(2A)-C(3A)	18.9(9)
C(6A)-O(2A)-C(2A)-C(1A)	132.6(9)
C(5A)-C(1A)-C(2A)-O(2A)	28.7(10)
N(1A)-C(1A)-C(2A)-O(2A)	-90.5(8)
C(5A)-C(1A)-C(2A)-C(3A)	140.4(8)
N(1A)-C(1A)-C(2A)-C(3A)	21.2(8)
C(6A)-O(3A)-C(3A)-C(2A)	-27.2(10)
C(6A)-O(3A)-C(3A)-C(4A)	-139.3(9)
O(2A)-C(2A)-C(3A)-O(3A)	4.7(9)
C(1A)-C(2A)-C(3A)-O(3A)	-110.2(7)
O(2A)-C(2A)-C(3A)-C(4A)	119.2(7)
C(1A)-C(2A)-C(3A)-C(4A)	4.3(8)
O(1A)-N(1A)-C(4A)-C(9A)	41.2(8)
C(1A)-N(1A)-C(4A)-C(9A)	-78.5(9)
O(1A)-N(1A)-C(4A)-C(3A)	164.1(5)
C(1A)-N(1A)-C(4A)-C(3A)	44.4(7)
O(3A)-C(3A)-C(4A)-N(1A)	83.4(8)
C(2A)-C(3A)-C(4A)-N(1A)	-27.7(7)
O(3A)-C(3A)-C(4A)-C(9A)	-150.4(8)
C(2A)-C(3A)-C(4A)-C(9A)	98.4(8)
N(1A)-C(1A)-C(5A)-Br(1C)	-100.8(10)
C(2A)-C(1A)-C(5A)-Br(1C)	147.1(9)
N(1A)-C(1A)-C(5A)-Br(1A)	-168.2(7)
C(2A)-C(1A)-C(5A)-Br(1A)	79.7(9)
C(3A)-O(3A)-C(6A)-O(2A)	40.0(10)
C(3A)-O(3A)-C(6A)-C(8A)	-80.6(12)
C(3A)-O(3A)-C(6A)-C(7A)	153.6(10)
C(2A)-O(2A)-C(6A)-O(3A)	-36.8(10)
C(2A)-O(2A)-C(6A)-C(8A)	83.8(10)
C(2A)-O(2A)-C(6A)-C(7A)	-149.9(8)
N(1A)-C(4A)-C(9A)-C(10A)	88.5(11)
C(3A)-C(4A)-C(9A)-C(10A)	-27.4(13)
N(1A)-C(4A)-C(9A)-C(14A)	-94.1(10)
C(3A)-C(4A)-C(9A)-C(14A)	150.0(9)
C(14A)-C(9A)-C(10A)-C(11A)	-1.4(17)
C(4A)-C(9A)-C(10A)-C(11A)	176.0(10)
C(9A)-C(10A)-C(11A)-C(12A)	-0.8(18)
C(10A)-C(11A)-C(12A)-C(13A)	3.6(19)
C(10A)-C(11A)-C(12A)-O(4A)	-176.5(11)
C(15A)-O(4A)-C(12A)-C(11A)	100.6(13)
C(15A)-O(4A)-C(12A)-C(13A)	-79.4(13)
C(11A)-C(12A)-C(13A)-C(14A)	-4.3(19)
O(4A)-C(12A)-C(13A)-C(14A)	175.8(10)
C(12A)-C(13A)-C(14A)-C(9A)	2.0(18)
C(10A)-C(9A)-C(14A)-C(13A)	0.7(16)
C(4A)-C(9A)-C(14A)-C(13A)	-176.8(10)
C(12A)-O(4A)-C(15A)-C(20A)	-11.5(15)
C(12A)-O(4A)-C(15A)-C(16A)	169.3(8)
O(4A)-C(15A)-C(16A)-C(17A)	177.4(10)
C(20A)-C(15A)-C(16A)-C(17A)	-2.0(14)
C(15A)-C(16A)-C(17A)-C(18A)	0.7(18)
C(16A)-C(17A)-C(18A)-C(19A)	1(2)
C(17A)-C(18A)-C(19A)-C(20A)	-1.2(19)
O(4A)-C(15A)-C(20A)-C(19A)	-177.6(12)
C(16A)-C(15A)-C(20A)-C(19A)	1.7(16)
C(18A)-C(19A)-C(20A)-C(15A)	0(2)
C(5B)-C(1B)-N(1B)-O(1B)	71.1(8)
C(2B)-C(1B)-N(1B)-O(1B)	-165.0(5)
C(5B)-C(1B)-N(1B)-C(4B)	-170.6(7)

C(2B)-C(1B)-N(1B)-C(4B)	-46.7(7)
C(6B)-O(2B)-C(2B)-C(1B)	135.5(7)
C(6B)-O(2B)-C(2B)-C(3B)	22.1(8)
N(1B)-C(1B)-C(2B)-O(2B)	-83.3(7)
C(5B)-C(1B)-C(2B)-O(2B)	34.9(10)
N(1B)-C(1B)-C(2B)-C(3B)	27.6(8)
C(5B)-C(1B)-C(2B)-C(3B)	145.9(7)
C(6B)-O(3B)-C(3B)-C(4B)	-133.6(7)
C(6B)-O(3B)-C(3B)-C(2B)	-19.9(8)
O(2B)-C(2B)-C(3B)-O(3B)	-1.6(8)
C(1B)-C(2B)-C(3B)-O(3B)	-117.7(7)
O(2B)-C(2B)-C(3B)-C(4B)	114.9(6)
C(1B)-C(2B)-C(3B)-C(4B)	-1.2(8)
O(1B)-N(1B)-C(4B)-C(9B)	39.9(8)
C(1B)-N(1B)-C(4B)-C(9B)	-77.1(8)
O(1B)-N(1B)-C(4B)-C(3B)	163.0(6)
C(1B)-N(1B)-C(4B)-C(3B)	46.0(7)
O(3B)-C(3B)-C(4B)-N(1B)	88.5(7)
C(2B)-C(3B)-C(4B)-N(1B)	-25.2(7)
O(3B)-C(3B)-C(4B)-C(9B)	-147.8(7)
C(2B)-C(3B)-C(4B)-C(9B)	98.4(8)
N(1B)-C(1B)-C(5B)-Br(1B)	-174.5(6)
C(2B)-C(1B)-C(5B)-Br(1B)	73.3(8)
C(3B)-O(3B)-C(6B)-O(2B)	34.2(8)
C(3B)-O(3B)-C(6B)-C(8B)	149.8(8)
C(3B)-O(3B)-C(6B)-C(7B)	-85.5(9)
C(2B)-O(2B)-C(6B)-O(3B)	-35.3(8)
C(2B)-O(2B)-C(6B)-C(8B)	-151.1(7)
C(2B)-O(2B)-C(6B)-C(7B)	84.2(8)
N(1B)-C(4B)-C(9B)-C(14B)	85.1(10)
C(3B)-C(4B)-C(9B)-C(14B)	-29.0(13)
N(1B)-C(4B)-C(9B)-C(10B)	-96.0(10)
C(3B)-C(4B)-C(9B)-C(10B)	149.9(8)
C(14B)-C(9B)-C(10B)-C(11B)	1.9(14)
C(4B)-C(9B)-C(10B)-C(11B)	-177.1(9)
C(9B)-C(10B)-C(11B)-C(12B)	-0.1(16)
C(10B)-C(11B)-C(12B)-C(13B)	-1.3(17)
C(10B)-C(11B)-C(12B)-O(4B)	179.9(9)
C(15B)-O(4B)-C(12B)-C(11B)	-128.8(12)
C(15B)-O(4B)-C(12B)-C(13B)	52.4(18)
C(11B)-C(12B)-C(13B)-C(14B)	0.9(18)
O(4B)-C(12B)-C(13B)-C(14B)	179.6(10)
C(10B)-C(9B)-C(14B)-C(13B)	-2.3(15)
C(4B)-C(9B)-C(14B)-C(13B)	176.6(9)
C(12B)-C(13B)-C(14B)-C(9B)	1.0(17)
C(12B)-O(4B)-C(15B)-C(20B)	17(2)
C(12B)-O(4B)-C(15B)-C(16B)	-166.0(12)
C(20B)-C(15B)-C(16B)-C(17B)	0(2)
O(4B)-C(15B)-C(16B)-C(17B)	-178.0(13)
C(15B)-C(16B)-C(17B)-C(18B)	0(2)
C(16B)-C(17B)-C(18B)-C(19B)	1(3)
C(17B)-C(18B)-C(19B)-C(20B)	-2(2)
O(4B)-C(15B)-C(20B)-C(19B)	176.6(13)
C(16B)-C(15B)-C(20B)-C(19B)	-1(2)
C(18B)-C(19B)-C(20B)-C(15B)	2(2)

H-Bond lengths [Å] and angles [°]

O1A-N1B	2.8155 (0.78)
H1AA-N1B	2.1293
O1B-N1A	2.8318 (0.91)
H1BB-N1A	2.1389 (0.1124)
O1A-H1AA-N1B	141.18
O1B-H1BB-N1A	151.19 (12.02)

9.6 (2*S*,3*R*,4*S*,5*S*)-2-Bromomethyl-5-(4-*N*,*N*-dimethylanilino)-1,3,4-trihydroxy-3,4-*O*-isopropylidene-pyrrolidine (51)

$C_{16}H_{23}BrN_2O_3$

monoclinic, $P2_1$

a = 6.3915(7) Å

b = 12.7516(19) Å

c = 11.0195(13) Å

α = 90 °

β = 101.856(9) °

γ = 90 °

V = 878.95(19) Å^3

Z = 2, $R(F)$ = 0.0429

$R_w(F^2)$ = 0.0880

Crystal size: 0.5 x 0.5 x 0.4 mm

Calculated density: 1.403 g/cm^3

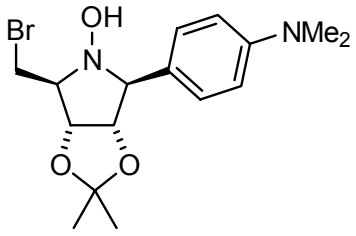
2θ-Range for data collection: 1.89 – 27.49 °

Independent reflections: 4036

Observed reflections: 2882

Contributed reflections to refinement: 4036

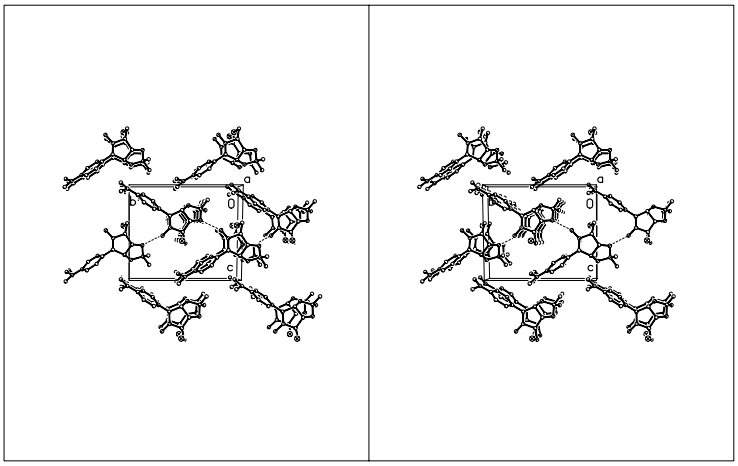
Refined parameters: 204



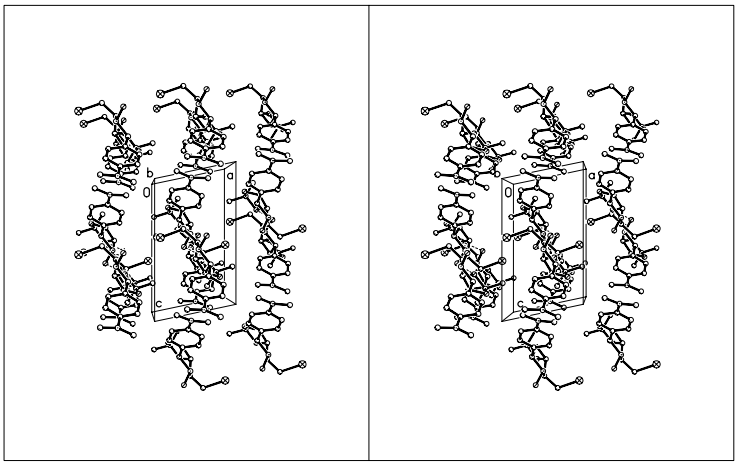
51

Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

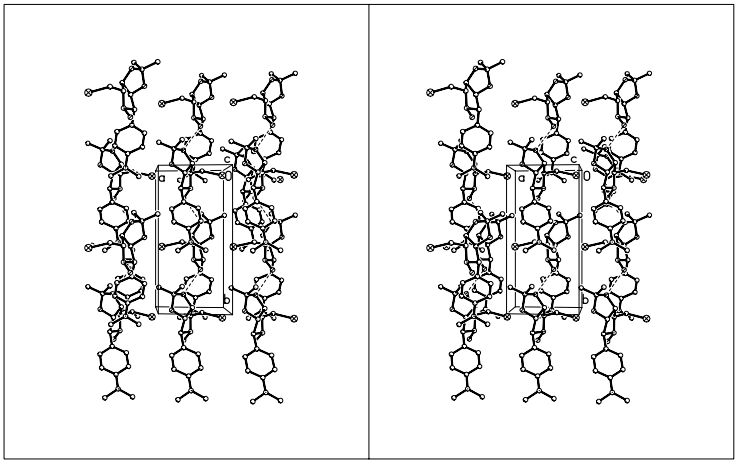
(a)



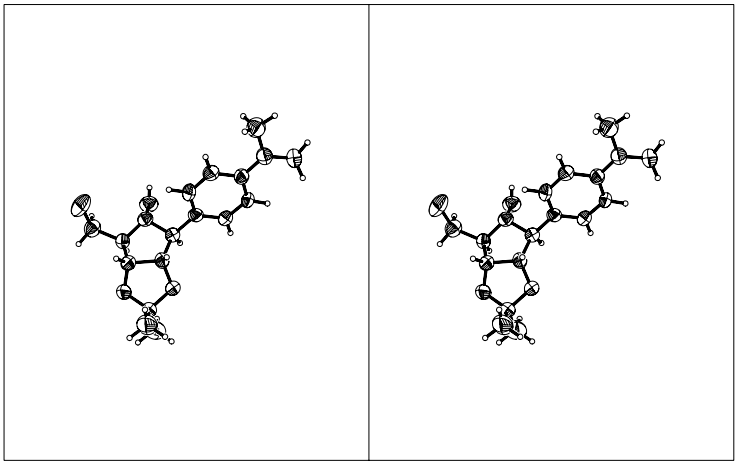
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

Br(1)-C(8)	1.933(4)	C(12)-N(2)-C(16)	119.6(3)
N(1)-O(1)	1.442(4)	C(12)-N(2)-C(15)	119.7(3)
N(1)-C(1)	1.457(5)	C(16)-N(2)-C(15)	117.9(3)
N(1)-C(4)	1.467(4)	O(2)-C(2)-C(1)	111.1(3)
C(1)-C(8)	1.513(5)	O(2)-C(2)-C(3)	105.0(3)
C(1)-C(2)	1.517(5)	C(1)-C(2)-C(3)	104.6(3)
C(1)-H(1)	0.98	O(2)-C(2)-H(2)	111.9
O(1)-H(1A)	0.84(4)	C(1)-C(2)-H(2)	111.9
O(2)-C(2)	1.413(5)	C(3)-C(2)-H(2)	111.9
O(2)-C(5)	1.421(4)	C(3)-O(3)-C(5)	108.6(3)
N(2)-C(12)	1.393(4)	O(3)-C(3)-C(2)	103.7(3)
N(2)-C(16)	1.428(5)	O(3)-C(3)-C(4)	111.7(3)
N(2)-C(15)	1.436(5)	C(2)-C(3)-C(4)	106.2(3)
C(2)-C(3)	1.537(5)	O(3)-C(3)-H(3)	111.6
C(2)-H(2)	0.98	C(2)-C(3)-H(3)	111.6
O(3)-C(3)	1.417(4)	C(4)-C(3)-H(3)	111.6
O(3)-C(5)	1.430(5)	N(1)-C(4)-C(9)	111.7(3)
C(3)-C(4)	1.542(5)	N(1)-C(4)-C(3)	101.6(3)
C(3)-H(3)	0.98	C(9)-C(4)-C(3)	115.1(3)
C(4)-C(9)	1.506(5)	N(1)-C(4)-H(4)	109.4
C(4)-H(4)	0.98	C(9)-C(4)-H(4)	109.4
C(5)-C(6)	1.492(7)	C(3)-C(4)-H(4)	109.4
C(5)-C(7)	1.498(7)	O(2)-C(5)-O(3)	103.8(3)
C(6)-H(6A)	0.96	O(2)-C(5)-C(6)	109.9(4)
C(6)-H(6B)	0.96	O(3)-C(5)-C(6)	108.5(4)
C(6)-H(6C)	0.96	O(2)-C(5)-C(7)	110.2(4)
C(7)-H(7A)	0.96	O(3)-C(5)-C(7)	110.0(4)
C(7)-H(7B)	0.96	C(6)-C(5)-C(7)	114.0(4)
C(7)-H(7C)	0.96	C(5)-C(6)-H(6A)	109.5
C(8)-H(8A)	0.97	C(5)-C(6)-H(6B)	109.5
C(8)-H(8B)	0.97	H(6A)-C(6)-H(6B)	109.5
C(9)-C(10)	1.384(5)	C(5)-C(6)-H(6C)	109.5
C(9)-C(14)	1.387(5)	H(6A)-C(6)-H(6C)	109.5
C(10)-C(11)	1.384(5)	H(6B)-C(6)-H(6C)	109.5
C(10)-H(10)	0.93	C(5)-C(7)-H(7A)	109.5
C(11)-C(12)	1.407(5)	C(5)-C(7)-H(7B)	109.5
C(11)-H(11)	0.93	H(7A)-C(7)-H(7B)	109.5
C(12)-C(13)	1.399(5)	C(5)-C(7)-H(7C)	109.5
C(13)-C(14)	1.376(5)	H(7A)-C(7)-H(7C)	109.5
C(13)-H(13)	0.93	H(7B)-C(7)-H(7C)	109.5
C(14)-H(14)	0.93	C(1)-C(8)-Br(1)	113.2(2)
C(15)-H(15A)	0.96	C(1)-C(8)-H(8A)	108.9
C(15)-H(15B)	0.96	Br(1)-C(8)-H(8A)	108.9
C(15)-H(15C)	0.96	C(1)-C(8)-H(8B)	108.9
C(16)-H(16A)	0.96	Br(1)-C(8)-H(8B)	108.9
C(16)-H(16B)	0.96	H(8A)-C(8)-H(8B)	107.8
C(16)-H(16C)	0.96	C(10)-C(9)-C(14)	116.8(3)
O(1)-N(1)-C(1)	107.7(3)	C(10)-C(9)-C(4)	122.3(3)
O(1)-N(1)-C(4)	108.3(3)	C(14)-C(9)-C(4)	121.0(3)
C(1)-N(1)-C(4)	107.2(3)	C(9)-C(10)-C(11)	122.0(3)
N(1)-C(1)-C(8)	113.3(3)	C(9)-C(10)-H(10)	119.0
N(1)-C(1)-C(2)	102.3(3)	C(11)-C(10)-H(10)	119.0
C(8)-C(1)-C(2)	116.0(3)	C(10)-C(11)-C(12)	121.0(3)
N(1)-C(1)-H(1)	108.3	C(10)-C(11)-H(11)	119.5
C(8)-C(1)-H(1)	108.3	C(12)-C(11)-H(11)	119.5
C(2)-C(1)-H(1)	108.3	N(2)-C(12)-C(13)	122.0(3)
N(1)-O(1)-H(1A)	96(3)	N(2)-C(12)-C(11)	121.3(3)
C(2)-O(2)-C(5)	109.3(3)	C(13)-C(12)-C(11)	116.7(3)

C(14)-C(13)-C(12)	121.1(3)
C(14)-C(13)-H(13)	119.5
C(12)-C(13)-H(13)	119.5
C(13)-C(14)-C(9)	122.4(3)
C(13)-C(14)-H(14)	118.8
C(9)-C(14)-H(14)	118.8
N(2)-C(15)-H(15A)	109.5
N(2)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
N(2)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
N(2)-C(16)-H(16A)	109.5
N(2)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
N(2)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5

Torsion angles [°]

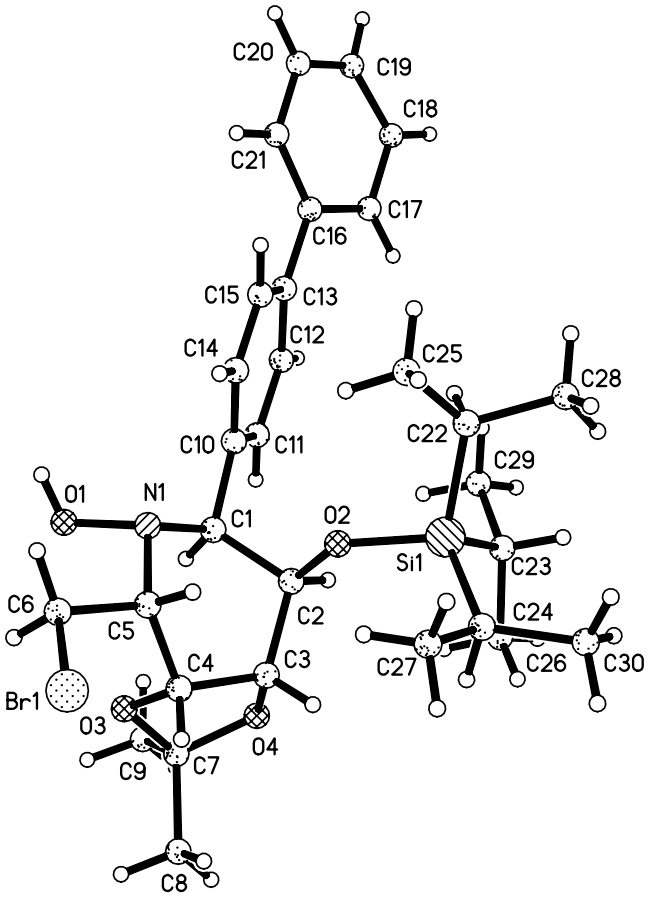
O(1)-N(1)-C(1)-C(8)	-74.5(3)
C(4)-N(1)-C(1)-C(8)	169.2(3)
O(1)-N(1)-C(1)-C(2)	159.9(3)
C(4)-N(1)-C(1)-C(2)	43.6(3)
C(5)-O(2)-C(2)-C(1)	127.2(3)
C(5)-O(2)-C(2)-C(3)	14.6(4)
N(1)-C(1)-C(2)-O(2)	-141.6(3)
C(8)-C(1)-C(2)-O(2)	94.6(4)
N(1)-C(1)-C(2)-C(3)	-28.7(3)
C(8)-C(1)-C(2)-C(3)	-152.6(3)
C(5)-O(3)-C(3)-C(2)	-22.5(4)
C(5)-O(3)-C(3)-C(4)	-136.5(3)
O(2)-C(2)-C(3)-O(3)	4.9(4)
C(1)-C(2)-C(3)-O(3)	-112.2(3)
O(2)-C(2)-C(3)-C(4)	122.7(3)
C(1)-C(2)-C(3)-C(4)	5.7(4)
O(1)-N(1)-C(4)-C(9)	81.6(3)
C(1)-N(1)-C(4)-C(9)	-162.5(3)
O(1)-N(1)-C(4)-C(3)	-155.3(3)
C(1)-N(1)-C(4)-C(3)	-39.3(3)
O(3)-C(3)-C(4)-N(1)	131.8(3)
C(2)-C(3)-C(4)-N(1)	19.3(3)
O(3)-C(3)-C(4)-C(9)	-107.4(3)
C(2)-C(3)-C(4)-C(9)	140.1(3)
C(2)-O(2)-C(5)-O(3)	-28.3(4)
C(2)-O(2)-C(5)-C(6)	-144.2(4)
C(2)-O(2)-C(5)-C(7)	89.4(4)
C(3)-O(3)-C(5)-O(2)	31.7(4)
C(3)-O(3)-C(5)-C(6)	148.6(4)
C(3)-O(3)-C(5)-C(7)	-86.1(4)
N(1)-C(1)-C(8)-Br(1)	-56.7(4)
C(2)-C(1)-C(8)-Br(1)	61.2(4)
N(1)-C(4)-C(9)-C(10)	36.0(5)
C(3)-C(4)-C(9)-C(10)	-79.1(4)
N(1)-C(4)-C(9)-C(14)	-144.5(3)
C(3)-C(4)-C(9)-C(14)	100.4(4)
C(14)-C(9)-C(10)-C(11)	1.3(5)
C(4)-C(9)-C(10)-C(11)	-179.2(3)
C(9)-C(10)-C(11)-C(12)	-0.4(6)
C(16)-N(2)-C(12)-C(13)	6.7(5)
C(15)-N(2)-C(12)-C(13)	167.2(4)
C(16)-N(2)-C(12)-C(11)	172.0(4)
C(15)-N(2)-C(12)-C(11)	-11.6(5)
C(10)-C(11)-C(12)-N(2)	177.8(3)
C(10)-C(11)-C(12)-C(13)	-1.0(5)
N(2)-C(12)-C(13)-C(14)	-177.2(3)
C(11)-C(12)-C(13)-C(14)	1.6(5)
C(12)-C(13)-C(14)-C(9)	-0.9(6)
C(10)-C(9)-C(14)-C(13)	-0.6(5)
C(4)-C(9)-C(14)-C(13)	179.9(3)

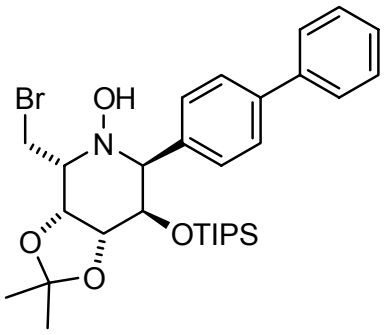
H-Bond lengths [Å] and angles [°]

O1-O2_$1	2.8031(0.0040)
H1A-O2_$1	2.0489(0.0423)
O1-H1A-O2_$1	149.10(3.87)

9.7 (2*R*,3*R*,4*R*,5*R*,6*S*)-6-([1,1'-Biphenyl]-4-yl)-2-bromomethyl-3,4,5-trihydroxy-3,4-*O*-isopropylidene-5-triisopropylsilyloxy-piperidine (52)

$C_{30}H_{44}BrCl_{0.67}NO_4Si$
hexagonal, *R*3
a = 32.128(3) Å
b = 32.128(3) Å
c = 8.545(2) Å
α = 90°
β = 90°
γ = 120°
V = 7639 Å^3
Z = 9, *R*(*F*) = 0.0781
$R_w(F^2)$ = 0.1342
Crystal size: 0.7 x 0.05 x 0.05 mm
Calculated density: 1.210 g/cm^3
2θ-Range for data collection: 2.54 – 22.50 °
Independent reflections: 7327
Observed reflections: 4435
Contributed reflections to refinement: 7327
Refined parameters: 348





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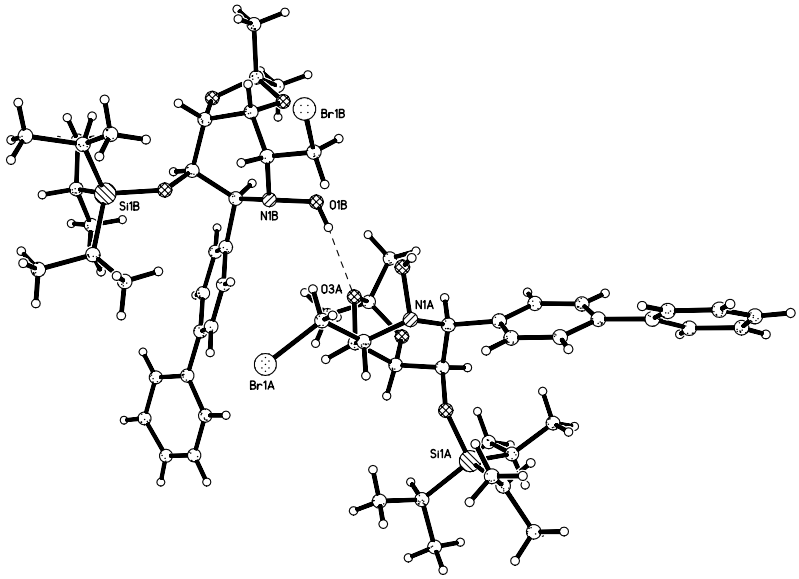
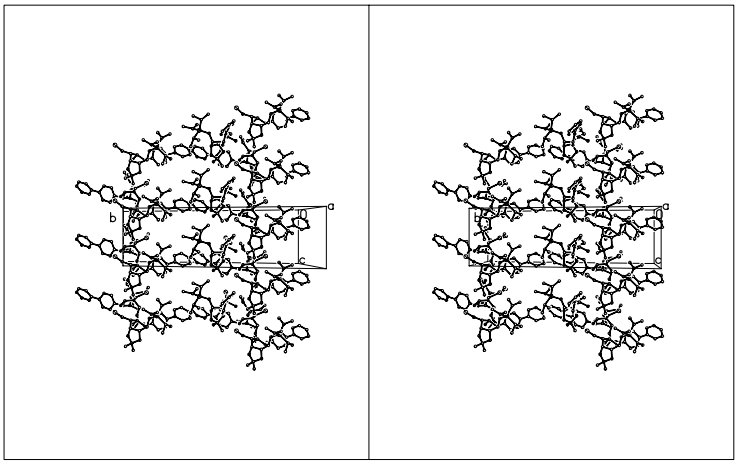
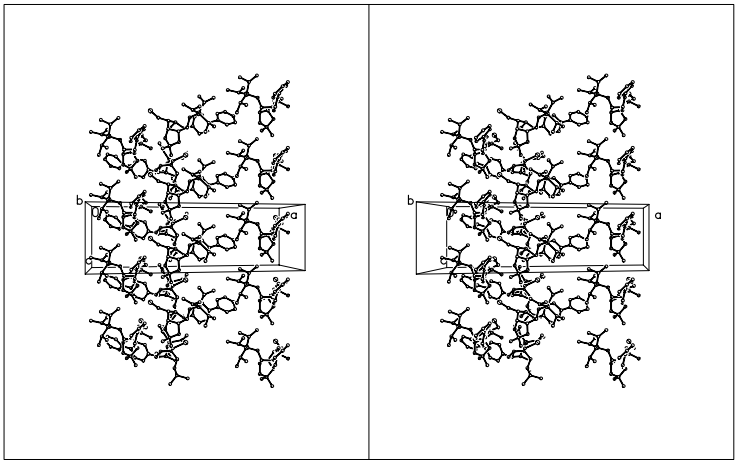


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

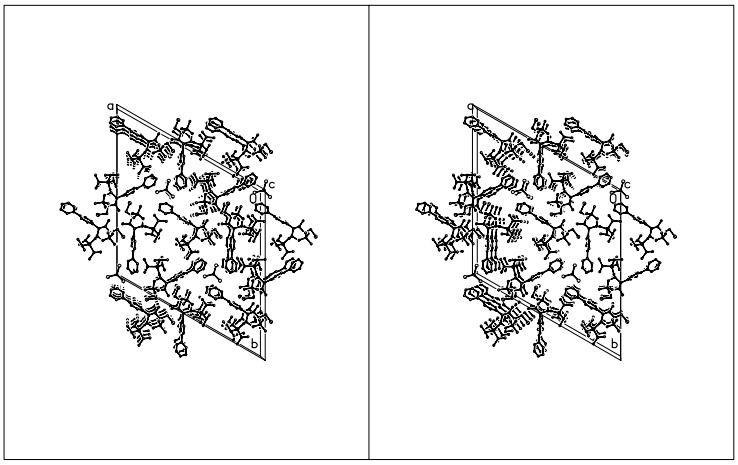
(a)



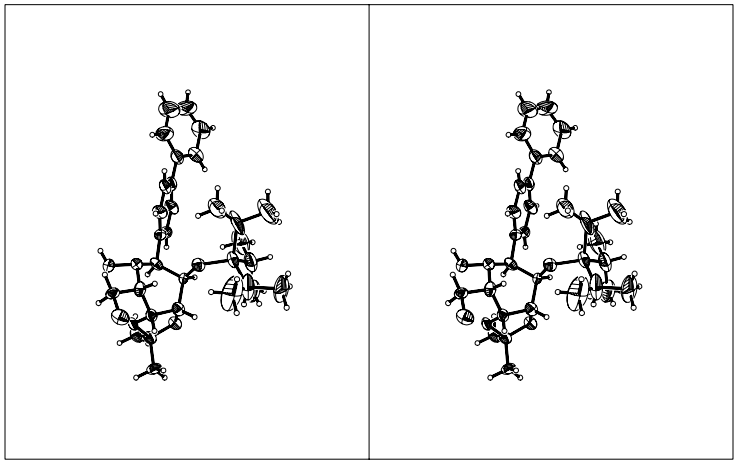
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

Br(1)-C(6)	1.938(10)	C(3)-H(3)	0.9800
Si(1)-O(2)	1.651(7)	O(4)-C(7)	1.402(12)
Si(1)-C(24)	1.821(15)	C(4)-C(5)	1.499(14)
Si(1)-C(22)	1.857(17)	C(4)-H(4)	0.9800
Si(1)-C(23)	1.860(15)	C(5)-C(6)	1.525(14)
O(1)-N(1)	1.451(10)	C(5)-H(5)	0.9800
O(1)-H(1A)	0.8200	C(6)-H(6A)	0.9700
N(1)-C(5)	1.474(13)	C(6)-H(6B)	0.9700
N(1)-C(1)	1.492(13)	C(7)-C(9)	1.509(15)
C(1)-C(2)	1.510(14)	C(7)-C(8)	1.548(16)
C(1)-C(10)	1.523(14)	C(8)-H(8A)	0.9600
C(1)-H(1)	0.9800	C(8)-H(8B)	0.9600
O(2)-C(2)	1.407(11)	C(8)-H(8C)	0.9600
C(2)-C(3)	1.531(13)	C(9)-H(9A)	0.9600
C(2)-H(2)	0.9800	C(9)-H(9B)	0.9600
O(3)-C(4)	1.421(12)	C(9)-H(9C)	0.9600
O(3)-C(7)	1.432(13)	C(10)-C(14)	1.363(14)
C(3)-O(4)	1.420(11)	C(10)-C(11)	1.370(15)
C(3)-C(4)	1.524(14)	C(11)-C(12)	1.394(15)

C(11)-H(11)	0.9300	N(1)-C(1)-C(10)	110.7(9)
C(12)-C(13)	1.330(16)	C(2)-C(1)-C(10)	111.9(8)
C(12)-H(12)	0.9300	N(1)-C(1)-H(1)	106.7
C(13)-C(15)	1.393(16)	C(2)-C(1)-H(1)	106.7
C(13)-C(16)	1.500(15)	C(10)-C(1)-H(1)	106.7
C(14)-C(15)	1.357(15)	C(2)-O(2)-Si(1)	128.0(6)
C(14)-H(14)	0.9300	O(2)-C(2)-C(1)	110.6(8)
C(15)-H(15)	0.9300	O(2)-C(2)-C(3)	111.2(8)
C(16)-C(17)	1.389(17)	C(1)-C(2)-C(3)	109.0(8)
C(16)-C(21)	1.394(18)	O(2)-C(2)-H(2)	108.7
C(17)-C(18)	1.419(19)	C(1)-C(2)-H(2)	108.7
C(17)-H(17)	0.9300	C(3)-C(2)-H(2)	108.7
C(18)-C(19)	1.38(2)	C(4)-O(3)-C(7)	106.2(7)
C(18)-H(18)	0.9300	O(4)-C(3)-C(4)	104.8(8)
C(19)-C(20)	1.35(3)	O(4)-C(3)-C(2)	109.4(8)
C(19)-H(19)	0.9300	C(4)-C(3)-C(2)	115.3(9)
C(20)-C(21)	1.37(2)	O(4)-C(3)-H(3)	109.0
C(20)-H(20)	0.9300	C(4)-C(3)-H(3)	109.0
C(21)-H(21)	0.9300	C(2)-C(3)-H(3)	109.0
C(22)-C(25)	1.39(2)	C(7)-O(4)-C(3)	109.4(8)
C(22)-C(28)	1.522(19)	O(3)-C(4)-C(5)	110.6(8)
C(22)-H(22)	0.9800	O(3)-C(4)-C(3)	102.4(8)
C(23)-C(29)	1.49(2)	C(5)-C(4)-C(3)	111.3(9)
C(23)-C(26)	1.52(2)	O(3)-C(4)-H(4)	110.8
C(23)-H(23)	0.9800	C(5)-C(4)-H(4)	110.8
C(24)-C(27)	1.45(2)	C(3)-C(4)-H(4)	110.8
C(24)-C(30)	1.540(19)	N(1)-C(5)-C(4)	117.2(8)
C(24)-H(24)	0.9800	N(1)-C(5)-C(6)	108.7(8)
C(25)-H(25A)	0.9600	C(4)-C(5)-C(6)	115.4(9)
C(25)-H(25B)	0.9600	N(1)-C(5)-H(5)	104.7
C(25)-H(25C)	0.9600	C(4)-C(5)-H(5)	104.7
C(26)-H(26A)	0.9600	C(6)-C(5)-H(5)	104.7
C(26)-H(26B)	0.9600	C(5)-C(6)-Br(1)	110.8(7)
C(26)-H(26C)	0.9600	C(5)-C(6)-H(6A)	109.5
C(27)-H(27A)	0.9600	Br(1)-C(6)-H(6A)	109.5
C(27)-H(27B)	0.9600	C(5)-C(6)-H(6B)	109.5
C(27)-H(27C)	0.9600	Br(1)-C(6)-H(6B)	109.5
C(28)-H(28A)	0.9600	H(6A)-C(6)-H(6B)	108.1
C(28)-H(28B)	0.9600	O(4)-C(7)-O(3)	104.4(8)
C(28)-H(28C)	0.9600	O(4)-C(7)-C(9)	110.1(9)
C(29)-H(29A)	0.9600	O(3)-C(7)-C(9)	108.7(9)
C(29)-H(29B)	0.9600	O(4)-C(7)-C(8)	111.4(10)
C(29)-H(29C)	0.9600	O(3)-C(7)-C(8)	110.5(9)
C(30)-H(30A)	0.9600	C(9)-C(7)-C(8)	111.5(10)
C(30)-H(30B)	0.9600	C(7)-C(8)-H(8A)	109.5
C(30)-H(30C)	0.9600	C(7)-C(8)-H(8B)	109.5
C(1S)-Cl(1S)#1	1.74(4)	H(8A)-C(8)-H(8B)	109.5
C(1S)-Cl(1S)#2	1.74(4)	C(7)-C(8)-H(8C)	109.5
C(1S)-Cl(1S)	1.74(4)	H(8A)-C(8)-H(8C)	109.5
O(2)-Si(1)-C(24)	107.2(6)	H(8B)-C(8)-H(8C)	109.5
O(2)-Si(1)-C(22)	105.1(6)	C(7)-C(9)-H(9A)	109.5
C(24)-Si(1)-C(22)	113.3(11)	C(7)-C(9)-H(9B)	109.5
O(2)-Si(1)-C(23)	113.5(6)	H(9A)-C(9)-H(9B)	109.5
C(24)-Si(1)-C(23)	109.0(8)	C(7)-C(9)-H(9C)	109.5
C(22)-Si(1)-C(23)	108.7(9)	H(9A)-C(9)-H(9C)	109.5
N(1)-O(1)-H(1A)	109.5	H(9B)-C(9)-H(9C)	109.5
O(1)-N(1)-C(5)	108.0(7)	C(14)-C(10)-C(11)	119.0(10)
O(1)-N(1)-C(1)	106.0(8)	C(14)-C(10)-C(1)	122.5(10)
C(5)-N(1)-C(1)	112.4(8)	C(11)-C(10)-C(1)	118.5(10)
N(1)-C(1)-C(2)	113.7(8)	C(10)-C(11)-C(12)	118.4(11)

C(10)-C(11)-H(11)	120.8
C(12)-C(11)-H(11)	120.8
C(13)-C(12)-C(11)	123.6(11)
C(13)-C(12)-H(12)	118.2
C(11)-C(12)-H(12)	118.2
C(12)-C(13)-C(15)	116.6(11)
C(12)-C(13)-C(16)	123.0(11)
C(15)-C(13)-C(16)	120.3(12)
C(15)-C(14)-C(10)	121.0(11)
C(15)-C(14)-H(14)	119.5
C(10)-C(14)-H(14)	119.5
C(14)-C(15)-C(13)	121.3(12)
C(14)-C(15)-H(15)	119.3
C(13)-C(15)-H(15)	119.3
C(17)-C(16)-C(21)	117.8(13)
C(17)-C(16)-C(13)	120.6(13)
C(21)-C(16)-C(13)	121.6(13)
C(16)-C(17)-C(18)	120.1(15)
C(16)-C(17)-H(17)	120.0
C(18)-C(17)-H(17)	120.0
C(19)-C(18)-C(17)	117.5(16)
C(19)-C(18)-H(18)	121.2
C(17)-C(18)-H(18)	121.2
C(20)-C(19)-C(18)	123.9(18)
C(20)-C(19)-H(19)	118.1
C(18)-C(19)-H(19)	118.1
C(19)-C(20)-C(21)	117.3(19)
C(19)-C(20)-H(20)	121.4
C(21)-C(20)-H(20)	121.4
C(20)-C(21)-C(16)	123.4(17)
C(20)-C(21)-H(21)	118.3
C(16)-C(21)-H(21)	118.3
C(25)-C(22)-C(28)	115.6(15)
C(25)-C(22)-Si(1)	125.2(13)
C(28)-C(22)-Si(1)	115.0(13)
C(25)-C(22)-H(22)	96.7
C(28)-C(22)-H(22)	96.7
Si(1)-C(22)-H(22)	96.7
C(29)-C(23)-C(26)	110.8(16)
C(29)-C(23)-Si(1)	115.6(13)
C(26)-C(23)-Si(1)	115.0(11)
C(29)-C(23)-H(23)	104.7
C(26)-C(23)-H(23)	104.7
Si(1)-C(23)-H(23)	104.7
C(27)-C(24)-C(30)	113.1(15)
C(27)-C(24)-Si(1)	116.6(13)
C(30)-C(24)-Si(1)	119.2(13)
C(27)-C(24)-H(24)	101.2
C(30)-C(24)-H(24)	101.2
Si(1)-C(24)-H(24)	101.2
C(22)-C(25)-H(25A)	109.5
C(22)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
C(22)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
C(23)-C(26)-H(26A)	109.5
C(23)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(23)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
C(24)-C(27)-H(27A)	109.5
C(24)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	109.5
C(24)-C(27)-H(27C)	109.5
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
C(22)-C(28)-H(28A)	109.5
C(22)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
C(22)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(23)-C(29)-H(29A)	109.5
C(23)-C(29)-H(29B)	109.5
H(29A)-C(29)-H(29B)	109.5
C(23)-C(29)-H(29C)	109.5
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
C(24)-C(30)-H(30A)	109.5
C(24)-C(30)-H(30B)	109.5
H(30A)-C(30)-H(30B)	109.5
C(24)-C(30)-H(30C)	109.5
H(30A)-C(30)-H(30C)	109.5
H(30B)-C(30)-H(30C)	109.5

Torsion angles [°]

Cl(1S)#1-C(1S)-Cl(1S)#2	120.0(3)
Cl(1S)#1-C(1S)-Cl(1S)	120.0(3)
Cl(1S)#2-C(1S)-Cl(1S)	120.0(3)
O(1)-N(1)-C(1)-C(2)	-147.3(8)
C(5)-N(1)-C(1)-C(2)	-29.5(11)
O(1)-N(1)-C(1)-C(10)	85.9(9)
C(5)-N(1)-C(1)-C(10)	-156.4(8)
C(24)-Si(1)-O(2)-C(2)	-100.1(11)
C(22)-Si(1)-O(2)-C(2)	139.0(12)
C(23)-Si(1)-O(2)-C(2)	20.3(11)
Si(1)-O(2)-C(2)-C(1)	-143.2(7)
Si(1)-O(2)-C(2)-C(3)	95.7(10)
N(1)-C(1)-C(2)-O(2)	-60.6(11)
C(10)-C(1)-C(2)-O(2)	65.6(11)
N(1)-C(1)-C(2)-C(3)	61.9(11)
C(10)-C(1)-C(2)-C(3)	-171.9(9)
O(2)-C(2)-C(3)-O(4)	-156.0(8)
C(1)-C(2)-C(3)-O(4)	81.9(10)
O(2)-C(2)-C(3)-C(4)	86.2(10)
C(1)-C(2)-C(3)-C(4)	-36.0(12)
C(4)-C(3)-O(4)-C(7)	-2.2(11)
C(2)-C(3)-O(4)-C(7)	-126.4(9)
C(7)-O(3)-C(4)-C(5)	152.1(9)
C(7)-O(3)-C(4)-C(3)	33.5(10)
O(4)-C(3)-C(4)-O(3)	-19.2(10)
C(2)-C(3)-C(4)-O(3)	101.2(10)
O(4)-C(3)-C(4)-C(5)	-137.4(9)
C(2)-C(3)-C(4)-C(5)	-17.0(12)
O(1)-N(1)-C(5)-C(4)	88.1(10)
C(1)-N(1)-C(5)-C(4)	-28.5(12)

O(1)-N(1)-C(5)-C(6)	-44.9(10)
C(1)-N(1)-C(5)-C(6)	-161.5(8)
O(3)-C(4)-C(5)-N(1)	-60.6(12)
C(3)-C(4)-C(5)-N(1)	52.5(12)
O(3)-C(4)-C(5)-C(6)	69.4(12)
C(3)-C(4)-C(5)-C(6)	-177.5(9)
N(1)-C(5)-C(6)-Br(1)	-158.0(7)
C(4)-C(5)-C(6)-Br(1)	68.0(11)
C(3)-O(4)-C(7)-O(3)	22.8(11)
C(3)-O(4)-C(7)-C(9)	139.3(9)
C(3)-O(4)-C(7)-C(8)	-96.5(11)
C(4)-O(3)-C(7)-O(4)	-35.8(11)
C(4)-O(3)-C(7)-C(9)	-153.3(9)
C(4)-O(3)-C(7)-C(8)	84.1(10)
N(1)-C(1)-C(10)-C(14)	32.2(14)
C(2)-C(1)-C(10)-C(14)	-95.7(13)
N(1)-C(1)-C(10)-C(11)	-150.0(9)
C(2)-C(1)-C(10)-C(11)	82.1(12)
C(14)-C(10)-C(11)-C(12)	3.0(16)
C(1)-C(10)-C(11)-C(12)	-175.0(10)
C(10)-C(11)-C(12)-C(13)	-3.6(18)
C(11)-C(12)-C(13)-C(15)	2.9(18)
C(11)-C(12)-C(13)-C(16)	179.5(11)
C(11)-C(10)-C(14)-C(15)	-2.1(17)
C(1)-C(10)-C(14)-C(15)	175.8(10)
C(10)-C(14)-C(15)-C(13)	1.5(18)
C(12)-C(13)-C(15)-C(14)	-1.9(18)
C(16)-C(13)-C(15)-C(14)	-178.6(11)
C(12)-C(13)-C(16)-C(17)	-32.3(18)
C(15)-C(13)-C(16)-C(17)	144.2(12)
C(12)-C(13)-C(16)-C(21)	148.1(14)
C(15)-C(13)-C(16)-C(21)	-35.4(18)
C(21)-C(16)-C(17)-C(18)	-3(2)
C(13)-C(16)-C(17)-C(18)	177.7(13)
C(16)-C(17)-C(18)-C(19)	2(2)
C(17)-C(18)-C(19)-C(20)	0(3)
C(18)-C(19)-C(20)-C(21)	-1(4)
C(19)-C(20)-C(21)-C(16)	0(3)
C(17)-C(16)-C(21)-C(20)	2(3)
C(13)-C(16)-C(21)-C(20)	-178.2(17)
O(2)-Si(1)-C(22)-C(25)	28(3)
C(24)-Si(1)-C(22)-C(25)	-89(2)
C(23)-Si(1)-C(22)-C(25)	150(2)
O(2)-Si(1)-C(22)-C(28)	-175.6(18)
C(24)-Si(1)-C(22)-C(28)	68(2)
C(23)-Si(1)-C(22)-C(28)	-54(2)
O(2)-Si(1)-C(23)-C(29)	59.6(14)
C(24)-Si(1)-C(23)-C(29)	179.1(14)
C(22)-Si(1)-C(23)-C(29)	-57.0(16)
O(2)-Si(1)-C(23)-C(26)	-71.4(13)
C(24)-Si(1)-C(23)-C(26)	48.0(14)
C(22)-Si(1)-C(23)-C(26)	172.0(13)
O(2)-Si(1)-C(24)-C(27)	-43(2)
C(22)-Si(1)-C(24)-C(27)	73(2)
C(23)-Si(1)-C(24)-C(27)	-166.1(19)
O(2)-Si(1)-C(24)-C(30)	175.6(15)
C(22)-Si(1)-C(24)-C(30)	-68.8(19)
C(23)-Si(1)-C(24)-C(30)	52.4(19)

9.8 (2*S*,3*R*,4*S*)-5-Bromomethyl-3,4-dihydroxy-3,4-*O*-isopropylidene-2-(4-methoxyphenyl)-3,4-dihydro-2*H*-pyrrole-1-oxide (63)

$C_{15}H_{28}BrNO_4$

orthorhombic, $P2_12_12_1$

a = 6.0358(4) Å

b = 15.959(2) Å

c = 16.6193(9) Å

α = 90 °

β = 90 °

γ = 90 °

V = 1600.8(3) Å^3

Z = 4, $R(F)$ = 0.0540

$R_w(F^2)$ = 0.1332

Crystal size: 0.5 x 0.1 x 0.03 mm

Calculated density: 1.478 g/cm^3

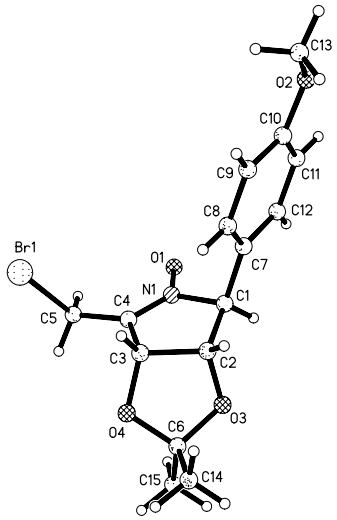
2θ-Range for data collection: 3.84 - 59.95 °

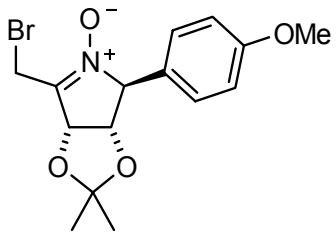
Independent reflections: 1716

Observed reflections: 1089

Contributed reflections to refinement: 1716

Refined parameters: 191

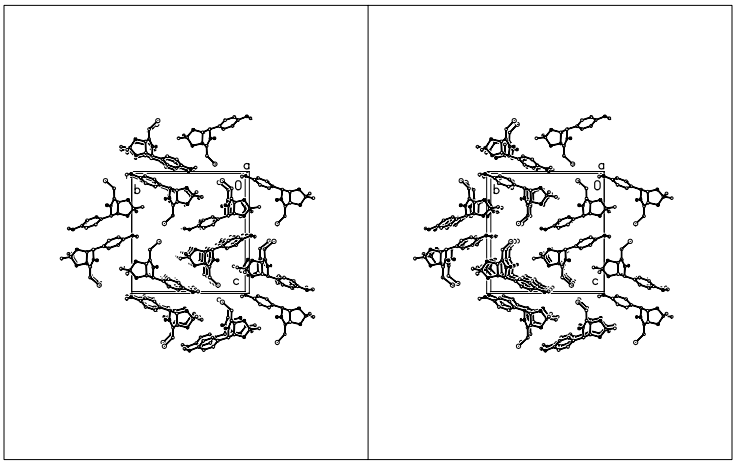




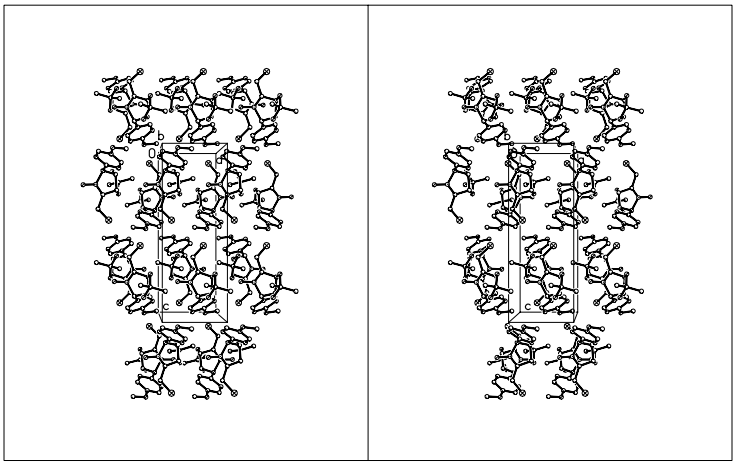
63

Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

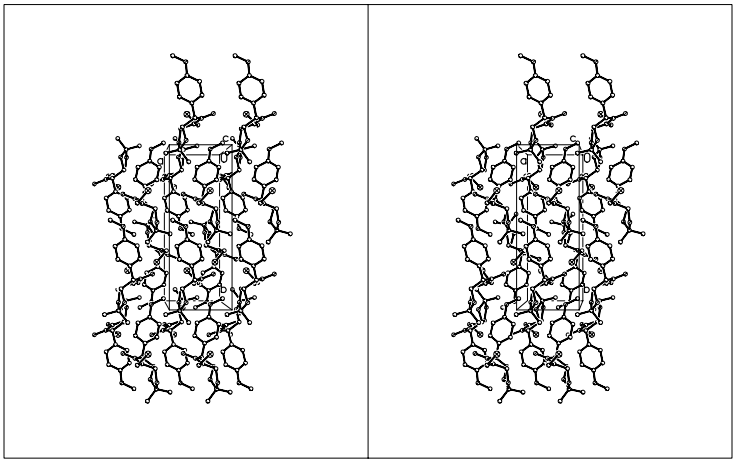
(a)



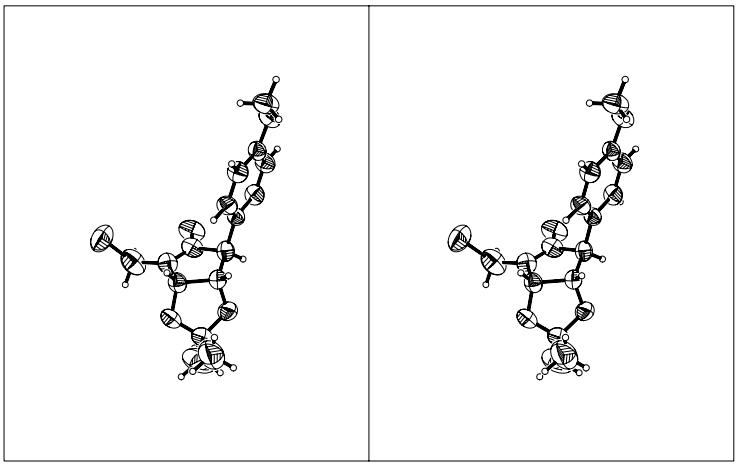
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

Br(1)-C(5)	1.916(12)	C(5)-H(5A)	0.97
N(1)-C(4)	1.281(10)	C(5)-H(5B)	0.97
N(1)-O(1)	1.293(8)	C(6)-C(14)	1.487(17)
N(1)-C(1)	1.511(10)	C(6)-C(15)	1.501(15)
C(1)-C(7)	1.523(11)	C(7)-C(8)	1.380(12)
C(1)-C(2)	1.524(12)	C(7)-C(12)	1.397(11)
C(1)-H(1)	0.98	C(8)-C(9)	1.391(11)
O(2)-C(10)	1.371(10)	C(8)-H(8)	0.93
O(2)-C(13)	1.402(12)	C(9)-C(10)	1.382(12)
C(2)-O(3)	1.424(9)	C(9)-H(9)	0.93
C(2)-C(3)	1.549(10)	C(10)-C(11)	1.374(12)
C(2)-H(2)	0.98	C(11)-C(12)	1.385(12)
O(3)-C(6)	1.444(10)	C(11)-H(11)	0.93
C(3)-O(4)	1.419(9)	C(12)-H(12)	0.93
C(3)-C(4)	1.498(12)	C(13)-H(13A)	0.96
C(3)-H(3)	0.98	C(13)-H(13B)	0.96
O(4)-C(6)	1.4(10)	C(13)-H(13C)	0.96
C(4)-C(5)	1.478(12)	C(14)-H(14A)	0.96

C(14)-H(14B)	0.96
C(14)-H(14C)	0.96
C(15)-H(15A)	0.96
C(15)-H(15B)	0.96
C(15)-H(15C)	0.96
C(4)-N(1)-O(1)	127.2(8)
C(4)-N(1)-C(1)	113.8(7)
O(1)-N(1)-C(1)	119.0(8)
N(1)-C(1)-C(7)	109.1(7)
N(1)-C(1)-C(2)	102.0(6)
C(7)-C(1)-C(2)	115.5(7)
N(1)-C(1)-H(1)	110.0
C(7)-C(1)-H(1)	110.0
C(2)-C(1)-H(1)	110.0
C(10)-O(2)-C(13)	117.9(8)
O(3)-C(2)-C(1)	107.8(7)
O(3)-C(2)-C(3)	102.5(7)
C(1)-C(2)-C(3)	106.6(7)
O(3)-C(2)-H(2)	113.0
C(1)-C(2)-H(2)	113.0
C(3)-C(2)-H(2)	113.0
C(2)-O(3)-C(6)	107.2(6)
O(4)-C(3)-C(4)	111.9(8)
O(4)-C(3)-C(2)	104.9(7)
C(4)-C(3)-C(2)	102.5(7)
O(4)-C(3)-H(3)	112.3
C(4)-C(3)-H(3)	112.3
C(2)-C(3)-H(3)	112.3
C(6)-O(4)-C(3)	110.2(6)
N(1)-C(4)-C(5)	123.4(9)
N(1)-C(4)-C(3)	112.5(7)
C(5)-C(4)-C(3)	124.0(9)
C(4)-C(5)-Br(1)	111.8(7)
C(4)-C(5)-H(5A)	109.3
Br(1)-C(5)-H(5A)	109.3
C(4)-C(5)-H(5B)	109.3
Br(1)-C(5)-H(5B)	109.3
H(5A)-C(5)-H(5B)	107.9
O(4)-C(6)-O(3)	105.9(6)
O(4)-C(6)-C(14)	109.0(11)
O(3)-C(6)-C(14)	109.9(9)
O(4)-C(6)-C(15)	109.9(9)
O(3)-C(6)-C(15)	108.5(9)
C(14)-C(6)-C(15)	113.3(11)
C(8)-C(7)-C(12)	118.8(7)
C(8)-C(7)-C(1)	122.7(8)
C(12)-C(7)-C(1)	118.4(8)
C(7)-C(8)-C(9)	120.6(9)
C(7)-C(8)-H(8)	119.7
C(9)-C(8)-H(8)	119.7
C(10)-C(9)-C(8)	120.1(10)
C(10)-C(9)-H(9)	119.9
C(8)-C(9)-H(9)	119.9
O(2)-C(10)-C(11)	115.4(8)
O(2)-C(10)-C(9)	124.9(9)
C(11)-C(10)-C(9)	119.7(8)
C(10)-C(11)-C(12)	120.5(9)
C(10)-C(11)-H(11)	119.8
C(12)-C(11)-H(11)	119.8
C(11)-C(12)-C(7)	120.3(9)
C(11)-C(12)-H(12)	119.9
C(7)-C(12)-H(12)	119.9
O(2)-C(13)-H(13A)	109.5
O(2)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
O(2)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(6)-C(14)-H(14A)	109.5
C(6)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(6)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(6)-C(15)-H(15A)	109.5
C(6)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(6)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5

Torsion angles [°]

C(4)-N(1)-C(1)-C(7)	-112.5(9)
O(1)-N(1)-C(1)-C(7)	69.3(9)
C(4)-N(1)-C(1)-C(2)	10.2(9)
O(1)-N(1)-C(1)-C(2)	-168.1(7)
N(1)-C(1)-C(2)-O(3)	94.3(7)
C(7)-C(1)-C(2)-O(3)	-147.6(6)
N(1)-C(1)-C(2)-C(3)	-15.2(8)
C(7)-C(1)-C(2)-C(3)	102.9(8)
C(1)-C(2)-O(3)-C(6)	-141.9(7)
C(3)-C(2)-O(3)-C(6)	-29.6(9)
O(3)-C(2)-C(3)-O(4)	19.1(10)
C(1)-C(2)-C(3)-O(4)	132.3(8)
O(3)-C(2)-C(3)-C(4)	-97.9(8)
C(1)-C(2)-C(3)-C(4)	15.2(9)
C(4)-C(3)-O(4)-C(6)	108.9(9)
C(2)-C(3)-O(4)-C(6)	-1.5(12)
O(1)-N(1)-C(4)-C(5)	-4.7(14)
C(1)-N(1)-C(4)-C(5)	177.2(9)
O(1)-N(1)-C(4)-C(3)	177.7(8)
C(1)-N(1)-C(4)-C(3)	-0.4(10)
O(4)-C(3)-C(4)-N(1)	-121.4(8)
C(2)-C(3)-C(4)-N(1)	-9.5(10)
O(4)-C(3)-C(4)-C(5)	61.0(12)
C(2)-C(3)-C(4)-C(5)	172.9(9)
N(1)-C(4)-C(5)-Br(1)	-111.6(9)
C(3)-C(4)-C(5)-Br(1)	65.7(12)
C(3)-O(4)-C(6)-O(3)	-16.8(12)
C(3)-O(4)-C(6)-C(14)	101.4(10)
C(3)-O(4)-C(6)-C(15)	-133.8(10)
C(2)-O(3)-C(6)-O(4)	29.9(10)
C(2)-O(3)-C(6)-C(14)	-87.7(11)
C(2)-O(3)-C(6)-C(15)	147.9(10)
N(1)-C(1)-C(7)-C(8)	91.1(9)
C(2)-C(1)-C(7)-C(8)	-23.0(11)
N(1)-C(1)-C(7)-C(12)	-88.2(9)
C(2)-C(1)-C(7)-C(12)	157.7(7)

C(12)-C(7)-C(8)-C(9)	-1.2(12)	O(2)-C(10)-C(11)-C(12)	179.1(8)
C(1)-C(7)-C(8)-C(9)	179.5(8)	C(9)-C(10)-C(11)-C(12)	-0.5(13)
C(7)-C(8)-C(9)-C(10)	0.3(13)	C(10)-C(11)-C(12)-C(7)	-0.4(13)
C(13)-O(2)-C(10)-C(11)	-173.3(8)	C(8)-C(7)-C(12)-C(11)	1.2(12)
C(13)-O(2)-C(10)-C(9)	6.3(13)	C(1)-C(7)-C(12)-C(11)	-179.4(8)
C(8)-C(9)-C(10)-O(2)	-179.0(8)		
C(8)-C(9)-C(10)-C(11)	0.5(13)		

9.9 (1*R*,2*S*,4a*S*,8a*S*,9a*R*)-1,2-Dihydroxy-*O*-1,2-isopropylidene-octahydro-4-oxa-3a-aza-cyclopenta[b]napthalene-9a-carbonitrile (67)

$C_{15}H_{22}N_2O_3$

orthorhombic, $P2_12_12_1$

a = 6.3199(8) Å

b = 11.4810(17) Å

c = 20.832(3) Å

α = 90 °

β = 90 °

γ = 90 °

V = 1511.6(3) Å^3

Z = 4, $R(F)$ = 0.0654

$R_w(F^2)$ = 0.1114

Crystal size: 0.45 x 0.30 x 0.25 mm

Calculated density: 1.223 g/cm^3

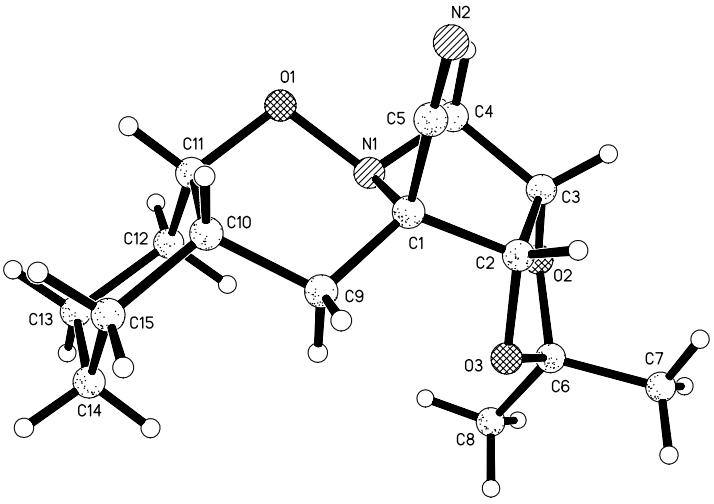
2θ-Range for data collection: 1.96 – 24.99 °

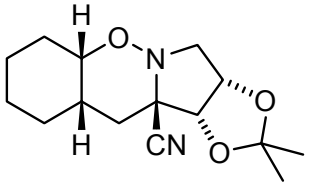
Independent reflections: 3056

Observed reflections: 2667

Contributed reflections to refinement: 3056

Refined parameters: 182

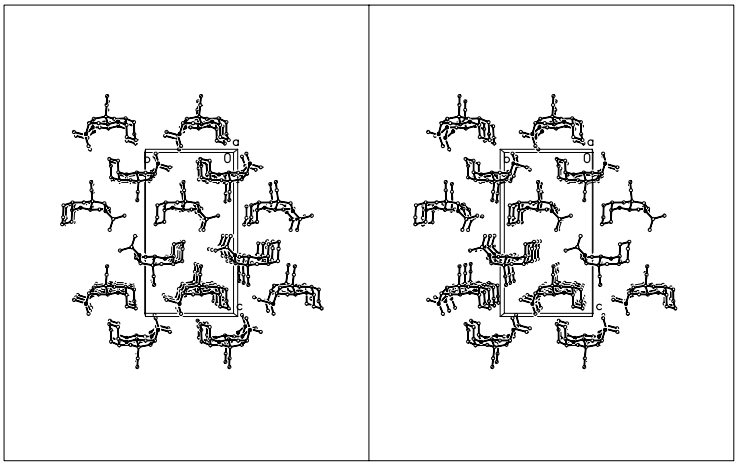




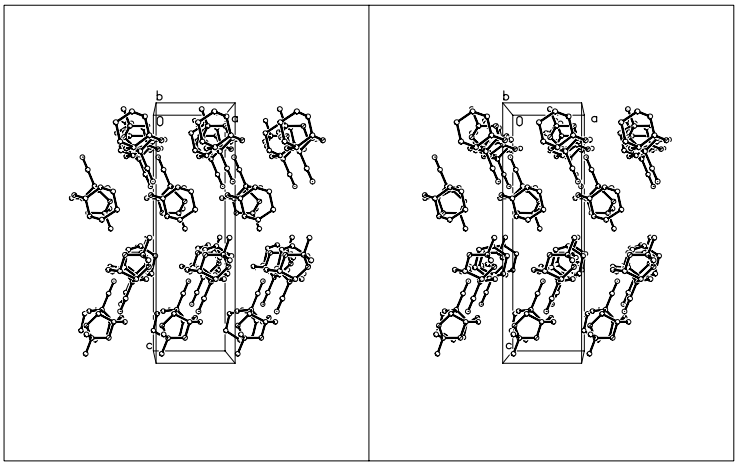
67

Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

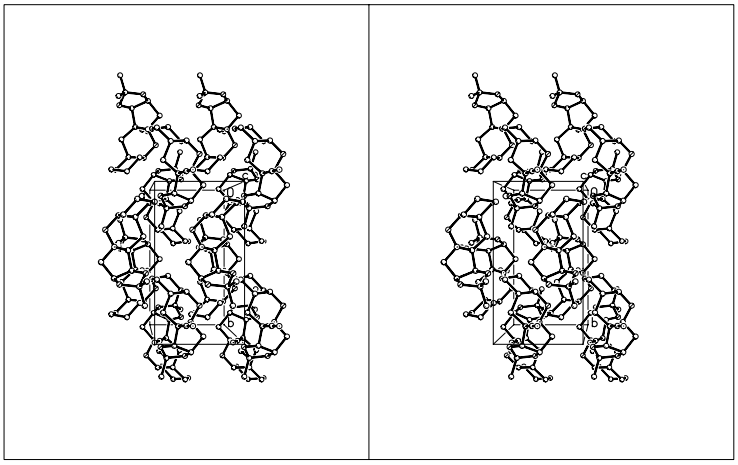
(a)



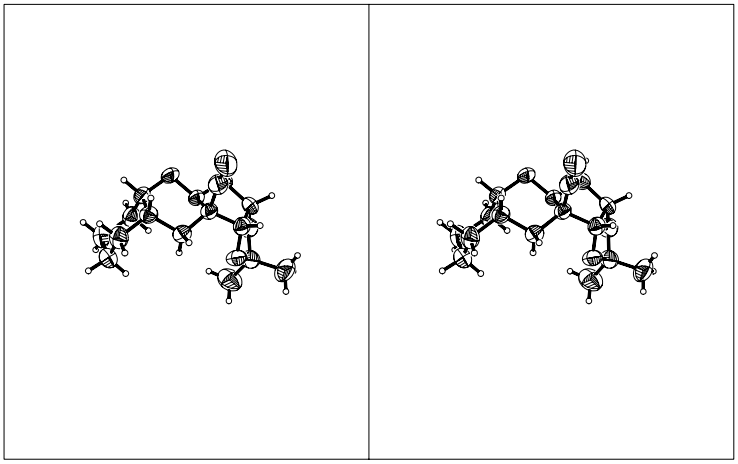
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

N(1)-O(1)	1.441(4)	C(13)-C(14)	1.497(5)
N(1)-C(4)	1.457(5)	C(13)-H(13A)	0.9700
N(1)-C(1)	1.459(5)	C(13)-H(13B)	0.9700
O(1)-C(11)	1.447(5)	C(14)-C(15)	1.511(6)
C(1)-C(5)	1.491(6)	C(14)-H(14A)	0.9700
C(1)-C(9)	1.514(5)	C(14)-H(14B)	0.9700
C(1)-C(2)	1.543(5)	C(15)-H(15A)	0.9700
O(2)-C(3)	1.409(5)	C(15)-H(15B)	0.9700
O(2)-C(6)	1.425(5)	O(1)-N(1)-C(4)	110.5(3)
C(2)-O(3)	1.403(5)	O(1)-N(1)-C(1)	109.1(3)
C(2)-C(3)	1.539(5)	C(4)-N(1)-C(1)	106.7(3)
C(2)-H(2)	0.9800	N(1)-O(1)-C(11)	106.5(3)
N(2)-C(5)	1.135(5)	N(1)-C(1)-C(5)	112.6(4)
O(3)-C(6)	1.425(5)	N(1)-C(1)-C(9)	107.6(3)
C(3)-C(4)	1.516(5)	C(5)-C(1)-C(9)	109.6(3)
C(3)-H(3)	0.9800	N(1)-C(1)-C(2)	99.7(3)
C(4)-H(4A)	0.9700	C(5)-C(1)-C(2)	107.3(3)
C(4)-H(4B)	0.9700	C(9)-C(1)-C(2)	119.8(3)
C(6)-C(8)	1.493(6)	C(3)-O(2)-C(6)	108.6(3)
C(6)-C(7)	1.506(6)	O(3)-C(2)-C(3)	105.4(3)
C(7)-H(7A)	0.9600	O(3)-C(2)-C(1)	110.5(3)
C(7)-H(7B)	0.9600	C(3)-C(2)-C(1)	105.1(3)
C(7)-H(7C)	0.9600	O(3)-C(2)-H(2)	111.8
C(8)-H(8A)	0.9600	C(3)-C(2)-H(2)	111.8
C(8)-H(8B)	0.9600	C(1)-C(2)-H(2)	111.8
C(8)-H(8C)	0.9600	C(2)-O(3)-C(6)	108.1(3)
C(9)-C(10)	1.535(5)	O(2)-C(3)-C(4)	110.5(4)
C(9)-H(9A)	0.9700	O(2)-C(3)-C(2)	104.1(3)
C(9)-H(9B)	0.9700	C(4)-C(3)-C(2)	105.2(3)
C(10)-C(15)	1.524(5)	O(2)-C(3)-H(3)	112.2
C(10)-C(11)	1.539(5)	C(4)-C(3)-H(3)	112.2
C(10)-H(10)	0.9800	C(2)-C(3)-H(3)	112.2
C(11)-C(12)	1.501(5)	N(1)-C(4)-C(3)	100.8(3)
C(11)-H(11)	0.9800	N(1)-C(4)-H(4A)	111.6
C(12)-C(13)	1.525(6)	C(3)-C(4)-H(4A)	111.6
C(12)-H(12A)	0.9700	N(1)-C(4)-H(4B)	111.6
C(12)-H(12B)	0.9700	C(3)-C(4)-H(4B)	111.6

H(4A)-C(4)-H(4B)	109.4
N(2)-C(5)-C(1)	178.5(6)
O(2)-C(6)-O(3)	104.6(3)
O(2)-C(6)-C(8)	108.6(4)
O(3)-C(6)-C(8)	108.4(4)
O(2)-C(6)-C(7)	111.2(4)
O(3)-C(6)-C(7)	109.7(4)
C(8)-C(6)-C(7)	113.9(4)
C(6)-C(7)-H(7A)	109.5
C(6)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
C(6)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(6)-C(8)-H(8A)	109.5
C(6)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(6)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(1)-C(9)-C(10)	110.1(3)
C(1)-C(9)-H(9A)	109.6
C(10)-C(9)-H(9A)	109.6
C(1)-C(9)-H(9B)	109.6
C(10)-C(9)-H(9B)	109.6
H(9A)-C(9)-H(9B)	108.2
C(15)-C(10)-C(9)	112.4(3)
C(15)-C(10)-C(11)	110.4(3)
C(9)-C(10)-C(11)	111.2(3)
C(15)-C(10)-H(10)	107.6
C(9)-C(10)-H(10)	107.6
C(11)-C(10)-H(10)	107.6
O(1)-C(11)-C(12)	112.6(3)
O(1)-C(11)-C(10)	110.3(3)
C(12)-C(11)-C(10)	113.5(3)
O(1)-C(11)-H(11)	106.7
C(12)-C(11)-H(11)	106.7
C(10)-C(11)-H(11)	106.7
C(11)-C(12)-C(13)	110.9(4)
C(11)-C(12)-H(12A)	109.5
C(13)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
C(13)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	108.0
C(14)-C(13)-C(12)	110.6(3)
C(14)-C(13)-H(13A)	109.5
C(12)-C(13)-H(13A)	109.5
C(14)-C(13)-H(13B)	109.5
C(12)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	108.1
C(13)-C(14)-C(15)	111.2(4)
C(13)-C(14)-H(14A)	109.4
C(15)-C(14)-H(14A)	109.4
C(13)-C(14)-H(14B)	109.4
C(15)-C(14)-H(14B)	109.4
H(14A)-C(14)-H(14B)	108.0
C(14)-C(15)-C(10)	113.6(3)
C(14)-C(15)-H(15A)	108.9
C(10)-C(15)-H(15A)	108.9
C(14)-C(15)-H(15B)	108.9
C(10)-C(15)-H(15B)	108.9
H(15A)-C(15)-H(15B)	107.7

Torsion angles [°]

C(4)-N(1)-O(1)-C(11)	170.0(3)
C(1)-N(1)-O(1)-C(11)	-73.0(4)
O(1)-N(1)-C(1)-C(5)	-52.2(4)
C(4)-N(1)-C(1)-C(5)	67.2(4)
O(1)-N(1)-C(1)-C(9)	68.7(3)
C(4)-N(1)-C(1)-C(9)	-171.9(3)
O(1)-N(1)-C(1)-C(2)	-165.6(3)
C(4)-N(1)-C(1)-C(2)	-46.2(4)
N(1)-C(1)-C(2)-O(3)	-86.9(3)
C(5)-C(1)-C(2)-O(3)	155.6(4)
C(9)-C(1)-C(2)-O(3)	30.0(5)
N(1)-C(1)-C(2)-C(3)	26.4(4)
C(5)-C(1)-C(2)-C(3)	-91.1(4)
C(9)-C(1)-C(2)-C(3)	143.3(4)
C(3)-C(2)-O(3)-C(6)	17.6(4)
C(1)-C(2)-O(3)-C(6)	130.7(4)
C(6)-O(2)-C(3)-C(4)	-131.5(4)
C(6)-O(2)-C(3)-C(2)	-19.1(4)
O(3)-C(2)-C(3)-O(2)	0.9(4)
C(1)-C(2)-C(3)-O(2)	-116.0(3)
O(3)-C(2)-C(3)-C(4)	117.1(3)
C(1)-C(2)-C(3)-C(4)	0.3(4)
O(1)-N(1)-C(4)-C(3)	165.5(3)
C(1)-N(1)-C(4)-C(3)	47.0(4)
O(2)-C(3)-C(4)-N(1)	84.7(4)
C(2)-C(3)-C(4)-N(1)	-27.0(4)
N(1)-C(1)-C(5)-N(2)	-80(25)
C(9)-C(1)-C(5)-N(2)	161(25)
C(2)-C(1)-C(5)-N(2)	29(25)
C(3)-O(2)-C(6)-O(3)	30.2(5)
C(3)-O(2)-C(6)-C(8)	145.8(4)
C(3)-O(2)-C(6)-C(7)	-88.1(5)
C(2)-O(3)-C(6)-O(2)	-29.4(5)
C(2)-O(3)-C(6)-C(8)	-145.2(4)
C(2)-O(3)-C(6)-C(7)	89.9(4)
N(1)-C(1)-C(9)-C(10)	-54.9(4)
C(5)-C(1)-C(9)-C(10)	67.9(4)
C(2)-C(1)-C(9)-C(10)	-167.6(3)
C(1)-C(9)-C(10)-C(15)	171.7(3)
C(1)-C(9)-C(10)-C(11)	47.4(4)
N(1)-O(1)-C(11)-C(12)	-65.1(4)
N(1)-O(1)-C(11)-C(10)	62.8(4)
C(15)-C(10)-C(11)-O(1)	-177.3(3)
C(9)-C(10)-C(11)-O(1)	-51.9(4)
C(15)-C(10)-C(11)-C(12)	-49.9(5)
C(9)-C(10)-C(11)-C(12)	75.5(4)
O(1)-C(11)-C(12)-C(13)	-179.6(3)
C(10)-C(11)-C(12)-C(13)	54.3(5)
C(11)-C(12)-C(13)-C(14)	-57.5(5)
C(12)-C(13)-C(14)-C(15)	57.5(5)
C(13)-C(14)-C(15)-C(10)	-54.8(5)
C(9)-C(10)-C(15)-C(14)	-75.0(5)
C(11)-C(10)-C(15)-C(14)	49.7(5

9.10 (2*S*,3*S*,4*R*,5*S*)-1,3,4-Trihydroxy-2-isopropyl-3,4-*O*-isopropylidene-5-methyl-pyrrolidine (69)

$C_{11}H_{21}NO_3$

orthorhombic, $P2_12_12_1$

a = 8.0719(15) Å

b = 10.825(3) Å

c = 28.383(5) Å

α = 90 °

β = 90 °

γ = 90 °

V = 2480.0(9) Å^3

Z = 8, $R(F)$ = 0.0518

$R_w(F^2)$ = 0.1210

Crystal size: 1.2 x 1.0 x 0.7 mm

Calculated density: 1.153 g/cm^3

2θ-Range for data collection: 2.
27.99 °

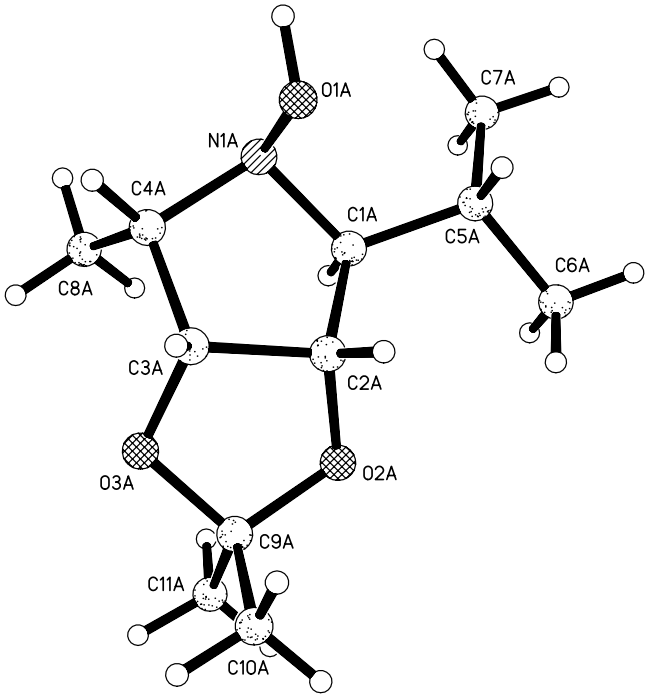
Independent reflections: 4128

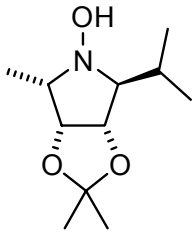
Observed Reflections: 4336

Contributed reflections to refinement

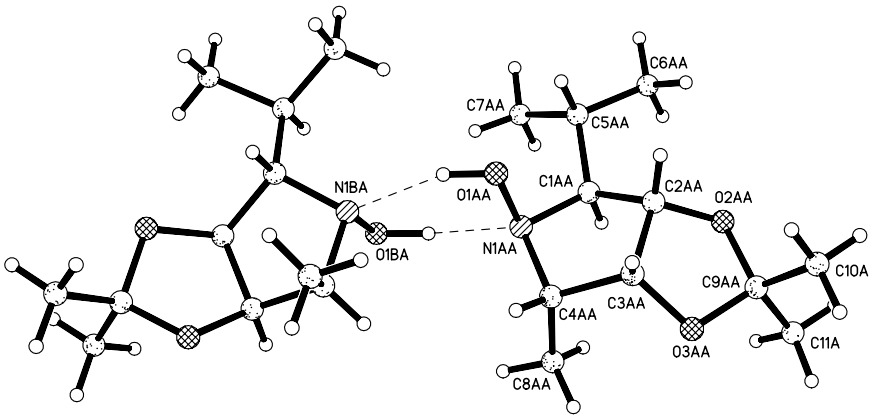
Herangezogenen Reflexe: 4128

Refined parameters: 280



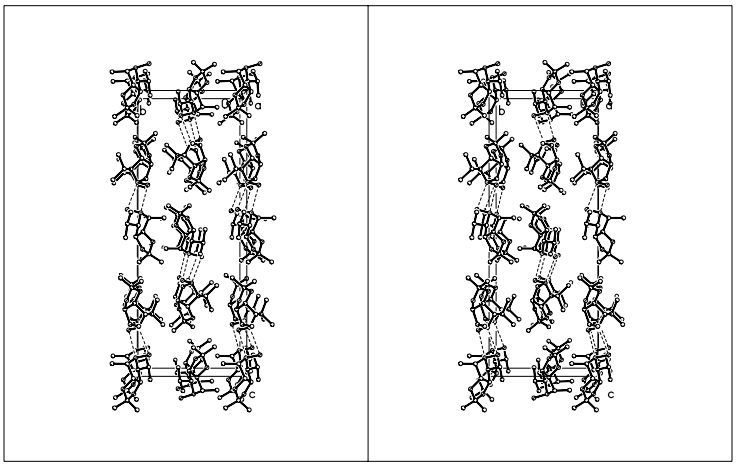


69

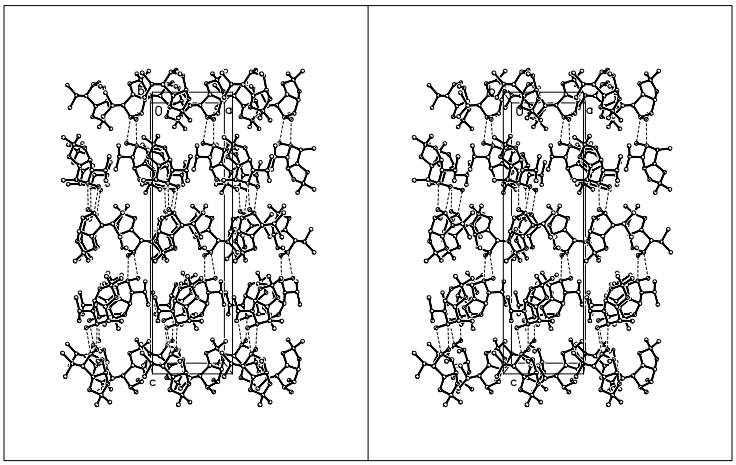


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

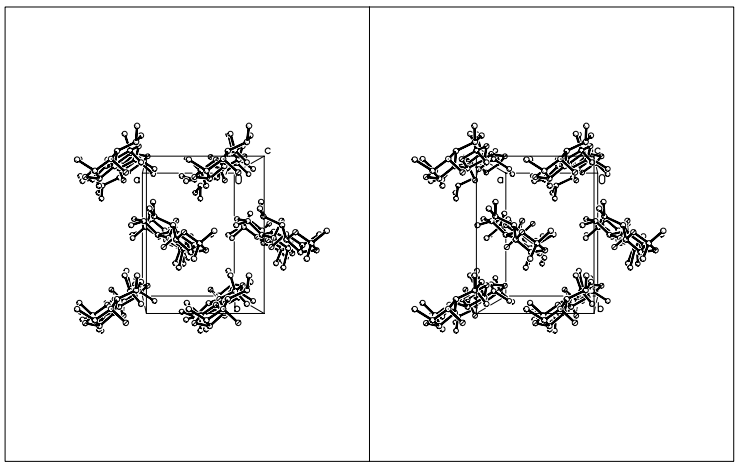
(a)



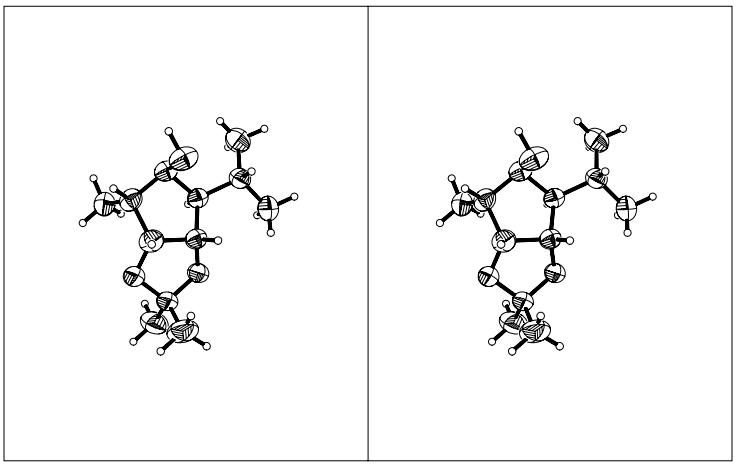
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

N(1A)-O(1A)	1.474(3)	N(1B)-O(1B)	1.470(3)
N(1A)-C(1A)	1.491(3)	N(1B)-C(4B)	1.488(4)
N(1A)-C(4A)	1.499(3)	N(1B)-C(1B)	1.498(3)
O(1A)-H(1AA)	0.89(3)	O(1B)-H(1BB)	0.93(3)
C(1A)-C(5A)	1.530(4)	C(1B)-C(5B)	1.530(4)
C(1A)-C(2A)	1.534(3)	C(1B)-C(2B)	1.536(3)
C(1A)-H(1A)	0.98	C(1B)-H(1B)	0.98
O(2A)-C(2A)	1.422(3)	O(2B)-C(9B)	1.421(3)
O(2A)-C(9A)	1.422(3)	O(2B)-C(2B)	1.425(3)
C(2A)-C(3A)	1.538(4)	C(2B)-C(3B)	1.547(4)
C(2A)-H(2A)	0.98	C(2B)-H(2B)	0.98
O(3A)-C(3A)	1.417(3)	O(3B)-C(9B)	1.419(3)
O(3A)-C(9A)	1.420(3)	O(3B)-C(3B)	1.424(4)
C(3A)-C(4A)	1.547(4)	C(3B)-C(4B)	1.539(4)
C(3A)-H(3A)	0.98	C(3B)-H(3B)	0.98
C(4A)-C(8A)	1.517(4)	C(4B)-C(8B)	1.524(4)
C(4A)-H(4A)	0.98	C(4B)-H(4B)	0.98
C(5A)-C(6A)	1.519(4)	C(5B)-C(7B)	1.518(4)
C(5A)-C(7A)	1.528(4)	C(5B)-C(6B)	1.526(4)
C(5A)-H(5A)	0.98	C(5B)-H(5B)	0.98
C(6A)-H(6A1)	0.96	C(6B)-H(6B1)	0.96
C(6A)-H(6A2)	0.96	C(6B)-H(6B2)	0.96
C(6A)-H(6A3)	0.96	C(6B)-H(6B3)	0.96
C(7A)-H(7A1)	0.96	C(7B)-H(7B1)	0.96
C(7A)-H(7A2)	0.96	C(7B)-H(7B2)	0.96
C(7A)-H(7A3)	0.96	C(7B)-H(7B3)	0.96
C(8A)-H(8A1)	0.96	C(8B)-H(8B1)	0.96
C(8A)-H(8A2)	0.96	C(8B)-H(8B2)	0.96
C(8A)-H(8A3)	0.96	C(8B)-H(8B3)	0.96
C(9A)-C(11A)	1.507(5)	C(9B)-C(10B)	1.511(5)
C(9A)-C(10A)	1.508(4)	C(9B)-C(11B)	1.512(5)
C(10A)-H(10A)	0.96	C(10B)-H(10D)	0.96
C(10A)-H(10B)	0.96	C(10B)-H(10E)	0.96
C(10A)-H(10C)	0.96	C(10B)-H(10F)	0.96
C(11A)-H(11A)	0.96	C(11B)-H(11D)	0.96
C(11A)-H(11B)	0.96	C(11B)-H(11E)	0.96
C(11A)-H(11C)	0.96	C(11B)-H(11F)	0.96

O(1A)-N(1A)-C(1A)	105.36(18)	C(9A)-C(10A)-H(10A)	109.5
O(1A)-N(1A)-C(4A)	104.55(18)	C(9A)-C(10A)-H(10B)	109.5
C(1A)-N(1A)-C(4A)	104.17(19)	H(10A)-C(10A)-H(10B)	109.5
N(1A)-O(1A)-H(1AA)	99(2)	C(9A)-C(10A)-H(10C)	109.5
N(1A)-C(1A)-C(5A)	114.6(2)	H(10A)-C(10A)-H(10C)	109.5
N(1A)-C(1A)-C(2A)	104.98(19)	H(10B)-C(10A)-H(10C)	109.5
C(5A)-C(1A)-C(2A)	115.5(2)	C(9A)-C(11A)-H(11A)	109.5
N(1A)-C(1A)-H(1A)	107.1	C(9A)-C(11A)-H(11B)	109.5
C(5A)-C(1A)-H(1A)	107.1	H(11A)-C(11A)-H(11B)	109.5
C(2A)-C(1A)-H(1A)	107.1	C(9A)-C(11A)-H(11C)	109.5
C(2A)-O(2A)-C(9A)	108.07(19)	H(11A)-C(11A)-H(11C)	109.5
O(2A)-C(2A)-C(1A)	112.3(2)	H(11B)-C(11A)-H(11C)	109.5
O(2A)-C(2A)-C(3A)	104.9(2)	O(1B)-N(1B)-C(4B)	105.2(2)
C(1A)-C(2A)-C(3A)	105.64(19)	O(1B)-N(1B)-C(1B)	104.87(18)
O(2A)-C(2A)-H(2A)	111.2	C(4B)-N(1B)-C(1B)	103.98(18)
C(1A)-C(2A)-H(2A)	111.2	N(1B)-O(1B)-H(1BB)	97.7(19)
C(3A)-C(2A)-H(2A)	111.2	N(1B)-C(1B)-C(5B)	114.5(2)
C(3A)-O(3A)-C(9A)	108.1(2)	N(1B)-C(1B)-C(2B)	104.7(2)
O(3A)-C(3A)-C(2A)	104.32(19)	C(5B)-C(1B)-C(2B)	116.0(2)
O(3A)-C(3A)-C(4A)	113.7(2)	N(1B)-C(1B)-H(1B)	107.0
C(2A)-C(3A)-C(4A)	105.2(2)	C(5B)-C(1B)-H(1B)	107.0
O(3A)-C(3A)-H(3A)	111.1	C(2B)-C(1B)-H(1B)	107.0
C(2A)-C(3A)-H(3A)	111.1	C(9B)-O(2B)-C(2B)	107.1(2)
C(4A)-C(3A)-H(3A)	111.1	O(2B)-C(2B)-C(1B)	112.4(2)
N(1A)-C(4A)-C(8A)	110.4(2)	O(2B)-C(2B)-C(3B)	104.6(2)
N(1A)-C(4A)-C(3A)	105.5(2)	C(1B)-C(2B)-C(3B)	105.22(19)
C(8A)-C(4A)-C(3A)	114.1(2)	O(2B)-C(2B)-H(2B)	111.4
N(1A)-C(4A)-H(4A)	108.9	C(1B)-C(2B)-H(2B)	111.4
C(8A)-C(4A)-H(4A)	108.9	C(3B)-C(2B)-H(2B)	111.4
C(3A)-C(4A)-H(4A)	108.9	C(9B)-O(3B)-C(3B)	107.5(2)
C(6A)-C(5A)-C(7A)	110.0(3)	O(3B)-C(3B)-C(4B)	113.6(3)
C(6A)-C(5A)-C(1A)	110.4(2)	O(3B)-C(3B)-C(2B)	103.9(2)
C(7A)-C(5A)-C(1A)	111.4(2)	C(4B)-C(3B)-C(2B)	105.1(2)
C(6A)-C(5A)-H(5A)	108.3	O(3B)-C(3B)-H(3B)	111.3
C(7A)-C(5A)-H(5A)	108.3	C(4B)-C(3B)-H(3B)	111.3
C(1A)-C(5A)-H(5A)	108.3	C(2B)-C(3B)-H(3B)	111.3
C(5A)-C(6A)-H(6A1)	109.5	N(1B)-C(4B)-C(8B)	110.6(2)
C(5A)-C(6A)-H(6A2)	109.5	N(1B)-C(4B)-C(3B)	105.2(2)
H(6A1)-C(6A)-H(6A2)	109.5	C(8B)-C(4B)-C(3B)	114.4(3)
C(5A)-C(6A)-H(6A3)	109.5	N(1B)-C(4B)-H(4B)	108.8
H(6A1)-C(6A)-H(6A3)	109.5	C(8B)-C(4B)-H(4B)	108.8
H(6A2)-C(6A)-H(6A3)	109.5	C(3B)-C(4B)-H(4B)	108.8
C(5A)-C(7A)-H(7A1)	109.5	C(7B)-C(5B)-C(6B)	110.0(3)
C(5A)-C(7A)-H(7A2)	109.5	C(7B)-C(5B)-C(1B)	112.4(2)
H(7A1)-C(7A)-H(7A2)	109.5	C(6B)-C(5B)-C(1B)	109.5(2)
C(5A)-C(7A)-H(7A3)	109.5	C(7B)-C(5B)-H(5B)	108.3
H(7A1)-C(7A)-H(7A3)	109.5	C(6B)-C(5B)-H(5B)	108.3
H(7A2)-C(7A)-H(7A3)	109.5	C(1B)-C(5B)-H(5B)	108.3
C(4A)-C(8A)-H(8A1)	109.5	C(5B)-C(6B)-H(6B1)	109.5
C(4A)-C(8A)-H(8A2)	109.5	C(5B)-C(6B)-H(6B2)	109.5
H(8A1)-C(8A)-H(8A2)	109.5	H(6B1)-C(6B)-H(6B2)	109.5
C(4A)-C(8A)-H(8A3)	109.5	C(5B)-C(6B)-H(6B3)	109.5
H(8A1)-C(8A)-H(8A3)	109.5	H(6B1)-C(6B)-H(6B3)	109.5
H(8A2)-C(8A)-H(8A3)	109.5	H(6B2)-C(6B)-H(6B3)	109.5
O(3A)-C(9A)-O(2A)	105.1(2)	C(5B)-C(7B)-H(7B1)	109.5
O(3A)-C(9A)-C(11A)	109.9(3)	C(5B)-C(7B)-H(7B2)	109.5
O(2A)-C(9A)-C(11A)	108.1(3)	H(7B1)-C(7B)-H(7B2)	109.5
O(3A)-C(9A)-C(10A)	111.4(3)	C(5B)-C(7B)-H(7B3)	109.5
O(2A)-C(9A)-C(10A)	110.5(3)	H(7B1)-C(7B)-H(7B3)	109.5
C(11A)-C(9A)-C(10A)	111.7(3)	H(7B2)-C(7B)-H(7B3)	109.5

C(4B)-C(8B)-H(8B1)	109.5
C(4B)-C(8B)-H(8B2)	109.5
H(8B1)-C(8B)-H(8B2)	109.5
C(4B)-C(8B)-H(8B3)	109.5
H(8B1)-C(8B)-H(8B3)	109.5
H(8B2)-C(8B)-H(8B3)	109.5
O(3B)-C(9B)-O(2B)	104.6(2)
O(3B)-C(9B)-C(10B)	111.2(3)
O(2B)-C(9B)-C(10B)	110.5(3)
O(3B)-C(9B)-C(11B)	109.5(3)
O(2B)-C(9B)-C(11B)	108.3(3)
C(10B)-C(9B)-C(11B)	112.4(3)
C(9B)-C(10B)-H(10D)	109.5
C(9B)-C(10B)-H(10E)	109.5
H(10D)-C(10B)-H(10E)	109.5
C(9B)-C(10B)-H(10F)	109.5
H(10D)-C(10B)-H(10F)	109.5
H(10E)-C(10B)-H(10F)	109.5
C(9B)-C(11B)-H(11D)	109.5
C(9B)-C(11B)-H(11E)	109.5
H(11D)-C(11B)-H(11E)	109.5
C(9B)-C(11B)-H(11F)	109.5
H(11D)-C(11B)-H(11F)	109.5
H(11E)-C(11B)-H(11F)	109.5

Torsion angles [°]

O(1A)-N(1A)-C(1A)-C(5A)	-56.2(3)
C(4A)-N(1A)-C(1A)-C(5A)	-166.0(2)
O(1A)-N(1A)-C(1A)-C(2A)	71.6(2)
C(4A)-N(1A)-C(1A)-C(2A)	-38.1(2)
C(9A)-O(2A)-C(2A)-C(1A)	-130.0(2)
C(9A)-O(2A)-C(2A)-C(3A)	-15.8(3)
N(1A)-C(1A)-C(2A)-O(2A)	139.8(2)
C(5A)-C(1A)-C(2A)-O(2A)	-92.9(3)
N(1A)-C(1A)-C(2A)-C(3A)	26.0(3)
C(5A)-C(1A)-C(2A)-C(3A)	153.3(2)
C(9A)-O(3A)-C(3A)-C(2A)	21.0(3)
C(9A)-O(3A)-C(3A)-C(4A)	135.0(2)
O(2A)-C(2A)-C(3A)-O(3A)	-3.2(3)
C(1A)-C(2A)-C(3A)-O(3A)	115.6(2)
O(2A)-C(2A)-C(3A)-C(4A)	-123.0(2)
C(1A)-C(2A)-C(3A)-C(4A)	-4.2(3)
O(1A)-N(1A)-C(4A)-C(8A)	161.4(2)
C(1A)-N(1A)-C(4A)-C(8A)	-88.3(2)
O(1A)-N(1A)-C(4A)-C(3)	-74.9(2)
C(1A)-N(1A)-C(4A)-C(3A)	35.4(2)
O(3A)-C(3A)-C(4A)-N(1A)	-132.4(2)
C(2A)-C(3A)-C(4A)-N(1A)	-18.9(3)
O(3A)-C(3A)-C(4A)-C(8A)	-11.1(3)
C(2A)-C(3A)-C(4A)-C(8A)	102.4(3)
N(1A)-C(1A)-C(5A)-C(6A)	176.4(3)
C(2A)-C(1A)-C(5A)-C(6A)	54.2(3)
N(1A)-C(1A)-C(5A)-C(7A)	-61.1(3)
C(2A)-C(1A)-C(5A)-C(7A)	176.7(2)
C(3A)-O(3A)-C(9A)-O(2A)	-31.3(3)
C(3A)-O(3A)-C(9A)-C(11A)	-147.3(3)
C(3A)-O(3A)-C(9A)-C(10A)	88.3(3)
C(2A)-O(2A)-C(9A)-O(3A)	29.0(3)
C(2A)-O(2A)-C(9A)-C(11A)	146.2(3)
C(2A)-O(2A)-C(9A)-C(10A)	-91.3(3)
O(1B)-N(1B)-C(1B)-C(5B)	-56.9(3)
C(4B)-N(1B)-C(1B)-C(5B)	-167.2(2)
O(1B)-N(1B)-C(1B)-C(2B)	71.2(2)
C(4B)-N(1B)-C(1B)-C(2B)	-39.0(2)
C(9B)-O(2B)-C(2B)-C(1B)	-134.0(2)
C(9B)-O(2B)-C(2B)-C(3B)	-20.4(3)
N(1B)-C(1B)-C(2B)-O(2B)	137.9(2)
C(5B)-C(1B)-C(2B)-O(2B)	-94.9(3)
N(1B)-C(1B)-C(2B)-C(3B)	24.6(3)
C(5B)-C(1B)-C(2B)-C(3B)	151.9(2)
C(9B)-O(3B)-C(3B)-C(4B)	135.2(3)
C(9B)-O(3B)-C(3B)-C(2B)	21.5(3)
O(2B)-C(2B)-C(3B)-O(3B)	-0.6(3)
C(1B)-C(2B)-C(3B)-O(3B)	118.0(2)
O(2B)-C(2B)-C(3B)-C(4B)	-120.2(2)
C(1B)-C(2B)-C(3B)-C(4B)	-1.6(3)
O(1B)-N(1B)-C(4B)-C(8B)	164.0(2)
C(1B)-N(1B)-C(4B)-C(8B)	-86.0(3)
O(1B)-N(1B)-C(4B)-C(3B)	-71.9(2)
C(1B)-N(1B)-C(4B)-C(3B)	38.1(3)
O(3B)-C(3B)-C(4B)-N(1B)	-135.1(2)
C(2B)-C(3B)-C(4B)-N(1B)	-22.2(3)
O(3B)-C(3B)-C(4B)-C(8B)	-13.5(4)
C(2B)-C(3B)-C(4B)-C(8B)	99.4(3)
N(1B)-C(1B)-C(5B)-C(7B)	-58.3(3)
C(2B)-C(1B)-C(5B)-C(7B)	179.5(2)
N(1B)-C(1B)-C(5B)-C(6B)	179.1(3)
C(2B)-C(1B)-C(5B)-C(6B)	57.0(3)
C(3B)-O(3B)-C(9B)-O(2B)	-34.9(3)
C(3B)-O(3B)-C(9B)-C(10B)	84.4(3)
C(3B)-O(3B)-C(9B)-C(11B)	-150.8(3)
C(2B)-O(2B)-C(9B)-O(3B)	34.2(3)
C(2B)-O(2B)-C(9B)-C(10B)	-85.5(3)
C(2B)-O(2B)-C(9B)-C(11B)	151.0(2)

H-Bond lengths[Å] and angles [°]

O1A-N1B_$1	2.8187(0.0028)
H1AA-N1B_$1	1.9725(0.0304)
O1B-N1A_$2	2.8556(0.0029)
H1BB-N1A_$2	1.9618(0.0322)
O1A-H1AA-N1B_$1	159.21(2.91)
O1B-H1BB-N1A_$2	160.45(2.77)

9.11 (2*R*,3*S*,4*R*)-3,4-Dihydroxy-3,4-*O*-isopropylidene-5-methyl-2-phenyl-2*H*-pyrrole-1-oxide (82)

$C_{14}H_{17}NO_3$

monoclinic, *C*2

a = 20.1885(15) Å

b = 6.1650(5) Å

c = 10.7093(8) Å

α = 90 °

β = 93.558(6) °

γ = 90 °

V = 1330.33(18) Å^3

Z = 4, $R(F)$ = 0.0634

$R_w(F^2)$ = 0.1737

Crystal size: 1.3 x 0.2 x 0.07 mm

Calculated density: 1.235 g/cm^3

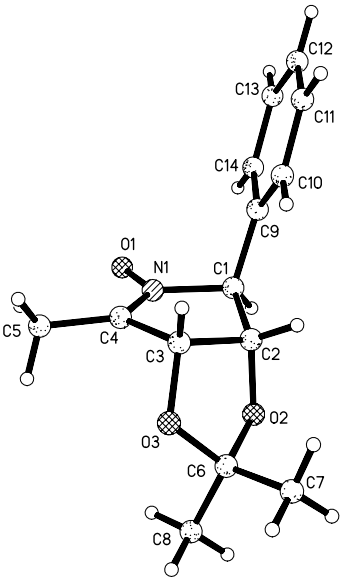
2θ-Range for data collection: 4.14 – 67.97 °

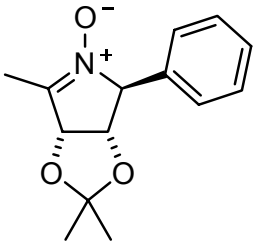
Independent reflections: 1523

Observed reflections: 1226

Contributed reflections to refinement: 1523

Refined parameters: 164

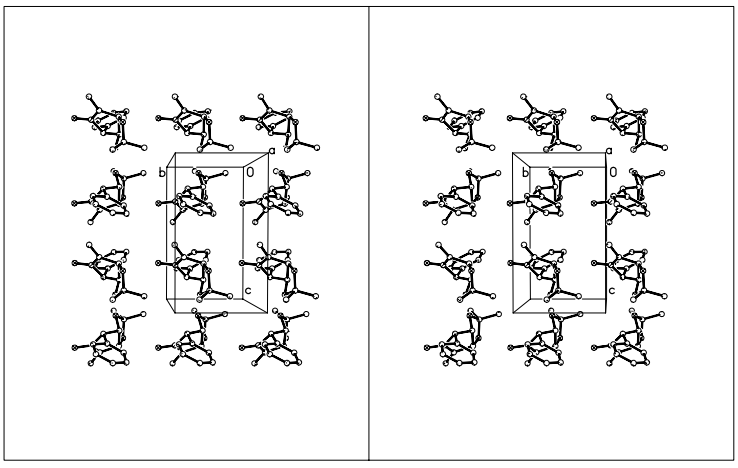




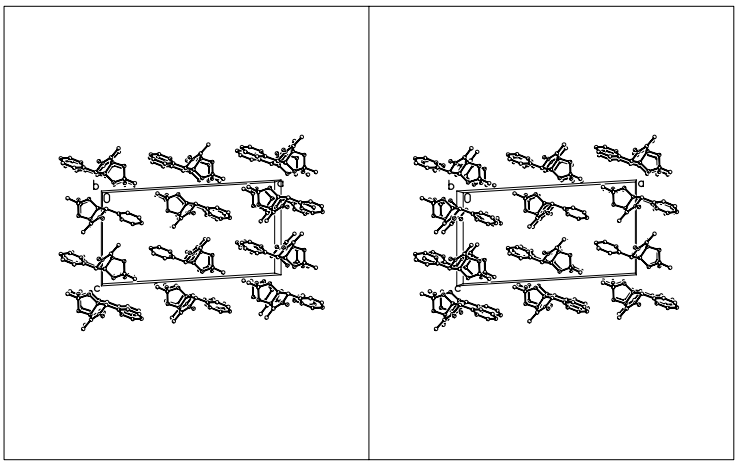
82

Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

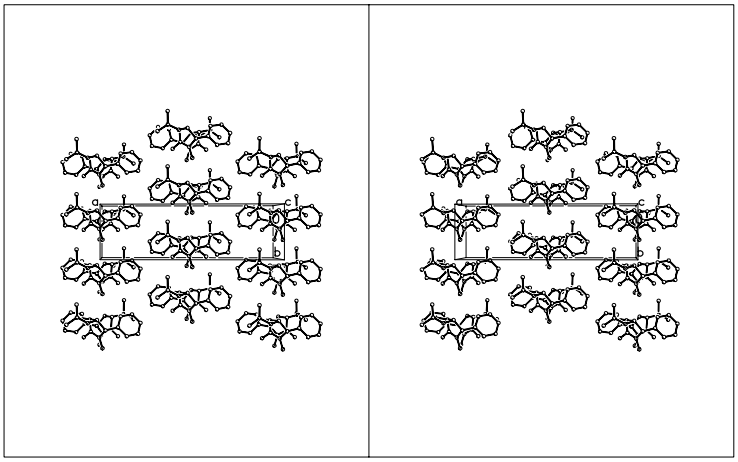
(a)



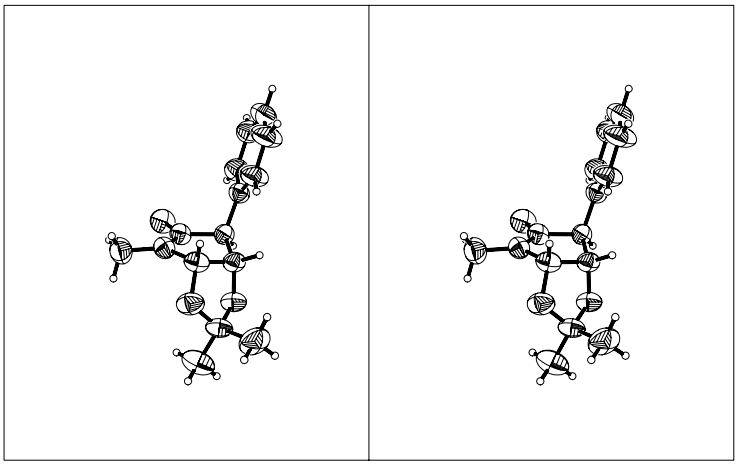
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

N(1)-O(1)	1.296(6)	C(2)-C(3)-H(3)	112.1
N(1)-C(4)	1.302(6)	N(1)-C(4)-C(5)	122.2(5)
N(1)-C(1)	1.497(6)	N(1)-C(4)-C(3)	109.7(4)
C(1)-C(9)	1.507(6)	C(5)-C(4)-C(3)	128.0(5)
C(1)-C(2)	1.510(6)	C(4)-C(5)-H(5A)	109.5
C(1)-H(1)	0.98	C(4)-C(5)-H(5B)	109.5
O(2)-C(6)	1.432(5)	H(5A)-C(5)-H(5B)	109.5
O(2)-C(2)	1.434(5)	C(4)-C(5)-H(5C)	109.5
C(2)-C(3)	1.531(6)	H(5A)-C(5)-H(5C)	109.5
C(2)-H(2)	1.423(5)	H(5B)-C(5)-H(5C)	109.5
O(3)-C(6)	1.428(6)	O(3)-C(6)-O(2)	105.3(3)
C(3)-C(4)	1.502(8)	O(3)-C(6)-C(8)	110.2(5)
C(3)-H(3)	0.98	O(2)-C(6)-C(8)	108.9(5)
C(4)-C(5)	1.457(7)	O(3)-C(6)-C(7)	108.6(5)
C(5)-H(5A)	0.96	O(2)-C(6)-C(7)	110.5(5)
C(5)-H(5B)	0.96	C(8)-C(6)-C(7)	113.0(5)
C(5)-H(5C)	0.96	C(6)-C(7)-H(7A)	109.5
C(6)-C(8)	1.496(8)	C(6)-C(7)-H(7B)	109.5
C(6)-C(7)	1.522(11)	H(7A)-C(7)-H(7B)	109.5
C(7)-H(7A)	0.96	C(6)-C(7)-H(7C)	109.5
C(7)-H(7B)	0.96	H(7A)-C(7)-H(7C)	109.5
C(7)-H(7C)	0.96	H(7B)-C(7)-H(7C)	109.5
C(8)-H(8A)	0.96	C(6)-C(8)-H(8A)	109.5
C(8)-H(8B)	0.96	C(6)-C(8)-H(8B)	109.5
C(8)-H(8C)	0.96	H(8A)-C(8)-H(8B)	109.5
C(9)-C(10)	1.389(9)	C(6)-C(8)-H(8C)	109.5
C(9)-C(14)	1.4(7)	H(8A)-C(8)-H(8C)	109.5
C(10)-C(11)	1.383(7)	H(8B)-C(8)-H(8C)	109.5
C(10)-H(10)	0.93	C(10)-C(9)-C(14)	118.3(5)
C(11)-C(12)	1.362(11)	C(10)-C(9)-C(1)	122.1(5)
C(11)-H(11)	0.93	C(14)-C(9)-C(1)	119.6(5)
C(12)-C(13)	1.368(12)	C(11)-C(10)-C(9)	119.7(7)
C(12)-H(12)	0.93	C(11)-C(10)-H(10)	120.2
C(13)-C(14)	1.394(7)	C(9)-C(10)-H(10)	120.2
C(13)-H(13)	0.93	C(12)-C(11)-C(10)	121.4(8)
C(14)-H(14)	0.93	C(12)-C(11)-H(11)	119.3
O(1)-N(1)-C(4)	126.5(5)	C(10)-C(11)-H(11)	119.3
O(1)-N(1)-C(1)	119.2(4)	C(11)-C(12)-C(13)	120.5(5)
C(4)-N(1)-C(1)	114.3(4)	C(11)-C(12)-H(12)	119.8
N(1)-C(1)-C(9)	109.2(3)	C(13)-C(12)-H(12)	119.8
N(1)-C(1)-C(2)	101.5(3)	C(12)-C(13)-C(14)	119.1(6)
C(9)-C(1)-C(2)	116.9(4)	C(12)-C(13)-H(13)	120.4
N(1)-C(1)-H(1)	109.6	C(14)-C(13)-H(13)	120.4
C(9)-C(1)-H(1)	109.6	C(13)-C(14)-C(9)	121.1(6)
C(2)-C(1)-H(1)	109.6	C(13)-C(14)-H(14)	119.5
C(6)-O(2)-C(2)	106.8(3)	C(9)-C(14)-H(14)	119.5
O(2)-C(2)-C(1)	109.6(4)		
O(2)-C(2)-C(3)	101.4(3)		
C(1)-C(2)-C(3)	105.8(4)	Torsion angles [°]	
O(2)-C(2)-H(2)	113.1		
C(1)-C(2)-H(2)	113.1	O(1)-N(1)-C(1)-C(9)	71.2(5)
C(3)-C(2)-H(2)	113.1	C(4)-N(1)-C(1)-C(9)	-108.1(4)
C(3)-O(3)-C(6)	108.7(3)	O(1)-N(1)-C(1)-C(2)	-164.8(4)
O(3)-C(3)-C(4)	111.1(4)	C(4)-N(1)-C(1)-C(2)	15.9(5)
O(3)-C(3)-C(2)	105.6(3)	C(6)-O(2)-C(2)-C(1)	-146.1(4)
C(4)-C(3)-C(2)	103.3(4)	C(6)-O(2)-C(2)-C(3)	-34.7(5)
O(3)-C(3)-H(3)	112.1	N(1)-C(1)-C(2)-O(2)	86.1(4)
C(4)-C(3)-H(3)	112.1	C(9)-C(1)-C(2)-O(2)	-155.3(4)

N(1)-C(1)-C(2)-C(3)	-22.5(4)	C(3)-O(3)-C(6)-C(7)	102.6(5)
C(9)-C(1)-C(2)-C(3)	96.2(4)	C(2)-O(2)-C(6)-O(3)	32.6(6)
C(6)-O(3)-C(3)-C(4)	105.8(5)	C(2)-O(2)-C(6)-C(8)	150.7(5)
C(6)-O(3)-C(3)-C(2)	-5.5(6)	C(2)-O(2)-C(6)-C(7)	-84.5(5)
O(2)-C(2)-C(3)-O(3)	24.4(5)	N(1)-C(1)-C(9)-C(10)	100.9(6)
C(1)-C(2)-C(3)-O(3)	138.7(4)	C(2)-C(1)-C(9)-C(10)	-13.6(7)
O(2)-C(2)-C(3)-C(4)	-92.3(4)	N(1)-C(1)-C(9)-C(14)	-76.0(5)
C(1)-C(2)-C(3)-C(4)	22.0(4)	C(2)-C(1)-C(9)-C(14)	169.6(4)
O(1)-N(1)-C(4)-C(5)	2.1(7)	C(14)-C(9)-C(10)-C(11)	-0.6(9)
C(1)-N(1)-C(4)-C(5)	-178.6(4)	C(1)-C(9)-C(10)-C(11)	-177.5(6)
O(1)-N(1)-C(4)-C(3)	178.7(4)	C(9)-C(10)-C(11)-C(12)	-0.3(11)
C(1)-N(1)-C(4)-C(3)	-2.0(5)	C(10)-C(11)-C(12)-C(13)	0.9(12)
O(3)-C(3)-C(4)-N(1)	-125.5(4)	C(11)-C(12)-C(13)-C(14)	-0.7(11)
C(2)-C(3)-C(4)-N(1)	-12.7(5)	C(12)-C(13)-C(14)-C(9)	-0.2(9)
O(3)-C(3)-C(4)-C(5)	50.8(6)	C(10)-C(9)-C(14)-C(13)	0.8(8)
C(2)-C(3)-C(4)-C(5)	163.6(5)	C(1)-C(9)-C(14)-C(13)	177.8(5)
C(3)-O(3)-C(6)-O(2)	-15.8(6)		
C(3)-O(3)-C(6)-C(8)	-133.1(5)		

9.12 (2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-2-(4-methoxyphenyl)-5-methylpyrrolidine hydrochloride (96·HCl·CH_3OH)

$C_{12}H_{18}ClNO_3 \cdot CH_3OH$

orthorhombic, $P2_12_12_1$

a = 5.6758(12) Å

b = 8.1820(18) Å

c = 33.088(5) Å

α = 90 °

β = 90 °

γ = 90 °

V = 1536.6(5) Å^3

Z = 4, $R(F)$ = 0.0443

$R_w(F^2)$ = 0.0976

Crystal size: 0.6 x 0.5 x 0.4 mm

Calculated density: 1.261 g/cm^3

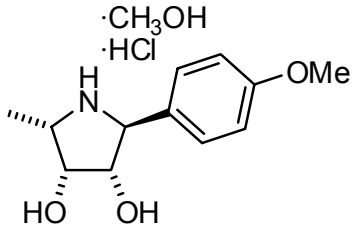
2θ-Range for data collection: 2.46 – 29.99 °

Independent reflections: 3247

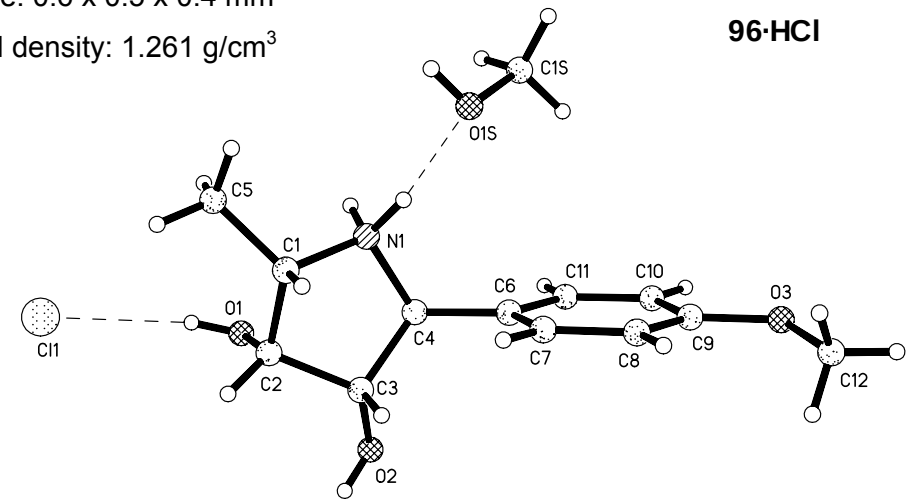
Observed reflections: 2682

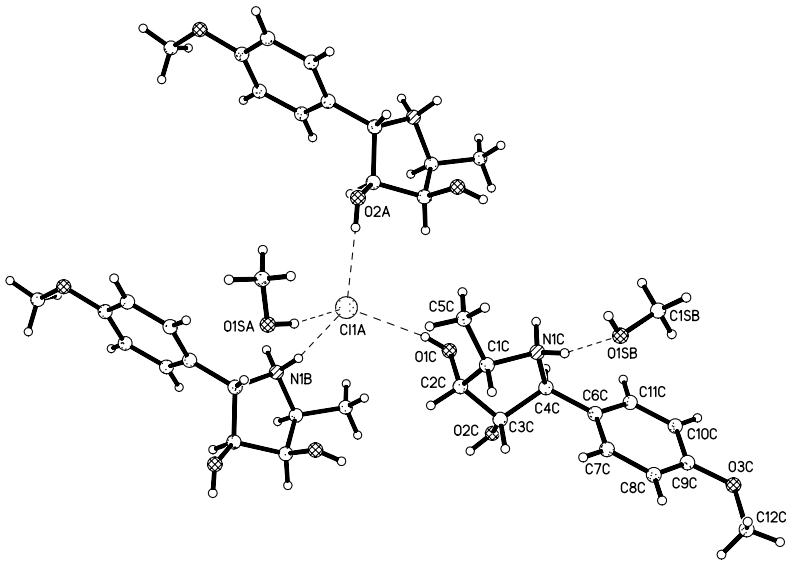
Contributed reflections to refinement: 3247

Refined parameters: 193



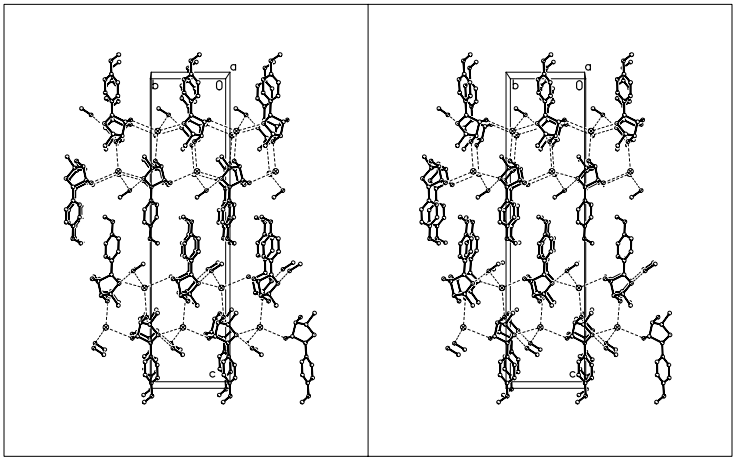
96·HCl



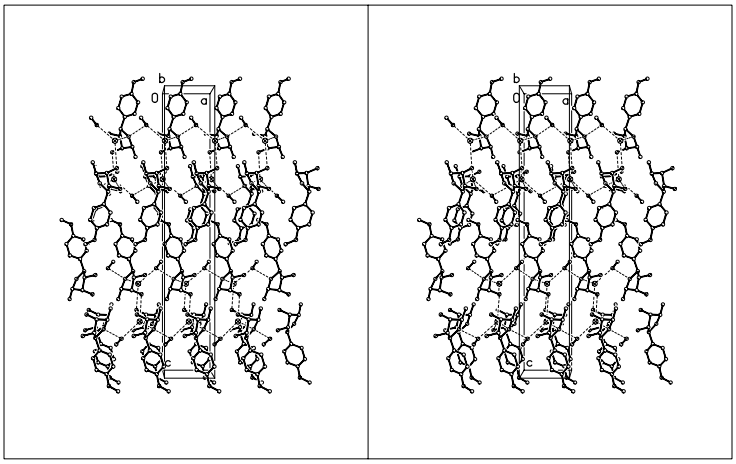


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

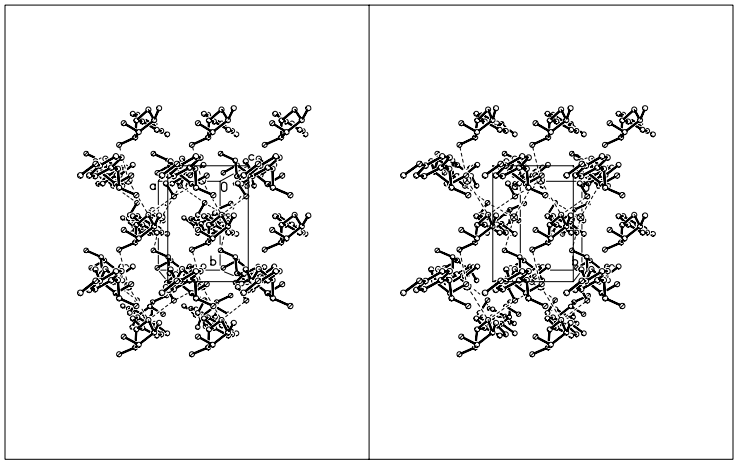
(a)



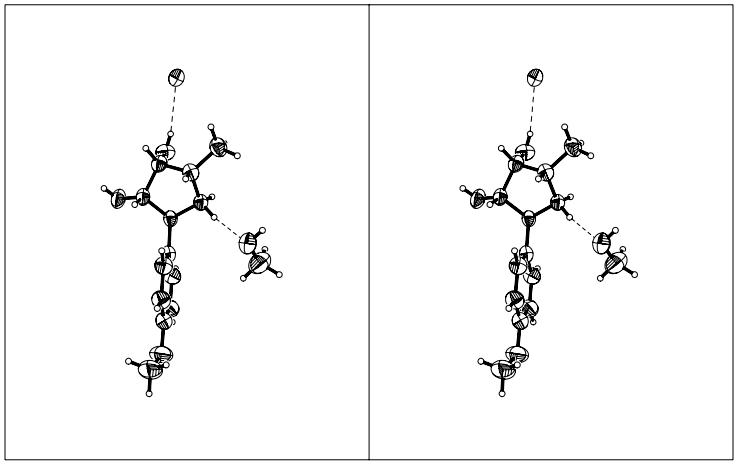
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

N(1)-C(1)	1.518(3)	C(5)-H(5C)	0.96
N(1)-C(4)	1.524(3)	C(6)-C(7)	1.383(3)
N(1)-H(1B)	0.93(3)	C(6)-C(11)	1.394(3)
N(1)-H(1C)	0.97(3)	C(7)-C(8)	1.396(3)
O(1)-C(2)	1.415(3)	C(7)-H(7)	0.93
O(1)-H(1A)	0.78(3)	C(8)-C(9)	1.381(4)
C(1)-C(5)	1.509(3)	C(8)-H(8)	0.93
C(1)-C(2)	1.518(3)	C(9)-C(10)	1.387(4)
C(1)-H(1)	0.98	C(10)-C(11)	1.389(3)
O(2)-C(3)	1.417(3)	C(10)-H(10)	0.93
O(2)-H(2A)	0.80(3)	C(11)-H(11)	0.93
C(2)-C(3)	1.534(3)	C(12)-H(12A)	0.96
C(2)-H(2)	0.98	C(12)-H(12B)	0.96
O(3)-C(9)	1.371(3)	C(12)-H(12C)	0.96
O(3)-C(12)	1.424(4)	O(1S)-C(1S)	1.412(4)
C(3)-C(4)	1.533(3)	O(1S)-H(1S)	0.79(3)
C(3)-H(3)	0.98	C(1S)-H(1S1)	0.96
C(4)-C(6)	1.509(3)	C(1S)-H(1S2)	0.96
C(4)-H(4)	0.98	C(1S)-H(1S3)	0.96
C(5)-H(5A)	0.96	C(1)-N(1)-C(4)	108.6(17)
C(5)-H(5B)	0.96	C(1)-N(1)-H(1B)	111.2(15)

C(4)-N(1)-H(1B)	104.9(17)
C(1)-N(1)-H(1C)	109.3(16)
C(4)-N(1)-H(1C)	113.1(16)
H(1B)-N(1)-H(1C)	110(2)
C(2)-O(1)-H(1A)	107(2)
C(5)-C(1)-C(2)	117.8(2)
C(5)-C(1)-N(1)	110.2(19)
C(2)-C(1)-N(1)	101.0(17)
C(5)-C(1)-H(1)	109.1
C(2)-C(1)-H(1)	109.1
N(1)-C(1)-H(1)	109.1
C(3)-O(2)-H(2A)	102(2)
O(1)-C(2)-C(1)	111.9(18)
O(1)-C(2)-C(3)	107.9(19)
C(1)-C(2)-C(3)	101.2(18)
O(1)-C(2)-H(2)	111.8
C(1)-C(2)-H(2)	111.8
C(3)-C(2)-H(2)	111.8
C(9)-O(3)-C(12)	117.5(2)
O(2)-C(3)-C(4)	108.0(19)
O(2)-C(3)-C(2)	115.9(2)
C(4)-C(3)-C(2)	103.5(17)
O(2)-C(3)-H(3)	109.7
C(4)-C(3)-H(3)	109.7
C(2)-C(3)-H(3)	109.7
C(6)-C(4)-N(1)	111.9(18)
C(6)-C(4)-C(3)	116.7(18)
N(1)-C(4)-C(3)	103.1(16)
C(6)-C(4)-H(4)	108.2
N(1)-C(4)-H(4)	108.2
C(3)-C(4)-H(4)	108.2
C(1)-C(5)-H(5A)	109.5
C(1)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
C(1)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(7)-C(6)-C(11)	118.3(2)
C(7)-C(6)-C(4)	122.7(19)
C(11)-C(6)-C(4)	118.9(2)
C(6)-C(7)-C(8)	121.3(2)
C(6)-C(7)-H(7)	119.3
C(8)-C(7)-H(7)	119.3
C(9)-C(8)-C(7)	119.6(2)
C(9)-C(8)-H(8)	120.2
C(7)-C(8)-H(8)	120.2
O(3)-C(9)-C(8)	125.1(2)
O(3)-C(9)-C(10)	115.0(2)
C(8)-C(9)-C(10)	119.9(2)
C(9)-C(10)-C(11)	120.0(2)
C(9)-C(10)-H(10)	120.0
C(11)-C(10)-H(10)	120.0
C(10)-C(11)-C(6)	120.9(2)
C(10)-C(11)-H(11)	119.6
C(6)-C(11)-H(11)	119.6
O(3)-C(12)-H(12A)	109.5
O(3)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
O(3)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(1S)-O(1S)-H(1S)	111(3)
O(1S)-C(1S)-H(1S1)	109.5
O(1S)-C(1S)-H(1S2)	109.5
H(1S1)-C(1S)-H(1S2)	109.5
O(1S)-C(1S)-H(1S3)	109.5
H(1S1)-C(1S)-H(1S3)	109.5
H(1S2)-C(1S)-H(1S3)	109.5

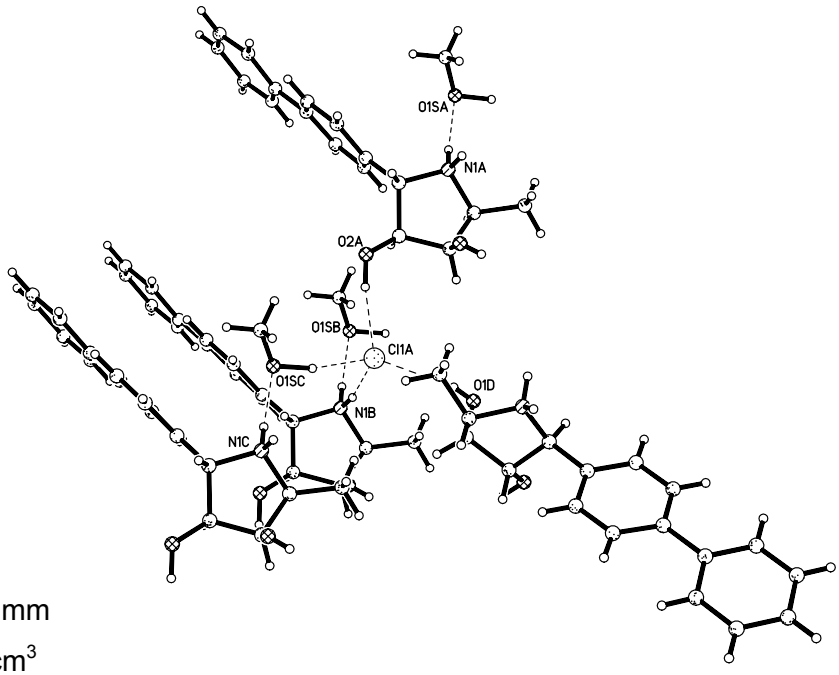
Torsion angles [°]

C(4)-N(1)-C(1)-C(5)	-154.02(19)
C(4)-N(1)-C(1)-C(2)	-28.6(2)
C(5)-C(1)-C(2)-O(1)	50.1(3)
N(1)-C(1)-C(2)-O(1)	-70.0(2)
C(5)-C(1)-C(2)-C(3)	164.8(2)
N(1)-C(1)-C(2)-C(3)	44.7(2)
O(1)-C(2)-C(3)-O(2)	-45.8(2)
C(1)-C(2)-C(3)-O(2)	-163.52(18)
O(1)-C(2)-C(3)-C(4)	72.2(2)
C(1)-C(2)-C(3)-C(4)	-45.5(2)
C(1)-N(1)-C(4)-C(6)	-125.44(19)
C(1)-N(1)-C(4)-C(3)	0.9(2)
O(2)-C(3)-C(4)-C(6)	-86.3(3)
C(2)-C(3)-C(4)-C(6)	150.29(19)
O(2)-C(3)-C(4)-N(1)	150.57(19)
C(2)-C(3)-C(4)-N(1)	27.1(2)
N(1)-C(4)-C(6)-C(7)	67.8(3)
C(3)-C(4)-C(6)-C(7)	-50.7(3)
N(1)-C(4)-C(6)-C(11)	-112.2(2)
C(3)-C(4)-C(6)-C(11)	129.3(2)
C(11)-C(6)-C(7)-C(8)	-1.8(4)
C(4)-C(6)-C(7)-C(8)	178.1(2)
C(6)-C(7)-C(8)-C(9)	0.5(4)
C(12)-O(3)-C(9)-C(10)	-171.7(3)
C(7)-C(8)-C(9)-O(3)	-178.5(3)
C(7)-C(8)-C(9)-C(10)	1.4(4)
O(3)-C(9)-C(10)-C(11)	178.0(3)
C(8)-C(9)-C(10)-C(11)	-1.8(4)
C(9)-C(10)-C(11)-C(6)	0.5(4)
C(7)-C(6)-C(11)-C(10)	1.4(4)
C(4)-C(6)-C(11)-C(10)	-178.6(2)

H-Bond lengths [Å] and angles [°]

O1-Cl1	3.1560(0.0019)
H1A-Cl1	2.3840(0.0270)
N1-O1S	2.7692(0.0029)
H1C-O1S	1.8257(0.0292)
O2-Cl1_$1	3.1506(0.0021)
H2A-Cl1_$1	2.3607(0.0326)
N1-Cl1_$2	3.1547(0.0022)
H1B-Cl1_$2	2.2424(0.0301)
O1S-Cl1_$3	3.0729(0.0025)
H1S-Cl1_$3	2.3152(0.0325)
O1-H1A-Cl1	170.06(2.89)
N1-H1C-O1S	163.07(2.54)
O2-H2A-Cl1_$1	168.28(3.24)
N1-H1B-Cl1_$2	167.31(2.23)
O1S-H1S-Cl1_$3	160.32(3.15)

9.13 (2*S*,3*S*,4*R*,5*S*)-2-([1,1'-Biphenyl]-4-yl)-3,4-dihydroxy-5-methylpyrrolidine hydrochloride (97·HCl·CH_3OH)



$C_{17}H_{20}ClNO_2 \cdot CH_3OH$

monoclinic, $P2_1$

a = 5.6733(11) Å

b = 8.1555(17) Å

c = 19.343(3) Å

α = 90 °

β = 96.745(15) °

γ = 90 °

V = 888.8(3) Å^3

Z = 2, $R(F)$ = 0.0823

$R_w(F^2)$ = 0.1173

Crystal size: 0.8 x 0.3 x 0.07 mm

Calculated density: 1.262 g/cm^3

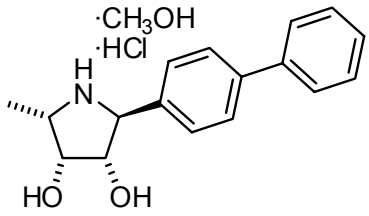
2θ-Range for data collection: 2.12 – 27.99 °

Independent reflections: 2587

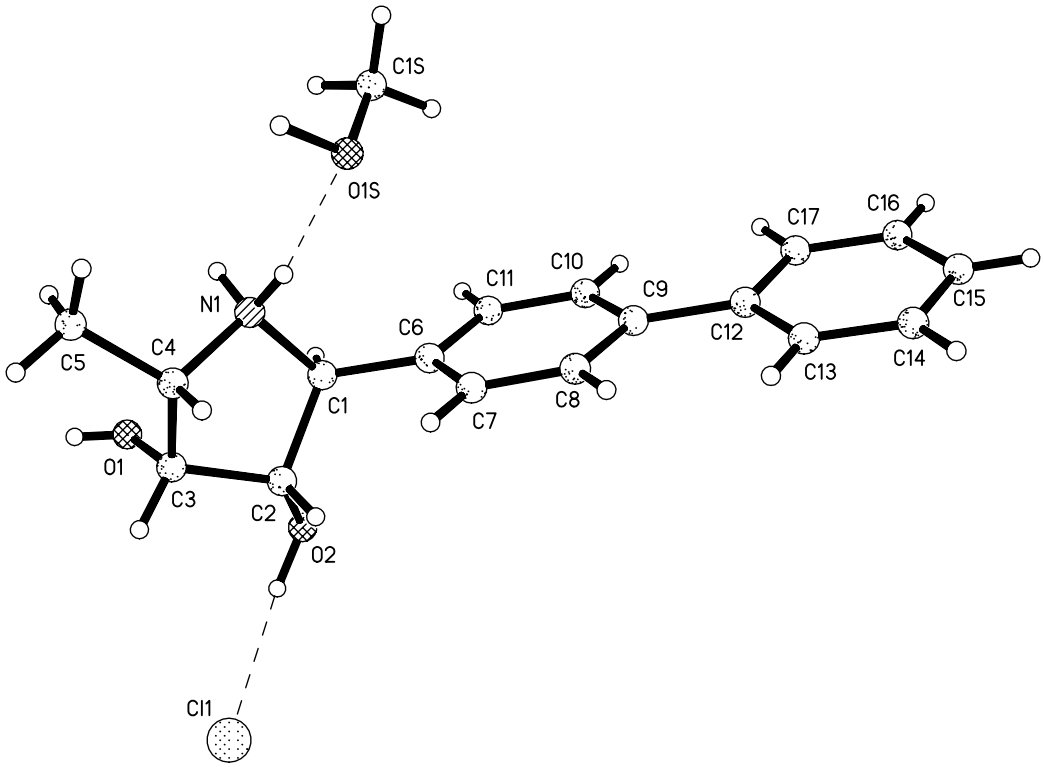
Observed reflections: 1627

Contributed reflections to refinement: 2587

Refined parameters: 221

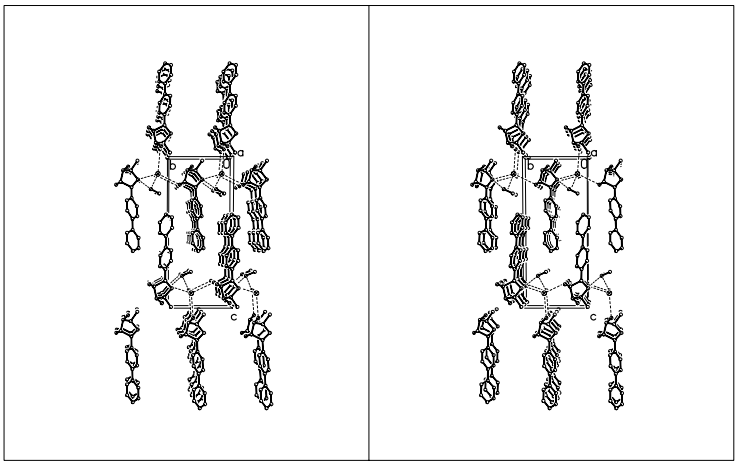


97·HCl

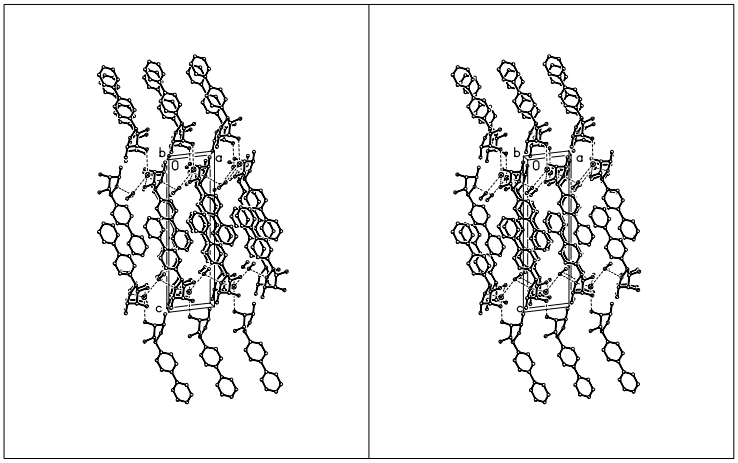


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

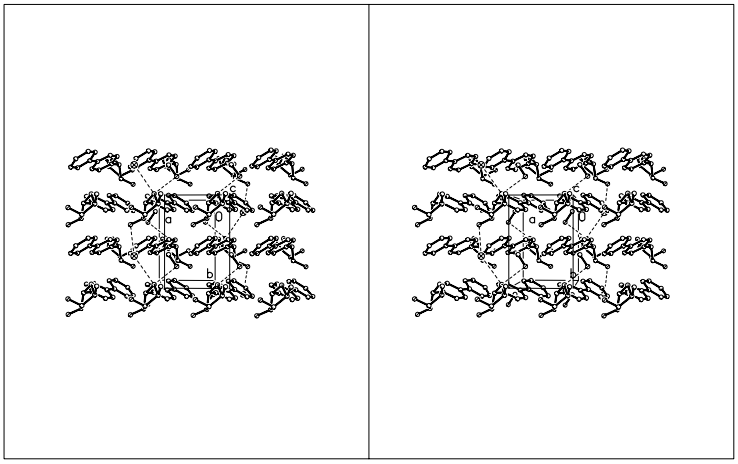
(a)



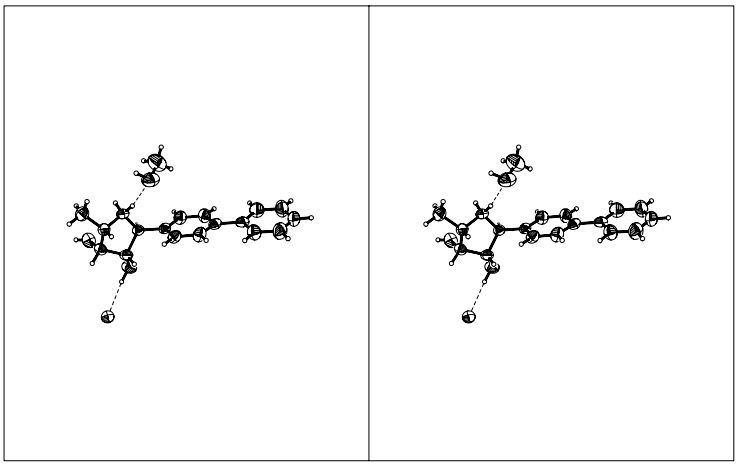
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

O(1)-C(3)	1.417(7)	C(15)-H(15)	0.93
O(1)-H(1)	0.62(6)	C(16)-C(17)	1.383(9)
N(1)-C(1)	1.516(7)	C(16)-H(16)	0.93
N(1)-C(4)	1.520(7)	C(17)-H(17)	0.93
N(1)-H(1A)	0.90	O(1S)-C(1S)	1.418(11)
N(1)-H(1B)	0.90	O(1S)-H(1S)	1.14(8)
C(1)-C(6)	1.524(7)	C(1S)-H(1S1)	0.96
C(1)-C(2)	1.547(7)	C(1S)-H(1S2)	0.96
C(1)-H(1C)	0.98	C(1S)-H(1S3)	0.96
O(2)-C(2)	1.412(8)	C(3)-O(1)-H(1)	117(8)
O(2)-H(2)	0.72(9)	C(1)-N(1)-C(4)	108.4(4)
C(2)-C(3)	1.527(7)	C(1)-N(1)-H(1A)	110.0
C(2)-H(2A)	0.98	C(4)-N(1)-H(1A)	110.0
C(3)-C(4)1	0.515(7)	C(1)-N(1)-H(1B)	110.0
C(3)-H(3)	0.98	C(4)-N(1)-H(1B)	110.0
C(4)-C(5)	1.511(8)	H(1A)-N(1)-H(1B)	108.4
C(4)-H(4)	0.98	N(1)-C(1)-C(6)	111.7(4)
C(5)-H(5A)	0.96	N(1)-C(1)-C(2)	103.6(4)
C(5)-H(5B)	0.96	C(6)-C(1)-C(2)	116.5(5)
C(5)-H(5C)	0.96	N(1)-C(1)-H(1C)	108.2
C(6)-C(11)	1.383(8)	C(6)-C(1)-H(1C)	108.2
C(6)-C(7)	1.391(7)	C(2)-C(1)-H(1C)	108.2
C(7)-C(8)	1.386(7)	C(2)-O(2)-H(2)	106(8)
C(7)-H(7)	0.93	O(2)-C(2)-C(3)	116.8(5)
C(8)-C(9)	1.4(8)	O(2)-C(2)-C(1)	108.4(5)
C(8)-H(8)	0.93	C(3)-C(2)-C(1)	103.4(4)
C(9)-C(10)	1.383(8)	O(2)-C(2)-H(2A)	109.3
C(9)-C(12)	1.507(8)	C(3)-C(2)-H(2A)	109.3
C(10)-C(11)	1.390(8)	C(1)-C(2)-H(2A)	109.3
C(10)-H(10)	0.93	O(1)-C(3)-C(4)	111.7(5)
C(11)-H(11)	0.93	O(1)-C(3)-C(2)	107.4(5)
C(12)-C(13)	1.374(8)	C(4)-C(3)-C(2)	102.1(4)
C(12)-C(17)	1.396(8)	O(1)-C(3)-H(3)	111.7
C(13)-C(14)	1.384(8)	C(4)-C(3)-H(3)	111.7
C(13)-H(13)	0.93	C(2)-C(3)-H(3)	111.7
C(14)-C(15)	1.373(10)	C(5)-C(4)-C(3)	118.5(5)
C(14)-H(14)	0.93	C(5)-C(4)-N(1)	109.4(5)
C(15)-C(16)	1.381(10)	C(3)-C(4)-N(1)	1.6(4)

C(5)-C(4)-H(4)	109.3
C(3)-C(4)-H(4)	109.3
N(1)-C(4)-H(4)	109.3
C(4)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
C(4)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(11)-C(6)-C(7)	118.1(5)
C(11)-C(6)-C(1)	119.4(5)
C(7)-C(6)-C(1)	122.5(5)
C(8)-C(7)-C(6)	121.0(6)
C(8)-C(7)-H(7)	119.5
C(6)-C(7)-H(7)	119.5
C(7)-C(8)-C(9)	121.0(5)
C(7)-C(8)-H(8)	119.5
C(9)-C(8)-H(8)	119.5
C(10)-C(9)-C(8)	117.4(5)
C(10)-C(9)-C(12)	121.9(5)
C(8)-C(9)-C(12)	120.7(5)
C(9)-C(10)-C(11)	121.6(6)
C(9)-C(10)-H(10)	119.2
C(11)-C(10)-H(10)	119.2
C(6)-C(11)-C(10)	120.9(6)
C(6)-C(11)-H(11)	119.6
C(10)-C(11)-H(11)	119.6
C(13)-C(12)-C(17)	116.9(6)
C(13)-C(12)-C(9)	122.5(6)
C(17)-C(12)-C(9)	120.6(6)
C(12)-C(13)-C(14)	122.0(7)
C(12)-C(13)-H(13)	119.0
C(14)-C(13)-H(13)	119.0
C(15)-C(14)-C(13)	120.5(7)
C(15)-C(14)-H(14)	119.8
C(13)-C(14)-H(14)	119.8
C(14)-C(15)-C(16)	118.9(6)
C(14)-C(15)-H(15)	120.6
C(16)-C(15)-H(15)	120.6
C(15)-C(16)-C(17)	120.1(7)
C(15)-C(16)-H(16)	119.9
C(17)-C(16)-H(16)	119.9
C(16)-C(17)-C(12)	121.6(7)
C(16)-C(17)-H(17)	119.2
C(12)-C(17)-H(17)	119.2
C(1S)-O(1S)-H(1S)	117(5)
O(1S)-C(1S)-H(1S1)	109.5
O(1S)-C(1S)-H(1S2)	109.5
H(1S1)-C(1S)-H(1S2)	109.5
O(1S)-C(1S)-H(1S3)	109.5
H(1S1)-C(1S)-H(1S3)	109.5
H(1S2)-C(1S)-H(1S3)	109.5

Torsion angles [°]

C(4)-N(1)-C(1)-C(6)	-120.9(5)
C(4)-N(1)-C(1)-C(2)	5.2(6)
N(1)-C(1)-C(2)-O(2)	147.7(5)
C(6)-C(1)-C(2)-O(2)	-89.2(7)
N(1)-C(1)-C(2)-C(3)	23.1(6)
C(6)-C(1)-C(2)-C(3)	146.1(5)
O(2)-C(2)-C(3)-O(1)	-44.7(7)
C(1)-C(2)-C(3)-O(1)	74.4(6)
O(2)-C(2)-C(3)-C(4)	-162.2(5)
C(1)-C(2)-C(3)-C(4)	-43.2(6)
O(1)-C(3)-C(4)-C(5)	49.9(7)
C(2)-C(3)-C(4)-C(5)	164.4(5)
O(1)-C(3)-C(4)-N(1)	-69.1(5)
C(2)-C(3)-C(4)-N(1)	45.3(5)
C(1)-N(1)-C(4)-C(5)	-157.0(4)
C(1)-N(1)-C(4)-C(3)	-31.5(5)
N(1)-C(1)-C(6)-C(11)	-110.6(6)
C(2)-C(1)-C(6)-C(11)	130.7(6)
N(1)-C(1)-C(6)-C(7)	67.0(7)
C(2)-C(1)-C(6)-C(7)	-51.7(8)
C(11)-C(6)-C(7)-C(8)	-1.0(10)
C(1)-C(6)-C(7)-C(8)	-178.6(7)
C(6)-C(7)-C(8)-C(9)	-0.4(11)
C(7)-C(8)-C(9)-C(10)	1.1(10)
C(7)-C(8)-C(9)-C(12)	-178.2(7)
C(8)-C(9)-C(10)-C(11)	-0.4(10)
C(12)-C(9)-C(10)-C(11)	178.9(6)
C(7)-C(6)-C(11)-C(10)	1.7(10)
C(1)-C(6)-C(11)-C(10)	179.4(6)
C(9)-C(10)-C(11)-C(6)	-1.0(11)
C(10)-C(9)-C(12)-C(13)	-174.5(7)
C(8)-C(9)-C(12)-C(13)	4.9(10)
C(10)-C(9)-C(12)-C(17)	5.5(10)
C(8)-C(9)-C(12)-C(17)	-175.2(7)
C(17)-C(12)-C(13)-C(14)	-1.5(11)
C(9)-C(12)-C(13)-C(14)	178.5(7)
C(12)-C(13)-C(14)-C(15)	1.2(13)
C(13)-C(14)-C(15)-C(16)	-0.3(13)
C(14)-C(15)-C(16)-C(17)	-0.1(13)
C(15)-C(16)-C(17)-C(12)	-0.3(13)
C(13)-C(12)-C(17)-C(16)	1.0(11)
C(9)-C(12)-C(17)-C(16)	-178.9(7)

H-Bond lengths [Å] and angles [°]

O1-Cl1_$1	3.1737(0.0056)
H1-Cl1_$1	2.5759(0.0619)
O2-Cl1	3.1714(0.0058)
H2-Cl1	2.4588(0.0864)
O1S-Cl1_$2	3.0720(0.0058)
H1S-Cl1_$2	1.9659(0.0798)
N1-Cl1_$3	3.1818(0.0049)
H1A-Cl1_$3	2.2943
N1-O1S	2.8147(0.0071)
H1B-O1S	1.9436
O1-H1-Cl1_$1	162.92(10.74)
O2-H2-Cl1	168.76(9.79)
O1S-H1S-Cl1_$2	162.69(6.35)
N1-H1A-Cl1_$3	168.72
N1-H1B-O1S	162.40

9.14 (2*S*,3*S*,4*R*,5*S*)-2-(4-Chlorophenyl)-3,4-dihydroxy-5-methylpyrrolidine hydrochloride (99·HCl·CH_3OH)

$C_{11}H_{15}Cl_2NO_2 \cdot CH_3OH$

orthorhombic, $P2_12_12_1$

a = 5.6069(7) Å

b = 8.1476(7) Å

c = 32.562(3) Å

α = 90 °

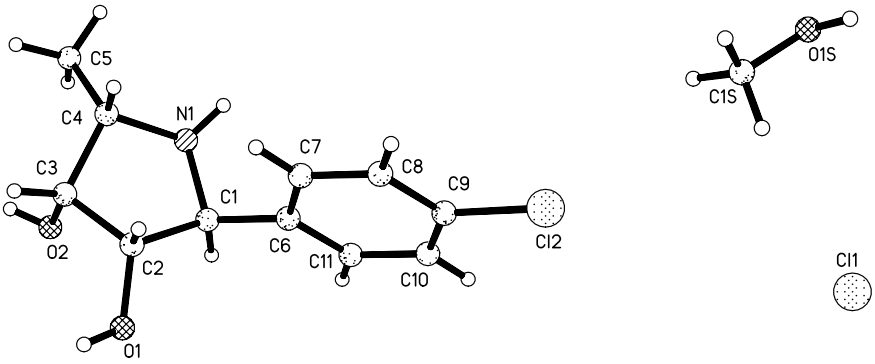
β = 90 °

γ = 90 °

V = 1487.5(3) Å^3

Z = 4, $R(F)$ = 0.0692

$R_w(F^2)$ = 0.1761



Crystal size: 1.0 x 0.25 x 0.15 mm

Calculated density: 1.323 g/cm^3

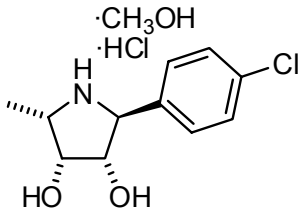
2θ-Range for data collection: 2.71 – 67.92 °

Independent reflections: 1965

Observed reflections: 1108

Contributed reflections to refinement: 1965

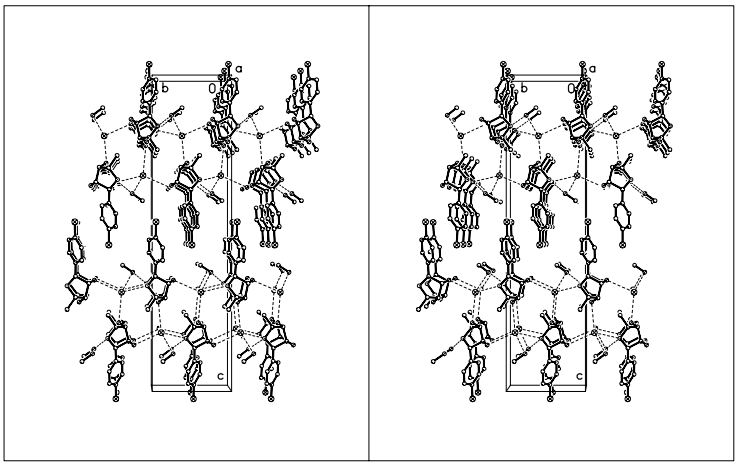
Refined parameters: 167



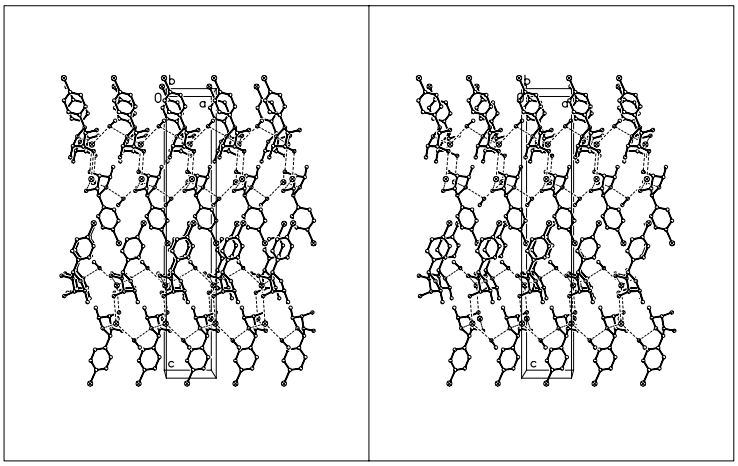
99·HCl

Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

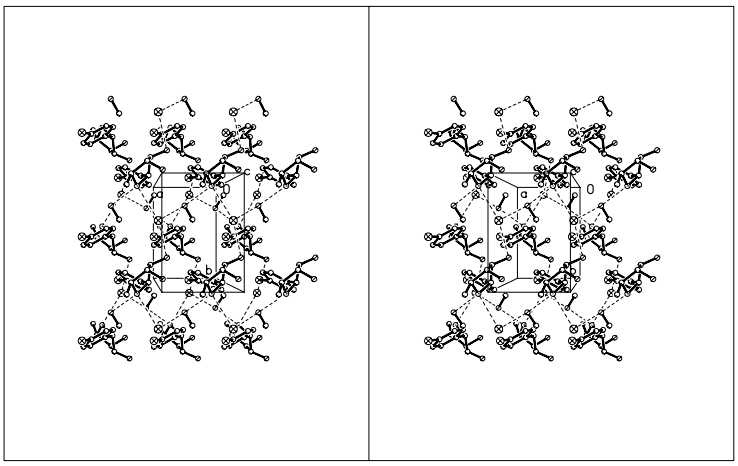
(a)



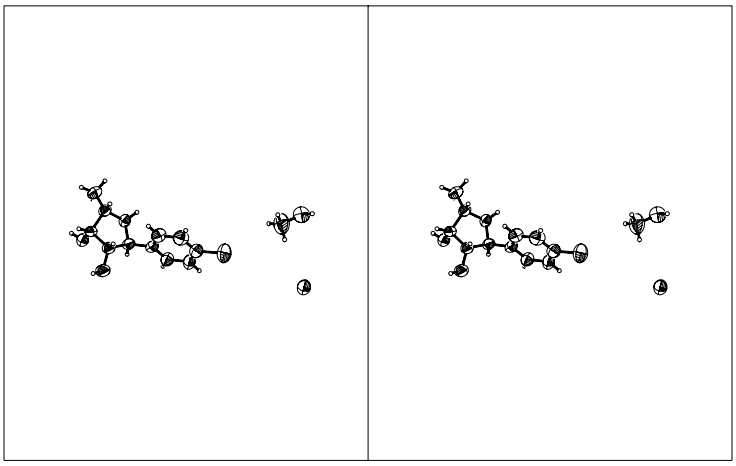
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

N(1)-C(4)	1.513(8)	C(1)-N(1)-H(1A)	110.0
N(1)-C(1)	1.523(8)	C(4)-N(1)-H(1B)	110.0
N(1)-H(1A)	0.90	C(1)-N(1)-H(1B)	110.0
N(1)-H(1B)	0.90	H(1A)-N(1)-H(1B)	108.3
O(1)-C(2)	1.391(8)	C(2)-O(1)-H(1)	109.5
O(1)-H(1)	0.82	C(6)-C(1)-N(1)	111.4(6)
C(1)-C(6)	1.522(9)	C(6)-C(1)-C(2)	116.9(6)
C(1)-C(2)	1.542(9)	N(1)-C(1)-C(2)	103.6(5)
C(1)-H(1C)	0.98	C(6)-C(1)-H(1C)	108.2
Cl(2)-C(9)	1.721(8)	N(1)-C(1)-H(1C)	108.2
O(2)-C(3)	1.402(9)	C(2)-C(1)-H(1C)	108.2
O(2)-H(2)	0.82	C(3)-O(2)-H(2)	109.5
C(2)-C(3)	1.535(9)	O(1)-C(2)-C(3)	115.9(6)
C(2)-H(2A)	0.98	O(1)-C(2)-C(1)	108.8(6)
C(3)-C(4)	1.531(10)	C(3)-C(2)-C(1)	103.8(6)
C(3)-H(3)	0.98	O(1)-C(2)-H(2A)	109.3
C(4)-C(5)	1.517(9)	C(3)-C(2)-H(2A)	109.3
C(4)-H(4)	0.98	C(1)-C(2)-H(2A)	109.3
C(5)-H(5A)	0.96	O(2)-C(3)-C(4)	111.5(6)
C(5)-H(5B)	0.96	O(2)-C(3)-C(2)	107.7(6)
C(5)-H(5C)	0.96	C(4)-C(3)-C(2)	101.4(6)
C(6)-C(11)	1.373(9)	O(2)-C(3)-H(3)	111.9
C(6)-C(7)	1.398(10)	C(4)-C(3)-H(3)	111.9
C(7)-C(8)	1.376(10)	C(2)-C(3)-H(3)	111.9
C(7)-H(7)	0.93	N(1)-C(4)-C(5)	110.2(6)
C(8)-C(9)	1.377(11)	N(1)-C(4)-C(3)	1.7(5)
C(8)-H(8)	0.93	C(5)-C(4)-C(3)	117.2(6)
C(9)-C(10)	1.364(12)	N(1)-C(4)-H(4)	109.4
C(10)-C(11)	1.404(10)	C(5)-C(4)-H(4)	109.4
C(10)-H(10)	0.93	C(3)-C(4)-H(4)	109.4
C(11)-H(11)	0.93	C(4)-C(5)-H(5A)	109.5
O(1S)-C(1S)	1.413(10)	C(4)-C(5)-H(5B)	109.5
O(1S)-H(1S)	0.82	H(5A)-C(5)-H(5B)	109.5
C(1S)-H(1S1)	0.96	C(4)-C(5)-H(5C)	109.5
C(1S)-H(1S2)	0.96	H(5A)-C(5)-H(5C)	109.5
C(1S)-H(1S3)	0.96	H(5B)-C(5)-H(5C)	109.5
C(4)-N(1)-C(1)	108.6(5)	C(11)-C(6)-C(7)	119.6(7)
C(4)-N(1)-H(1A)	110.0	C(11)-C(6)-C(1)	119.5(7)

C(7)-C(6)-C(1)	120.8(6)
C(8)-C(7)-C(6)	119.3(7)
C(8)-C(7)-H(7)	120.3
C(6)-C(7)-H(7)	120.3
C(7)-C(8)-C(9)	121.0(8)
C(7)-C(8)-H(8)	119.5
C(9)-C(8)-H(8)	119.5
C(10)-C(9)-C(8)	120.1(8)
C(10)-C(9)-Cl(2)	121.0(7)
C(8)-C(9)-Cl(2)	119.0(7)
C(9)-C(10)-C(11)	119.8(8)
C(9)-C(10)-H(10)	120.1
C(11)-C(10)-H(10)	120.1
C(6)-C(11)-C(10)	120.1(8)
C(6)-C(11)-H(11)	119.9
C(10)-C(11)-H(11)	119.9
C(1S)-O(1S)-H(1S)	109.5
O(1S)-C(1S)-H(1S1)	109.5
O(1S)-C(1S)-H(1S2)	109.5
H(1S1)-C(1S)-H(1S2)	109.5
O(1S)-C(1S)-H(1S3)	109.5
H(1S1)-C(1S)-H(1S3)	109.5
H(1S2)-C(1S)-H(1S3)	109.5

Torsion angles [°]

C(4)-N(1)-C(1)-C(6)	-121.2(6)
C(4)-N(1)-C(1)-C(2)	5.2(7)
C(6)-C(1)-C(2)-O(1)	-89.7(8)
N(1)-C(1)-C(2)-O(1)	147.4(6)
C(6)-C(1)-C(2)-C(3)	146.2(6)
N(1)-C(1)-C(2)-C(3)	23.3(7)
O(1)-C(2)-C(3)-O(2)	-45.2(9)
C(1)-C(2)-C(3)-O(2)	74.1(7)
O(1)-C(2)-C(3)-C(4)	-162.4(6)
C(1)-C(2)-C(3)-C(4)	-43.1(7)
C(1)-N(1)-C(4)-C(5)	-155.9(6)
C(1)-N(1)-C(4)-C(3)	-31.5(7)
O(2)-C(3)-C(4)-N(1)	-69.2(7)
C(2)-C(3)-C(4)-N(1)	45.2(7)
O(2)-C(3)-C(4)-C(5)	50.2(9)
C(2)-C(3)-C(4)-C(5)	164.6(7)
N(1)-C(1)-C(6)-C(11)	-110.2(8)
C(2)-C(1)-C(6)-C(11)	131.1(8)
N(1)-C(1)-C(6)-C(7)	67.8(9)
C(2)-C(1)-C(6)-C(7)	-51.0(9)
C(11)-C(6)-C(7)-C(8)	-1.2(12)
C(1)-C(6)-C(7)-C(8)	-179.1(7)
C(6)-C(7)-C(8)-C(9)	1.3(13)
C(7)-C(8)-C(9)-C(10)	-1.6(14)
C(7)-C(8)-C(9)-Cl(2)	179.1(6)
C(8)-C(9)-C(10)-C(11)	1.7(14)
Cl(2)-C(9)-C(10)-C(11)	-179.1(7)
C(7)-C(6)-C(11)-C(10)	1.3(13)
C(1)-C(6)-C(11)-C(10)	179.2(7)
C(9)-C(10)-C(11)-C(6)	-1.6(14)

H-Bond lengths [Å] and angles [°]

N1-O1S_$1	2.7811(0.0085)
H1A-O1S_$1	1.9069
N1-Cl1_$1	3.1464(0.0059)
H1B-Cl1_$1	2.2585
O1-Cl1_$2	3.1343(0.0057)
H1-Cl1_$2	2.3546
O2-Cl1_$3	3.1566(0.0048)
H2-Cl1_$3	2.3490
O1S-Cl1_$4	3.0727(0.0064)
H1S-Cl1_$4	2.2570
N1-H1A-O1S_$1	163.35
N1-H1B-Cl1_$1	168.87
O1-H1-Cl1_$2	159.11
O2-H2-Cl1_$3	168.43

9.15 (2*S*,3*S*,4*R*,5*S*)-2-(4-Bromophenyl)-3,4-dihydroxy-5-methylpyrrolidine hydrochloride (100·HCl)

$C_{11}H_{15}BrClNO_2 \cdot CH_3OH$

orthorhombic, $P2_12_12_1$

a = 5.6565(10) Å

b = 8.1868(14) Å

c = 32.847(5) Å

α = 90 °

β = 90 °

γ = 90 °

V = 1521.1(4) Å^3

Z = 4, $R(F)$ = 0.0405

$R_w(F^2)$ = 0.0700

Crystal size: 1.0 x 0.2 x 0.2 mm

Calculated density: 1.487 g/cm^3

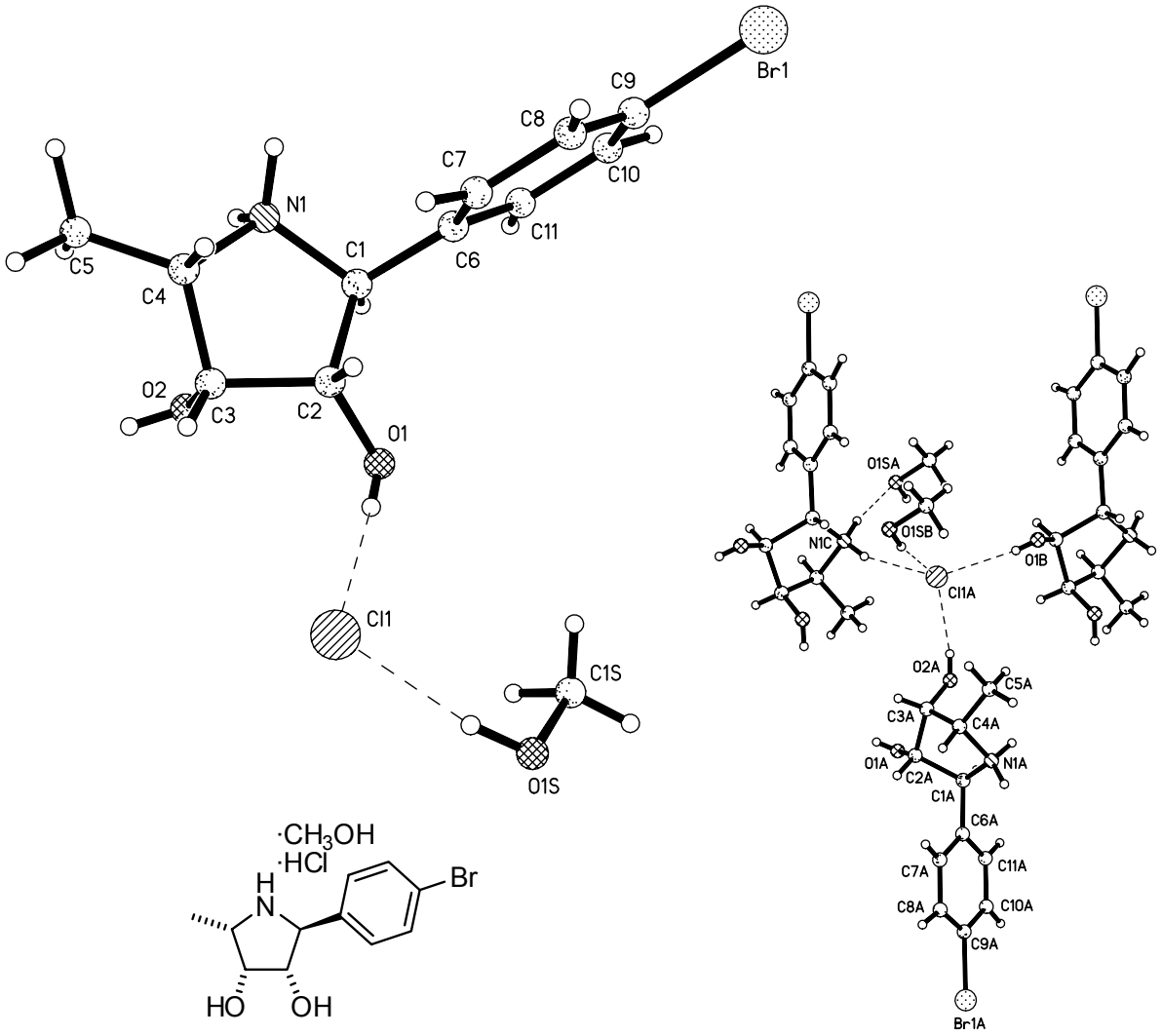
2θ-Range for data collection: 2.48 – 26.00 °

Independent reflections: 2917

Observed reflections: 2071

Contributed reflections to refinement: 2917

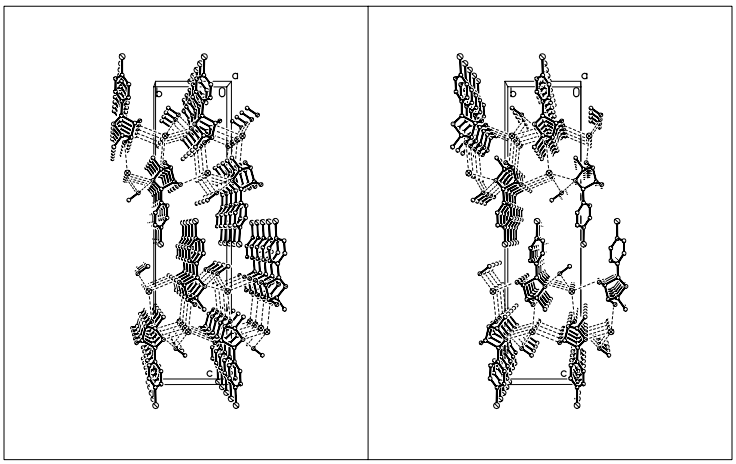
Refined parameters: 240



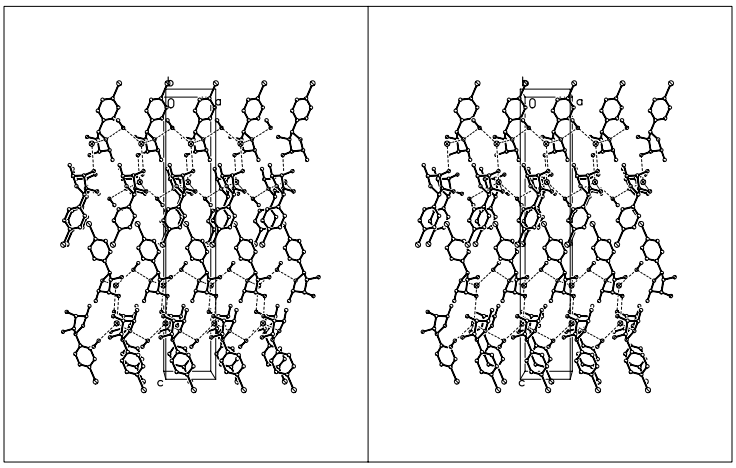
100·HCl

Unit cell; View along the a-axis (a) and b-axis (b)

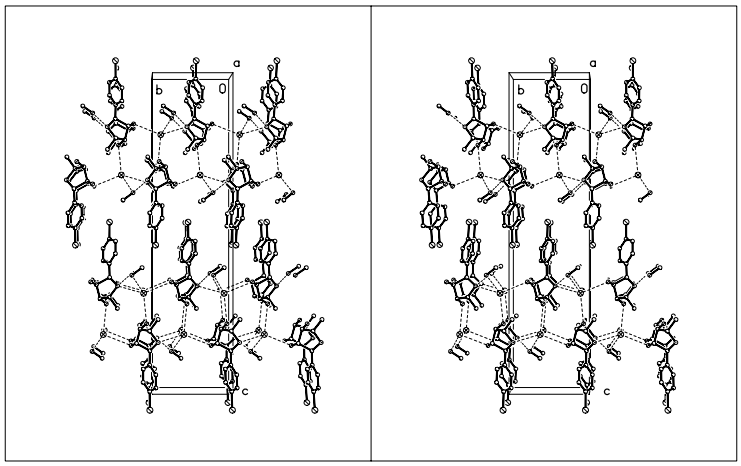
(a)



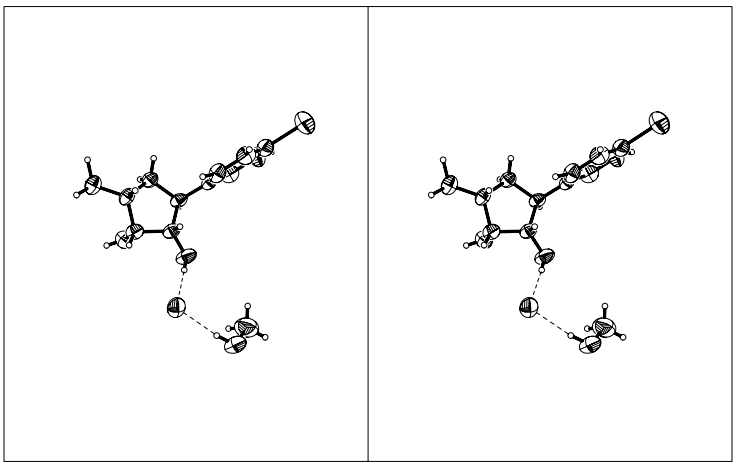
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

Br(1)-C(9)	1.908(4)	C(4)-C(3)-C(2)	101.3(3)
O(1)-C(2)	1.407(4)	O(2)-C(3)-H(3)	116(2)
O(1)-H(1C)	0.77(3)	C(4)-C(3)-H(3)	110(2)
N(1)-C(1)	1.511(4)	C(2)-C(3)-H(3)	108(2)
N(1)-C(4)	1.516(5)	C(5)-C(4)-N(1)	111.0(3)
N(1)-H(1A)	0.82(4)	C(5)-C(4)-C(3)	118.1(3)
N(1)-H(1B)	1.02(4)	N(1)-C(4)-C(3)	1.6(3)
C(1)-C(6)	1.523(5)	C(5)-C(4)-H(4)	110.3(17)
C(1)-C(2)	1.544(5)	N(1)-C(4)-H(4)	105.3(18)
C(1)-H(1)	0.95(3)	C(3)-C(4)-H(4)	110.4(18)
O(2)-C(3)	1.4(5)	C(4)-C(5)-H(5A)	112(2)
O(2)-H(2A)	0.78(3)	C(4)-C(5)-H(5B)	113(2)
C(2)-C(3)	1.530(5)	H(5A)-C(5)-H(5B)	97(3)
C(2)-H(2)	0.98(3)	C(4)-C(5)-H(5C)	110(3)
C(3)-C(4)	1.530(5)	H(5A)-C(5)-H(5C)	119(4)
C(3)-H(3)	0.96(4)	H(5B)-C(5)-H(5C)	106(3)
C(4)-C(5)	1.504(5)	C(7)-C(6)-C(11)	118.5(3)
C(4)-H(4)	0.93(3)	C(7)-C(6)-C(1)	121.8(3)
C(5)-H(5A)	1.02(5)	C(11)-C(6)-C(1)	119.7(3)
C(5)-H(5B)	1.01(4)	C(6)-C(7)-C(8)	121.5(4)
C(5)-H(5C)	0.91(5)	C(6)-C(7)-H(7)	121(2)
C(6)-C(7)	1.379(5)	C(8)-C(7)-H(7)	117(2)
C(6)-C(11)	1.385(5)	C(9)-C(8)-C(7)	118.7(4)
C(7)-C(8)	1.381(5)	C(9)-C(8)-H(8)	124(2)
C(7)-H(7)	0.91(3)	C(7)-C(8)-H(8)	117(2)
C(8)-C(9)	1.366(6)	C(8)-C(9)-C(10)	121.7(4)
C(8)-H(8)	0.98(4)	C(8)-C(9)-Br(1)	119.1(3)
C(9)-C(10)	1.369(6)	C(10)-C(9)-Br(1)	119.2(3)
C(10)-C(11)	1.387(5)	C(9)-C(10)-C(11)	119.1(4)
C(10)-H(10)	0.97(4)	C(9)-C(10)-H(10)	116(2)
C(11)-H(11)	0.98(4)	C(11)-C(10)-H(10)	125(2)
O(1S)-C(1S)	1.406(7)	C(6)-C(11)-C(10)	120.5(4)
O(1S)-H(1S)	1.05(6)	C(6)-C(11)-H(11)	121(2)
C(1S)-H(1S1)	0.89(8)	C(10)-C(11)-H(11)	118(2)
C(1S)-H(1S2)	0.80(11)	C(1S)-O(1S)-H(1S)	116(4)
C(1S)-H(1S3)	0.92(6)	O(1S)-C(1S)-H(1S1)	113(5)
C(2)-O(1)-H(1C)	106(4)	O(1S)-C(1S)-H(1S2)	94(8)
C(1)-N(1)-C(4)	109.0(3)	H(1S1)-C(1S)-H(1S2)	96(9)
C(1)-N(1)-H(1A)	107(3)	O(1S)-C(1S)-H(1S3)	103(4)
C(4)-N(1)-H(1A)	103(3)	H(1S1)-C(1S)-H(1S3)	110(6)
C(1)-N(1)-H(1B)	115.0(19)	H(1S2)-C(1S)-H(1S3)	139(10)
C(4)-N(1)-H(1B)	106(2)		
H(1A)-N(1)-H(1B)	115(3)	Torsion angles [°]	
N(1)-C(1)-C(6)	110.5(3)		
N(1)-C(1)-C(2)	103.5(3)	C(4)-N(1)-C(1)-C(6)	-122.4(3)
C(6)-C(1)-C(2)	116.3(3)	C(4)-N(1)-C(1)-C(2)	2.8(4)
N(1)-C(1)-H(1)	109.5(17)	N(1)-C(1)-C(2)-O(1)	149.1(3)
C(6)-C(1)-H(1)	110.3(17)	C(6)-C(1)-C(2)-O(1)	-89.5(4)
C(2)-C(1)-H(1)	106.4(17)	N(1)-C(1)-C(2)-C(3)	25.6(4)
C(3)-O(2)-H(2A)	108(3)	C(6)-C(1)-C(2)-C(3)	147.0(3)
O(1)-C(2)-C(3)	115.9(3)	O(1)-C(2)-C(3)-O(2)	-45.1(4)
O(1)-C(2)-C(1)	108.4(3)	C(1)-C(2)-C(3)-O(2)	73.3(3)
C(3)-C(2)-C(1)	103.4(3)	O(1)-C(2)-C(3)-C(4)	-162.8(3)
O(1)-C(2)-H(2)	114.5(17)	C(1)-C(2)-C(3)-C(4)	-44.4(4)
C(3)-C(2)-H(2)	110.4(17)	C(1)-N(1)-C(4)-C(5)	-155.7(3)
C(1)-C(2)-H(2)	102.8(18)	C(1)-N(1)-C(4)-C(3)	-29.8(4)
O(2)-C(3)-C(4)	111.8(3)	O(2)-C(3)-C(4)-C(5)	50.6(5)
O(2)-C(3)-C(2)	108.4(3)	C(2)-C(3)-C(4)-C(5)	165.8(3)

O(2)-C(3)-C(4)-N(1)	-70.3(4)
C(2)-C(3)-C(4)-N(1)	44.9(3)
N(1)-C(1)-C(6)-C(7)	66.7(4)
C(2)-C(1)-C(6)-C(7)	-50.9(5)
N(1)-C(1)-C(6)-C(11)	-112.2(4)
C(2)-C(1)-C(6)-C(11)	130.2(3)
C(11)-C(6)-C(7)-C(8)	0.2(6)
C(1)-C(6)-C(7)-C(8)	-178.7(3)
C(6)-C(7)-C(8)-C(9)	0.3(6)
C(7)-C(8)-C(9)-C(10)	-0.6(6)
C(7)-C(8)-C(9)-Br(1)	179.2(3)
C(8)-C(9)-C(10)-C(11)	0.4(7)
Br(1)-C(9)-C(10)-C(11)	-179.4(3)
C(7)-C(6)-C(11)-C(10)	-0.4(6)
C(1)-C(6)-C(11)-C(10)	178.5(4)
C(9)-C(10)-C(11)-C(6)	0.2(6)

H-Bond lengths [Å] and angles [°]

O1S-Cl1	3.0690(0.0032)
H1S-Cl1	2.0369(0.0607)
O1-Cl1	3.1363(0.0032)
H1C-Cl1	2.3903(0.0340)
O2-Cl1_$1	3.1641(0.0029)
H2A-Cl1_$1	2.3878(0.0295)
N1-Cl1_$2	3.1563(0.0036)
H1A-Cl1_$2	2.3740(0.0401)
N1-O1S_$3	2.7809(0.0043)
H1B-O1S_$3	1.7967(0.0412)
O1S-H1S-Cl1	168.80(5.10)
O1-H1C-Cl1	162.33(4.93)
O2-H2A-Cl1_$1	171.19(4.26)
N1-H1A-Cl1_$2	159.69(3.43)
N1-H1B-O1S_$3	159.72(3.16)

9.16 (2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-5-methyl-2-(4-methylthiophenyl)-pyrrolidine hydrochloride (101·HCl·CH_3OH)

$C_{13}H_{22}ClNO_3S$

monoclinic, $P2_1$

a = 5.6252(4) Å

b = 8.1671(5) Å

c = 17.5033(11) Å

α = 90 °

β = 96.813(7) °

γ = 90 °

V = 798.45(9) Å^3

Z = 2, $R(F)$ = 0.0721

$R_w(F^2)$= 0.1608

Crystal size: 0.7 x 0.10 x 0.07 mm

Calculated density: 1.280 g/cm^3

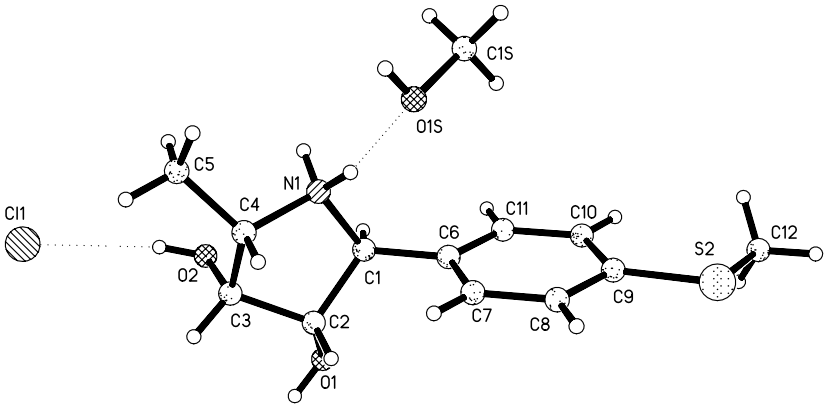
2θ-Range for data collection: 5.99 - 64.96 °

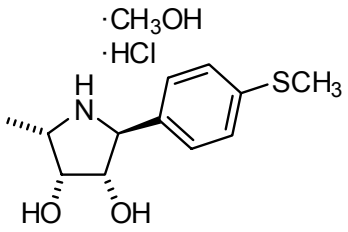
Independent reflections: 2513

Observed reflections: 2970

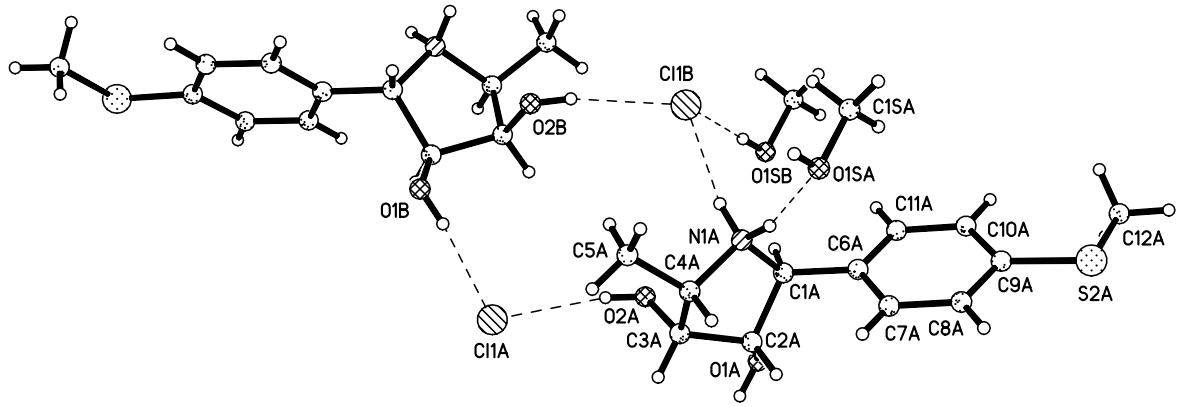
Contributed reflections to refinement: 2513

Refined parameters: 176



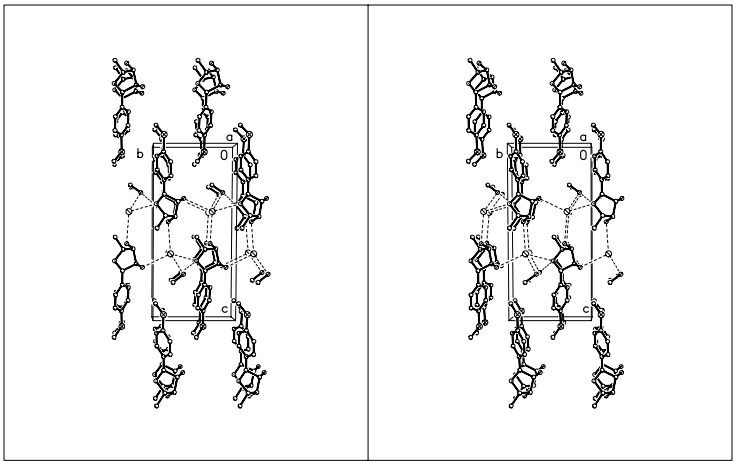


101 ·HCl

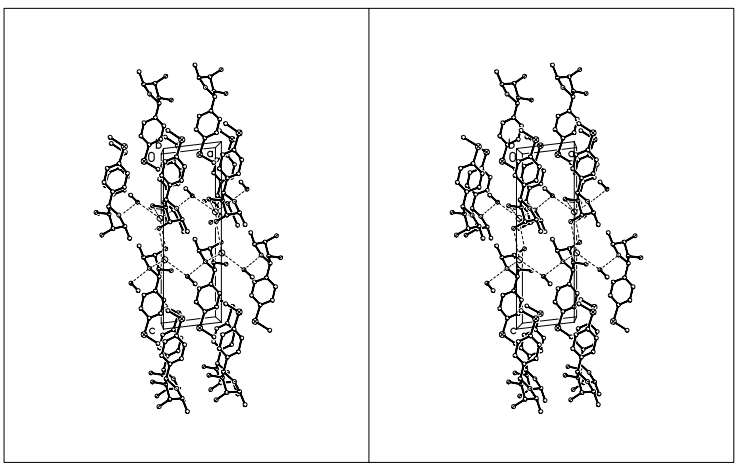


Unit cell; View along the b-axis (b) and c-axis (c):

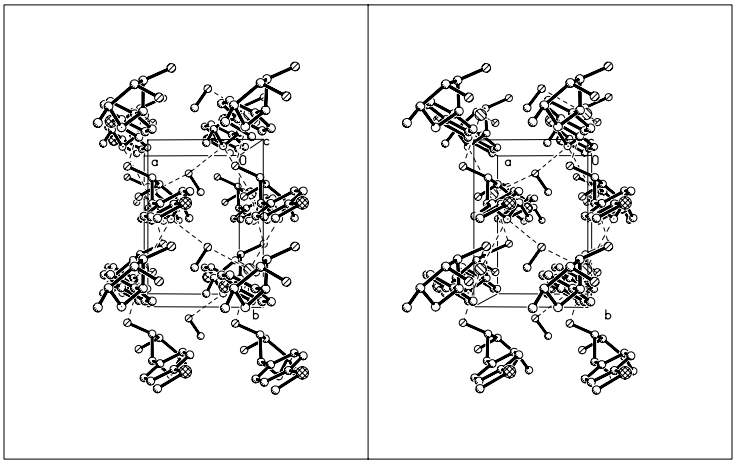
(a)



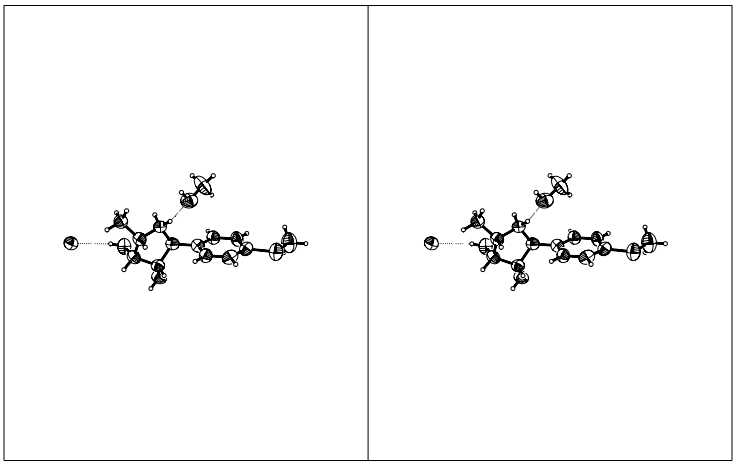
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

N(1)-C(4)	1.513(9)	C(4)-H(4)	0.9800
N(1)-C(1)	1.525(9)	C(5)-H(5A)	0.9600
N(1)-H(1A)	0.9000	C(5)-H(5B)	0.9600
N(1)-H(1B)	0.9000	C(5)-H(5C)	0.9600
O(1)-C(2)	1.429(8)	C(6)-C(11)	1.375(10)
O(1)-H(1C)	0.8200	C(6)-C(7)	1.379(10)
C(1)-C(6)	1.502(9)	C(7)-C(8)	1.395(9)
C(1)-C(2)	1.528(10)	C(7)-H(7)	0.9300
C(1)-H(1)	0.9800	C(8)-C(9)	1.395(11)
S(2)-C(9)	1.742(8)	C(8)-H(8)	0.9300
S(2)-C(12)	1.770(11)	C(9)-C(10)	1.390(10)
O(2)-C(3)	1.413(8)	C(10)-C(11)	1.393(10)
O(2)-H(2A)	0.8200	C(10)-H(10)	0.9300
C(2)-C(3)	1.527(10)	C(11)-H(11)	0.9300
C(2)-H(2)	0.9800	C(12)-H(12A)	0.9600
C(3)-C(4)	1.532(10)	C(12)-H(12B)	0.9600
C(3)-H(3)	0.9800	C(12)-H(12C)	0.9600
C(4)-C(5)	1.500(10)	O(1S)-C(1S)	1.432(12)

O(1S)-H(1S)	0.8200
C(1S)-H(1S1)	0.9600
C(1S)-H(1S2)	0.9600
C(1S)-H(1S3)	0.9600
C(4)-N(1)-C(1)	109.7(5)
C(4)-N(1)-H(1A)	109.7
C(1)-N(1)-H(1A)	109.7
C(4)-N(1)-H(1B)	109.7
C(1)-N(1)-H(1B)	109.7
H(1A)-N(1)-H(1B)	108.2
C(2)-O(1)-H(1C)	109.5
C(6)-C(1)-N(1)	111.5(6)
C(6)-C(1)-C(2)	117.3(6)
N(1)-C(1)-C(2)	102.3(5)
C(6)-C(1)-H(1)	108.4
N(1)-C(1)-H(1)	108.4
C(2)-C(1)-H(1)	108.4
C(9)-S(2)-C(12)	103.0(5)
C(3)-O(2)-H(2A)	109.5
O(1)-C(2)-C(3)	115.3(6)
O(1)-C(2)-C(1)	108.2(6)
C(3)-C(2)-C(1)	104.8(6)
O(1)-C(2)-H(2)	109.5
C(3)-C(2)-H(2)	109.5
C(1)-C(2)-H(2)	109.5
O(2)-C(3)-C(2)	108.5(6)
O(2)-C(3)-C(4)	110.9(6)
C(2)-C(3)-C(4)	101.1(6)
O(2)-C(3)-H(3)	111.9
C(2)-C(3)-H(3)	111.9
C(4)-C(3)-H(3)	111.9
C(5)-C(4)-N(1)	110.9(6)
C(5)-C(4)-C(3)	118.2(6)
N(1)-C(4)-C(3)	100.4(5)
C(5)-C(4)-H(4)	108.9
N(1)-C(4)-H(4)	108.9
C(3)-C(4)-H(4)	108.9
C(4)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
C(4)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(11)-C(6)-C(7)	119.4(7)
C(11)-C(6)-C(1)	119.1(7)
C(7)-C(6)-C(1)	121.5(6)
C(6)-C(7)-C(8)	119.8(7)
C(6)-C(7)-H(7)	120.1
C(8)-C(7)-H(7)	120.1
C(9)-C(8)-C(7)	122.0(7)
C(9)-C(8)-H(8)	119.0
C(7)-C(8)-H(8)	119.0
C(10)-C(9)-C(8)	116.7(7)
C(10)-C(9)-S(2)	126.2(6)
C(8)-C(9)-S(2)	117.1(6)
C(9)-C(10)-C(11)	121.6(7)
C(9)-C(10)-H(10)	119.2
C(11)-C(10)-H(10)	119.2
C(6)-C(11)-C(10)	120.5(7)
C(6)-C(11)-H(11)	119.8
C(10)-C(11)-H(11)	119.8
S(2)-C(12)-H(12A)	109.5
S(2)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
S(2)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(1S)-O(1S)-H(1S)	109.5
O(1S)-C(1S)-H(1S1)	109.5
O(1S)-C(1S)-H(1S2)	109.5
H(1S1)-C(1S)-H(1S2)	109.5
O(1S)-C(1S)-H(1S3)	109.5
H(1S1)-C(1S)-H(1S3)	109.5
H(1S2)-C(1S)-H(1S3)	109.5

Torsion angles [°]

C(4)-N(1)-C(1)-C(6)	-123.8(6)
C(4)-N(1)-C(1)-C(2)	2.5(7)
C(6)-C(1)-C(2)-O(1)	-88.3(8)
N(1)-C(1)-C(2)-O(1)	149.3(6)
C(6)-C(1)-C(2)-C(3)	148.2(6)
N(1)-C(1)-C(2)-C(3)	25.8(7)
O(1)-C(2)-C(3)-O(2)	-46.6(9)
C(1)-C(2)-C(3)-O(2)	72.2(7)
O(1)-C(2)-C(3)-C(4)	-163.2(6)
C(1)-C(2)-C(3)-C(4)	-44.4(7)
C(1)-N(1)-C(4)-C(5)	-155.0(6)
C(1)-N(1)-C(4)-C(3)	-29.2(7)
O(2)-C(3)-C(4)-C(5)	49.7(9)
C(2)-C(3)-C(4)-C(5)	164.6(7)
O(2)-C(3)-C(4)-N(1)	-71.0(7)
C(2)-C(3)-C(4)-N(1)	43.9(7)
N(1)-C(1)-C(6)-C(11)	-112.9(8)
C(2)-C(1)-C(6)-C(11)	129.6(8)
N(1)-C(1)-C(6)-C(7)	67.2(10)
C(2)-C(1)-C(6)-C(7)	-50.4(10)
C(11)-C(6)-C(7)-C(8)	-0.3(13)
C(1)-C(6)-C(7)-C(8)	179.6(8)
C(6)-C(7)-C(8)-C(9)	0.3(14)
C(7)-C(8)-C(9)-C(10)	0.1(13)
C(7)-C(8)-C(9)-S(2)	179.8(7)
C(12)-S(2)-C(9)-C(10)	-6.8(10)
C(12)-S(2)-C(9)-C(8)	173.5(8)
C(8)-C(9)-C(10)-C(11)	-0.5(13)
S(2)-C(9)-C(10)-C(11)	179.8(7)
C(7)-C(6)-C(11)-C(10)	0.0(13)
C(1)-C(6)-C(11)-C(10)	180.0(7)
C(9)-C(10)-C(11)-C(6)	0.5(13)

H-Bond lengths [Å] and angles [°]

N1-O1S	2.7650(0.0089)
H1A-O1S	1.8919
N1-Cl1_$1	3.1522(0.0061)
H1B-Cl1_$1	2.2641
O1-Cl1_$3	3.1422(0.0059)
H1C-Cl1_$3	2.3790

O2-Cl1	3.1566(0.0053)	N1-H1B-Cl1_$1	168.96
H2A-Cl1	2.3455	O1-H1C-Cl1_$3	155.19
O1S-Cl1_$2	3.0624(0.0068)	O2-H2A-Cl1	170.21
H1S-Cl1_$2	2.2451	O1S-H1S-Cl1_$2	174.50
N1-H1A-O1S	162.96		

9.17 (2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-5-methyl-2-(4-phenoxyphenyl)-pyrrolidine hydrochloride (102·HCl)

$C_{17}H_{20}ClNO_3$

monoclinic, $P2_1$

a = 8.917(3) Å

b = 16.415(5) Å

c = 11.287(3) Å

α = 90 °

β = 95.29(2) °

γ = 90 °

V = 1645.1(8) Å^3

Z = 4, $R(F)$ = 0.0508

$R_w(F^2)$ = 0.1027

Crystal size: 1.0 x 0.5 x 0.1 mm

Calculated density: 1.299 g/cm^3

2θ-Range for data collection: 1.81 – 27.50 °

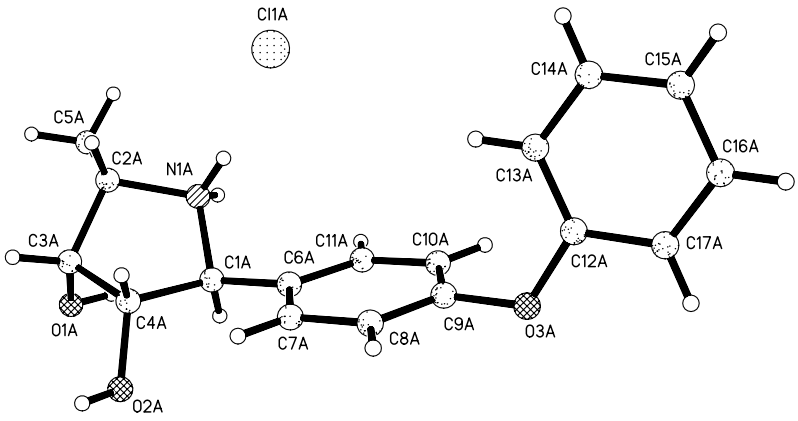
Independent reflections: 3923

Observed reflections: 3048

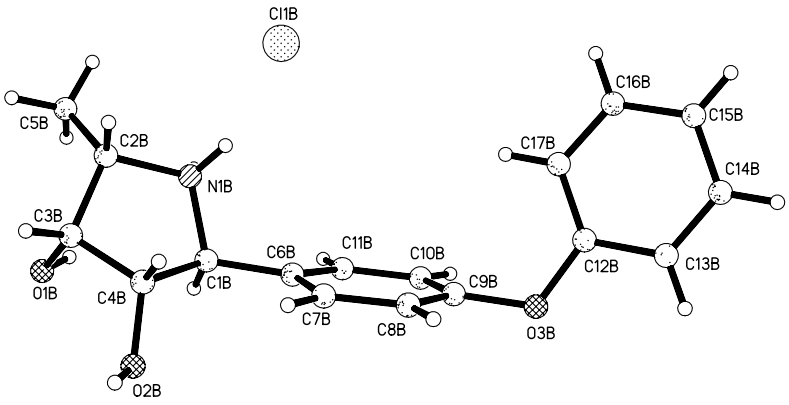
Contributed reflections to refinement: 3923

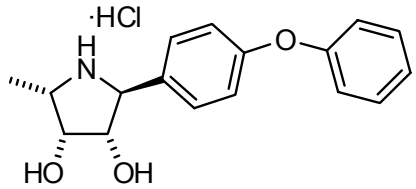
Refined parameters: 558

molecule A



molecule B

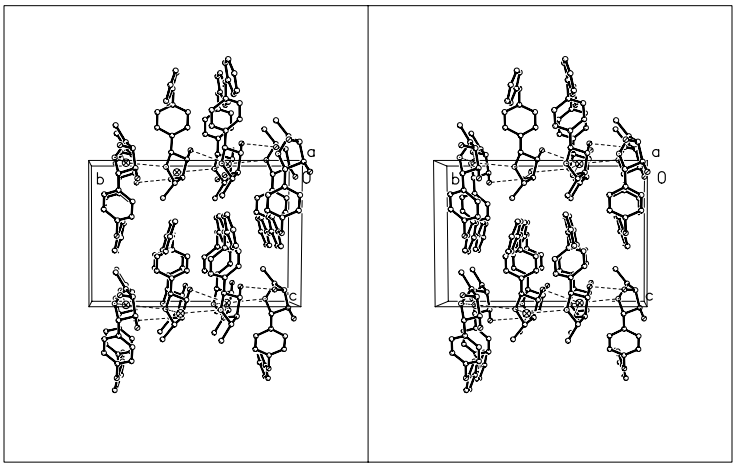




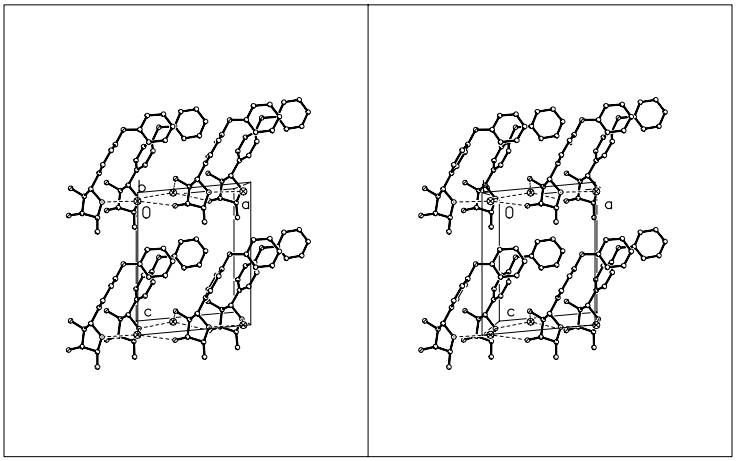
102·HCl

Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

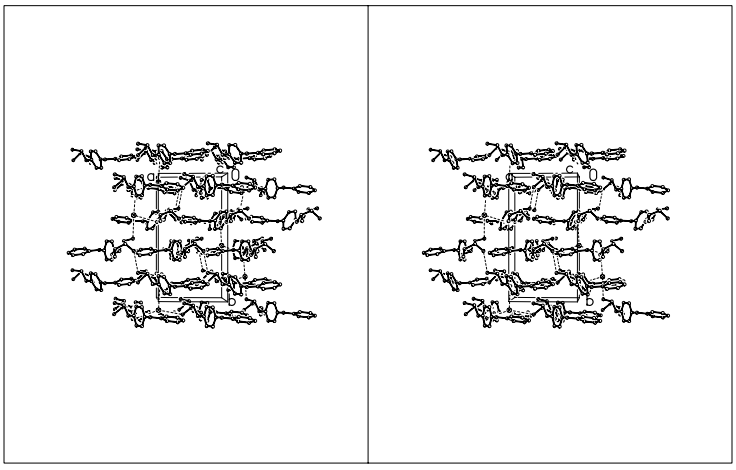
(a)



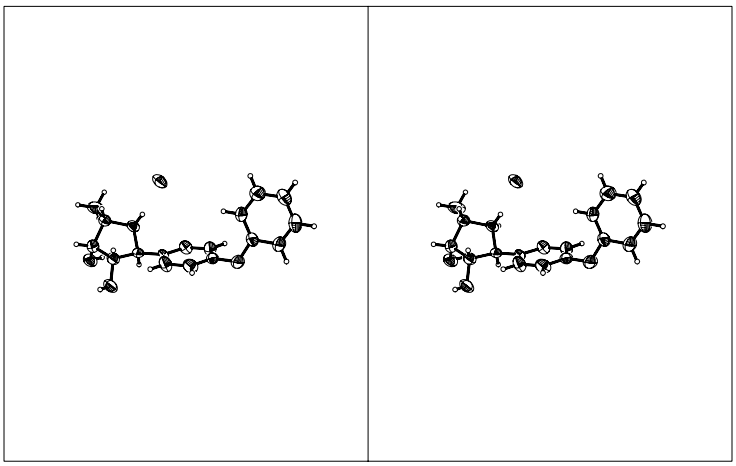
(b)



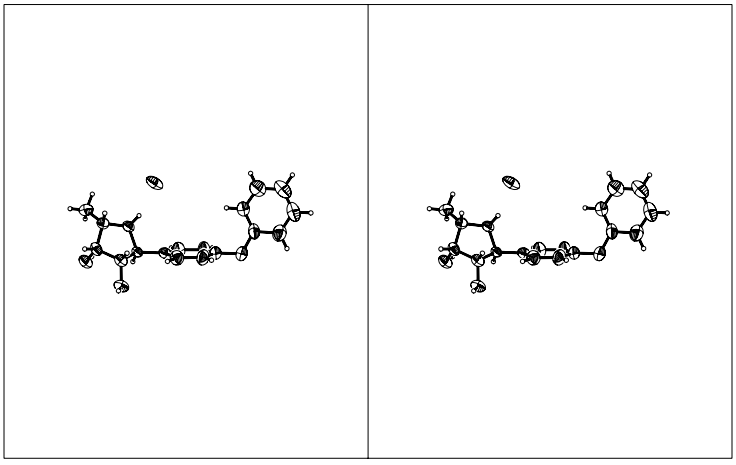
(c)



ORTEP Structure, molecule A



ORTEP Structure, molecule B



Bond lengths [Å] and angles [°]

O(1A)-C(3A)	1.417(6)	C(5A)-H(5A1)	0.96(7)
O(1A)-H(1A3)	0.69(5)	C(5A)-H(5A2)	1.08(8)
N(1A)-C(1A)	1.520(5)	C(5A)-H(5A3)	0.92(6)
N(1A)-C(2A)	1.531(6)	C(6A)-C(7A)	1.378(6)
N(1A)-H(1A1)	0.93(4)	C(6A)-C(11A)	1.387(6)
N(1A)-H(1A2)	0.89(5)	C(7A)-C(8A)	1.393(6)
C(1A)-C(4A)	1.513(6)	C(7A)-H(7A)	0.95(4)
C(1A)-C(6A)	1.515(5)	C(8A)-C(9A)	1.353(7)
C(1A)-H(1A)	0.95(4)	C(8A)-H(8A)	0.87(5)
C(2A)-C(5A)	1.510(8)	C(9A)-C(10A)	1.371(7)
C(2A)-C(3A)	1.514(6)	C(10A)-C(11A)	1.388(6)
C(2A)-H(2A)	1.00(5)	C(10A)-H(10A)	0.93(6)
O(2A)-C(4A)	1.413(5)	C(11A)-H(11A)	0.92(5)
O(2A)-H(2A1)	0.96(7)	C(12A)-C(13A)	1.371(6)
O(3A)-C(12A)	1.383(5)	C(12A)-C(17A)	1.375(6)
O(3A)-C(9A)	1.396(5)	C(13A)-C(14A)	1.371(7)
C(3A)-C(4A)	1.533(6)	C(13A)-H(13A)	0.89(5)
C(3A)-H(3A)	0.99(5)	C(14A)-C(15A)	1.377(8)
C(4A)-H(4A)	1.04(4)	C(14A)-H(14A)	0.95(6)

C(15A)-C(16A)	1.375(8)	N(1A)-C(1A)-H(1A)	104(2)
C(15A)-H(15A)	0.89(6)	C(5A)-C(2A)-C(3A)	116.5(4)
C(16A)-C(17A)	1.369(7)	C(5A)-C(2A)-N(1A)	110.9(5)
C(16A)-H(16A)	0.92(6)	C(3A)-C(2A)-N(1A)	103.4(3)
C(17A)-H(17A)	1.02(6)	C(5A)-C(2A)-H(2A)	112(3)
O(1B)-C(3B)	1.424(5)	C(3A)-C(2A)-H(2A)	107(3)
O(1B)-H(1B3)	0.76(6)	N(1A)-C(2A)-H(2A)	106(3)
N(1B)-C(1B)	1.522(5)	C(4A)-O(2A)-H(2A1)	104(4)
N(1B)-C(2B)	1.527(5)	C(12A)-O(3A)-C(9A)	118.7(3)
N(1B)-H(1B1)	1.01(5)	O(1A)-C(3A)-C(2A)	108.8(4)
N(1B)-H(1B2)	0.98(5)	O(1A)-C(3A)-C(4A)	111.3(4)
C(1B)-C(6B)	1.503(6)	C(2A)-C(3A)-C(4A)	101.8(3)
C(1B)-C(4B)	1.522(6)	O(1A)-C(3A)-H(3A)	113(3)
C(1B)-H(1B)	0.88(4)	C(2A)-C(3A)-H(3A)	109(3)
O(2B)-C(4B)	1.399(5)	C(4A)-C(3A)-H(3A)	112(3)
O(2B)-H(2B1)	1.02(7)	O(2A)-C(4A)-C(1A)	109.0(4)
C(2B)-C(5B)	1.508(8)	O(2A)-C(4A)-C(3A)	113.7(3)
C(2B)-C(3B)	1.514(6)	C(1A)-C(4A)-C(3A)	103.1(4)
C(2B)-H(2B)	0.98(4)	O(2A)-C(4A)-H(4A)	112(2)
O(3B)-C(12B)	1.384(6)	C(1A)-C(4A)-H(4A)	113(2)
O(3B)-C(9B)	1.401(5)	C(3A)-C(4A)-H(4A)	106(2)
C(3B)-C(4B)	1.524(6)	C(2A)-C(5A)-H(5A1)	108(4)
C(3B)-H(3B)	0.96(5)	C(2A)-C(5A)-H(5A2)	101(4)
C(4B)-H(4B)	1.03(4)	H(5A1)-C(5A)-H(5A2)	118(5)
C(5B)-H(5B1)	1.05(8)	C(2A)-C(5A)-H(5A3)	115(4)
C(5B)-H(5B2)	0.95(6)	H(5A1)-C(5A)-H(5A3)	95(6)
C(5B)-H(5B3)	0.97(8)	H(5A2)-C(5A)-H(5A3)	120(6)
C(6B)-C(7B)	1.378(7)	C(7A)-C(6A)-C(11A)	118.9(4)
C(6B)-C(11B)	1.393(7)	C(7A)-C(6A)-C(1A)	121.9(4)
C(7B)-C(8B)	1.409(7)	C(11A)-C(6A)-C(1A)	119.2(4)
C(7B)-H(7B)	0.92(6)	C(6A)-C(7A)-C(8A)	119.9(5)
C(8B)-C(9B)	1.361(8)	C(6A)-C(7A)-H(7A)	122(3)
C(8B)-H(8B)	1.01(6)	C(8A)-C(7A)-H(7A)	118(3)
C(9B)-C(10B)	1.351(8)	C(9A)-C(8A)-C(7A)	120.4(5)
C(10B)-C(11B)	1.389(7)	C(9A)-C(8A)-H(8A)	124(3)
C(10B)-H(10B)	0.78(5)	C(7A)-C(8A)-H(8A)	115(3)
C(11B)-H(11B)	0.95(5)	C(8A)-C(9A)-C(10A)	120.9(4)
C(12B)-C(17B)	1.373(7)	C(8A)-C(9A)-O(3A)	118.8(5)
C(12B)-C(13B)	1.382(7)	C(10A)-C(9A)-O(3A)	120.2(5)
C(13B)-C(14B)	1.371(9)	C(9A)-C(10A)-C(11A)	119.2(5)
C(13B)-H(13B)	0.95(5)	C(9A)-C(10A)-H(10A)	119(3)
C(14B)-C(15B)	1.377(11)	C(11A)-C(10A)-H(10A)	122(3)
C(14B)-H(14B)	0.90(6)	C(6A)-C(11A)-C(10A)	120.7(5)
C(15B)-C(16B)	1.366(10)	C(6A)-C(11A)-H(11A)	122(3)
C(15B)-H(15B)	0.89(8)	C(10A)-C(11A)-H(11A)	117(3)
C(16B)-C(17B)	1.380(8)	C(13A)-C(12A)-C(17A)	120.6(4)
C(16B)-H(16B)	1.03(9)	C(13A)-C(12A)-O(3A)	123.7(4)
C(17B)-H(17B)	0.97(6)	C(17A)-C(12A)-O(3A)	115.6(4)
C(3A)-O(1A)-H(1A3)	108(5)	C(12A)-C(13A)-C(14A)	118.9(4)
C(1A)-N(1A)-C(2A)	108.3(3)	C(12A)-C(13A)-H(13A)	122(3)
C(1A)-N(1A)-H(1A1)	112(2)	C(14A)-C(13A)-H(13A)	119(3)
C(2A)-N(1A)-H(1A1)	109(2)	C(13A)-C(14A)-C(15A)	120.9(5)
C(1A)-N(1A)-H(1A2)	112(3)	C(13A)-C(14A)-H(14A)	120(4)
C(2A)-N(1A)-H(1A2)	105(3)	C(15A)-C(14A)-H(14A)	119(4)
H(1A1)-N(1A)-H(1A2)	110(4)	C(16A)-C(15A)-C(14A)	119.8(5)
C(4A)-C(1A)-C(6A)	118.2(4)	C(16A)-C(15A)-H(15A)	125(4)
C(4A)-C(1A)-N(1A)	102.8(3)	C(14A)-C(15A)-H(15A)	115(4)
C(6A)-C(1A)-N(1A)	111.8(3)	C(17A)-C(16A)-C(15A)	119.6(5)
C(4A)-C(1A)-H(1A)	109(2)	C(17A)-C(16A)-H(16A)	122(4)
C(6A)-C(1A)-H(1A)	110(2)	C(15A)-C(16A)-H(16A)	118(4)

C(16A)-C(17A)-C(12A)	120.3(5)
C(16A)-C(17A)-H(17A)	118(3)
C(12A)-C(17A)-H(17A)	122(3)
C(3B)-O(1B)-H(1B3)	102(5)
C(1B)-N(1B)-C(2B)	108.6(3)
C(1B)-N(1B)-H(1B1)	109(2)
C(2B)-N(1B)-H(1B1)	114(2)
C(1B)-N(1B)-H(1B2)	114(3)
C(2B)-N(1B)-H(1B2)	106(3)
H(1B1)-N(1B)-H(1B2)	106(4)
C(6B)-C(1B)-N(1B)	112.2(3)
C(6B)-C(1B)-C(4B)	118.9(4)
N(1B)-C(1B)-C(4B)	101.8(3)
C(6B)-C(1B)-H(1B)	113(3)
N(1B)-C(1B)-H(1B)	103(2)
C(4B)-C(1B)-H(1B)	106(3)
C(4B)-O(2B)-H(2B1)	108(3)
C(5B)-C(2B)-C(3B)	117.9(4)
C(5B)-C(2B)-N(1B)	111.2(5)
C(3B)-C(2B)-N(1B)	103.5(3)
C(5B)-C(2B)-H(2B)	113(2)
C(3B)-C(2B)-H(2B)	107(2)
N(1B)-C(2B)-H(2B)	103(2)
C(12B)-O(3B)-C(9B)	117.7(4)
O(1B)-C(3B)-C(2B)	109.5(4)
O(1B)-C(3B)-C(4B)	111.9(4)
C(2B)-C(3B)-C(4B)	102.5(3)
O(1B)-C(3B)-H(3B)	113(3)
C(2B)-C(3B)-H(3B)	109(3)
C(4B)-C(3B)-H(3B)	110(3)
O(2B)-C(4B)-C(1B)	109.4(4)
O(2B)-C(4B)-C(3B)	114.6(3)
C(1B)-C(4B)-C(3B)	102.6(4)
O(2B)-C(4B)-H(4B)	105(2)
C(1B)-C(4B)-H(4B)	115(2)
C(3B)-C(4B)-H(4B)	110(2)
C(2B)-C(5B)-H(5B1)	117(4)
C(2B)-C(5B)-H(5B2)	106(4)
H(5B1)-C(5B)-H(5B2)	113(6)
C(2B)-C(5B)-H(5B3)	118(4)
H(5B1)-C(5B)-H(5B3)	93(6)
H(5B2)-C(5B)-H(5B3)	109(6)
C(7B)-C(6B)-C(11B)	118.8(4)
C(7B)-C(6B)-C(1B)	122.2(4)
C(11B)-C(6B)-C(1B)	119.0(4)
C(6B)-C(7B)-C(8B)	120.5(5)
C(6B)-C(7B)-H(7B)	122(4)
C(8B)-C(7B)-H(7B)	118(4)
C(9B)-C(8B)-C(7B)	119.1(5)
C(9B)-C(8B)-H(8B)	124(3)
C(7B)-C(8B)-H(8B)	116(3)
C(10B)-C(9B)-C(8B)	121.0(4)
C(10B)-C(9B)-O(3B)	121.2(5)
C(8B)-C(9B)-O(3B)	117.6(5)
C(9B)-C(10B)-C(11B)	120.9(5)
C(9B)-C(10B)-H(10B)	122(4)
C(11B)-C(10B)-H(10B)	117(4)
C(10B)-C(11B)-C(6B)	119.5(5)
C(10B)-C(11B)-H(11B)	120(3)
C(6B)-C(11B)-H(11B)	120(3)
C(17B)-C(12B)-C(13B)	120.4(5)
C(17B)-C(12B)-O(3B)	124.1(4)
C(13B)-C(12B)-O(3B)	115.5(5)
C(14B)-C(13B)-C(12B)	119.3(6)
C(14B)-C(13B)-H(13B)	125(3)
C(12B)-C(13B)-H(13B)	116(3)
C(13B)-C(14B)-C(15B)	121.1(6)
C(13B)-C(14B)-H(14B)	121(4)
C(15B)-C(14B)-H(14B)	118(4)
C(16B)-C(15B)-C(14B)	118.8(7)
C(16B)-C(15B)-H(15B)	120(6)
C(14B)-C(15B)-H(15B)	118(6)
C(15B)-C(16B)-C(17B)	121.4(7)
C(15B)-C(16B)-H(16B)	118(5)
C(17B)-C(16B)-H(16B)	120(5)
C(12B)-C(17B)-C(16B)	119.0(6)
C(12B)-C(17B)-H(17B)	120(3)
C(16B)-C(17B)-H(17B)	121(3)

Torsion angles [°]

C(2A)-N(1A)-C(1A)-C(4A)	-14.6(4)
C(2A)-N(1A)-C(1A)-C(6A)	-142.3(4)
C(1A)-N(1A)-C(2A)-C(5A)	-139.3(4)
C(1A)-N(1A)-C(2A)-C(3A)	-13.6(5)
C(5A)-C(2A)-C(3A)-O(1A)	40.3(7)
N(1A)-C(2A)-C(3A)-O(1A)	-81.7(4)
C(5A)-C(2A)-C(3A)-C(4A)	157.9(6)
N(1A)-C(2A)-C(3A)-C(4A)	36.0(5)
C(6A)-C(1A)-C(4A)-O(2A)	-78.3(4)
N(1A)-C(1A)-C(4A)-O(2A)	158.1(3)
C(6A)-C(1A)-C(4A)-C(3A)	160.6(3)
N(1A)-C(1A)-C(4A)-C(3A)	37.0(4)
O(1A)-C(3A)-C(4A)-O(2A)	-48.2(6)
C(2A)-C(3A)-C(4A)-O(2A)	-164.0(4)
O(1A)-C(3A)-C(4A)-C(1A)	69.7(4)
C(2A)-C(3A)-C(4A)-C(1A)	-46.2(5)
C(4A)-C(1A)-C(6A)-C(7A)	8.0(6)
N(1A)-C(1A)-C(6A)-C(7A)	127.0(4)
C(4A)-C(1A)-C(6A)-C(11A)	-174.9(4)
N(1A)-C(1A)-C(6A)-C(11A)	-56.0(5)
C(11A)-C(6A)-C(7A)-C(8A)	1.1(7)
C(1A)-C(6A)-C(7A)-C(8A)	178.1(4)
C(6A)-C(7A)-C(8A)-C(9A)	-0.5(8)
C(7A)-C(8A)-C(9A)-C(10A)	0.6(8)
C(7A)-C(8A)-C(9A)-O(3A)	-176.0(5)
C(12A)-O(3A)-C(9A)-C(8A)	-109.7(6)
C(12A)-O(3A)-C(9A)-C(10A)	73.7(6)
C(8A)-C(9A)-C(10A)-C(11A)	-1.3(7)
O(3A)-C(9A)-C(10A)-C(11A)	175.2(4)
C(7A)-C(6A)-C(11A)-C(10A)	-1.9(7)
C(1A)-C(6A)-C(11A)-C(10A)	-179.0(4)
C(9A)-C(10A)-C(11A)-C(6A)	2.0(7)
C(9A)-O(3A)-C(12A)-C(13A)	12.7(8)
C(9A)-O(3A)-C(12A)-C(17A)	-171.7(5)
C(17A)-C(12A)-C(13A)-C(14A)	0.2(8)
O(3A)-C(12A)-C(13A)-C(14A)	175.6(6)
C(12A)-C(13A)-C(14A)-C(15A)	0.5(10)
C(13A)-C(14A)-C(15A)-C(16A)	-0.3(11)

C(14A)-C(15A)-C(16A)-C(17A)	-0.5(11)
C(15A)-C(16A)-C(17A)-C(12A)	1.1(10)
C(13A)-C(12A)-C(17A)-C(16A)	-1.0(9)
O(3A)-C(12A)-C(17A)-C(16A)	-176.8(6)
C(2B)-N(1B)-C(1B)-C(6B)	-147.0(4)
C(2B)-N(1B)-C(1B)-C(4B)	-18.8(4)
C(1B)-N(1B)-C(2B)-C(5B)	-136.8(4)
C(1B)-N(1B)-C(2B)-C(3B)	-9.3(4)
C(5B)-C(2B)-C(3B)-O(1B)	37.9(6)
N(1B)-C(2B)-C(3B)-O(1B)	-85.3(4)
C(5B)-C(2B)-C(3B)-C(4B)	156.8(5)
N(1B)-C(2B)-C(3B)-C(4B)	33.7(4)
C(6B)-C(1B)-C(4B)-O(2B)	-74.5(5)
N(1B)-C(1B)-C(4B)-O(2B)	161.6(3)
C(6B)-C(1B)-C(4B)-C(3B)	163.4(3)
N(1B)-C(1B)-C(4B)-C(3B)	39.5(4)
O(1B)-C(3B)-C(4B)-O(2B)	-47.7(5)
C(2B)-C(3B)-C(4B)-O(2B)	-164.9(4)
O(1B)-C(3B)-C(4B)-C(1B)	70.9(4)
C(2B)-C(3B)-C(4B)-C(1B)	-46.4(4)
N(1B)-C(1B)-C(6B)-C(7B)	108.0(5)
C(4B)-C(1B)-C(6B)-C(7B)	-10.5(6)
N(1B)-C(1B)-C(6B)-C(11B)	-72.4(5)
C(4B)-C(1B)-C(6B)-C(11B)	169.1(4)
C(11B)-C(6B)-C(7B)-C(8B)	3.8(7)
C(1B)-C(6B)-C(7B)-C(8B)	-176.6(4)
C(6B)-C(7B)-C(8B)-C(9B)	-0.6(8)
C(7B)-C(8B)-C(9B)-C(10B)	-2.9(8)
C(7B)-C(8B)-C(9B)-O(3B)	-177.7(5)
C(12B)-O(3B)-C(9B)-C(10B)	85.6(7)
C(12B)-O(3B)-C(9B)-C(8B)	-99.7(6)
C(8B)-C(9B)-C(10B)-C(11B)	3.1(8)
O(3B)-C(9B)-C(10B)-C(11B)	177.7(5)
C(9B)-C(10B)-C(11B)-C(6B)	0.2(9)
C(7B)-C(6B)-C(11B)-C(10B)	-3.6(7)
C(1B)-C(6B)-C(11B)-C(10B)	176.7(5)
C(9B)-O(3B)-C(12B)-C(17B)	-12.6(9)
C(9B)-O(3B)-C(12B)-C(13B)	168.8(5)
C(17B)-C(12B)-C(13B)-C(14B)	0.8(10)
O(3B)-C(12B)-C(13B)-C(14B)	179.5(6)
C(12B)-C(13B)-C(14B)-C(15B)	-1.0(12)
C(13B)-C(14B)-C(15B)-C(16B)	-0.3(13)
C(14B)-C(15B)-C(16B)-C(17B)	1.8(13)
C(13B)-C(12B)-C(17B)-C(16B)	0.6(9)
O(3B)-C(12B)-C(17B)-C(16B)	-177.9(6)
C(15B)-C(16B)-C(17B)-C(12B)	-1.9(11)

H-Bond lengths [Å] and angles [°]

N1A-Cl1B	3.1345(0.0042)
H1A1-Cl1B	2.2284(0.0420)
N1A-Cl1A	3.1561(0.0035)
H1A2-Cl1A	2.2655(0.0528)
O2A-O1B_$1	2.8089(0.0054)
H2A1-O1B_$1	1.8580(0.0672)
N1B-Cl1A_$2	3.1761(0.0041)
H1B1-Cl1A_$2	2.1919(0.0473)
N1B-Cl1B	3.1346(0.0035)
H1B2-Cl1B	2.1729(0.0474)
O1B-Cl1B_$4	3.2387(0.0035)
H1B3-Cl1B_$4	2.5683(0.0614)
O2B-Cl1A_$4	3.2061(0.0041)
H2B1-Cl1A_$4	2.2763(0.0641)
O1A-Cl1A_$4	3.2593(0.0035)
H1A3-Cl1A_$4	2.6094(0.0522)
N1A-H1A1-Cl1B	165.94(3.12)
N1A-H1A2-Cl1A	178.29(4.84)
O2A-H2A1-O1B_$1	172.22(5.33)
N1B-H1B1-Cl1A_$2	164.73(3.46)
N1B-H1B2-Cl1B	166.78(3.69)
O1B-H1B3-Cl1B_$4	148.68(6.07)
O2B-H2B1-Cl1A_$4	151.59(4.71)
O1A-H1A3-Cl1A_$4	158.78(5.52)

9.18 (2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-5-methyl-2-(4-phenoxyphenyl)-pyrrolidine hydrochloride (102·HBr)

$C_{17}H_{20}BrNO_3$

monoclinic, $P2_1$

a = 9.2010(13) Å

b = 16.725(2) Å

c = 11.1005(14) Å

α = 90 °

β = 95.225(11) °

γ = 90 °

V = 1701.1(4) Å^3

Z = 4, $R(F)$ = 0.0433

$R_w(F^2)$ = 0.0900

Crystal size: 0.95 x 0.60 x 0.30 mm

Calculated density: 1.430 g/cm^3

2θ-Range for data collection: 1.84 – 27.49 °

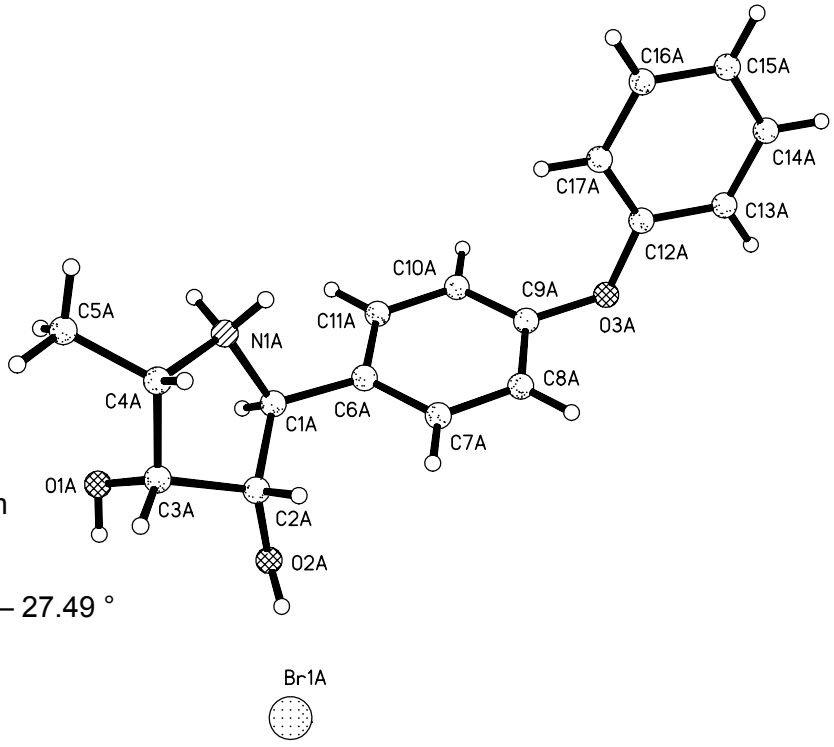
Independent reflections: 7784

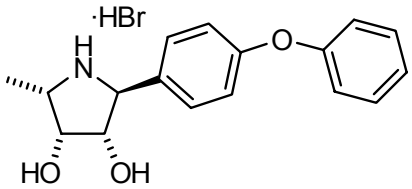
Observed reflections: 5811

Contributed reflections to refinement: 7784

Refined parameters: 402

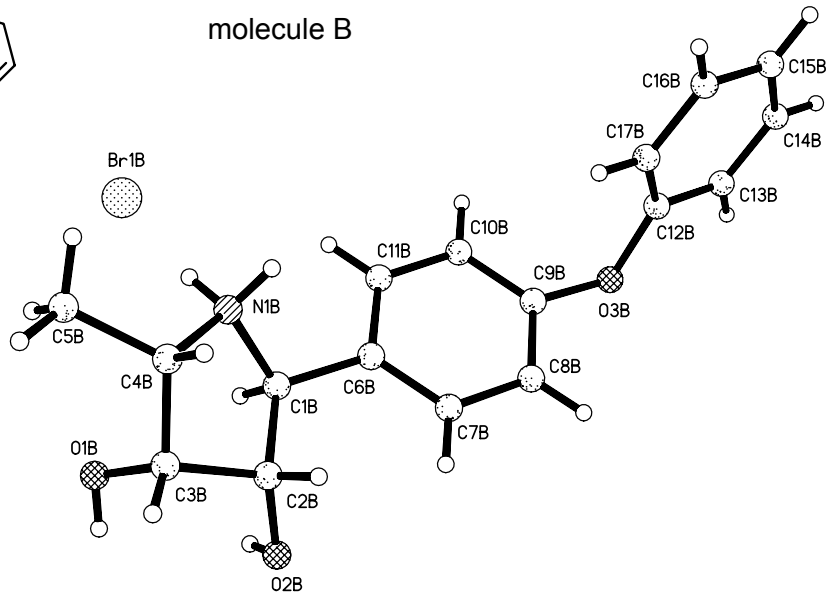
molecule A





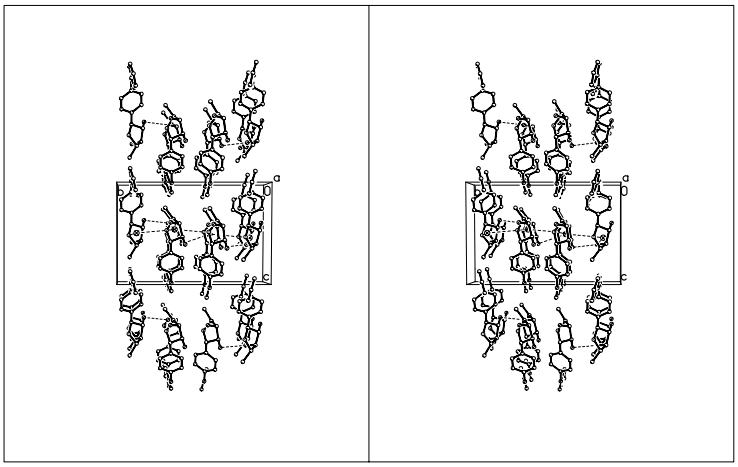
102·HBr

molecule B

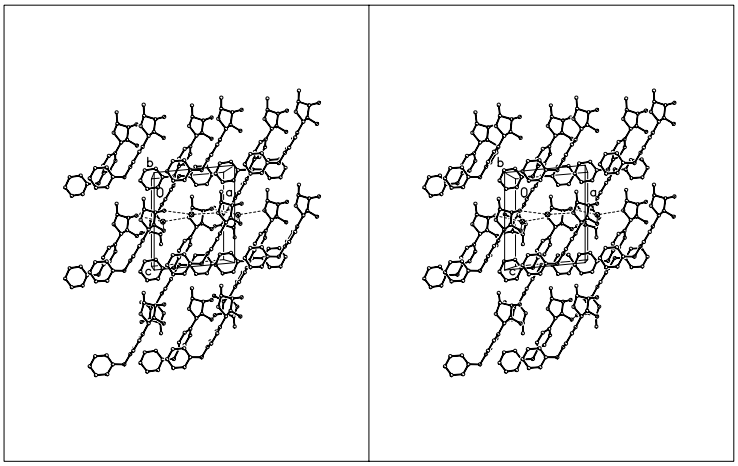


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

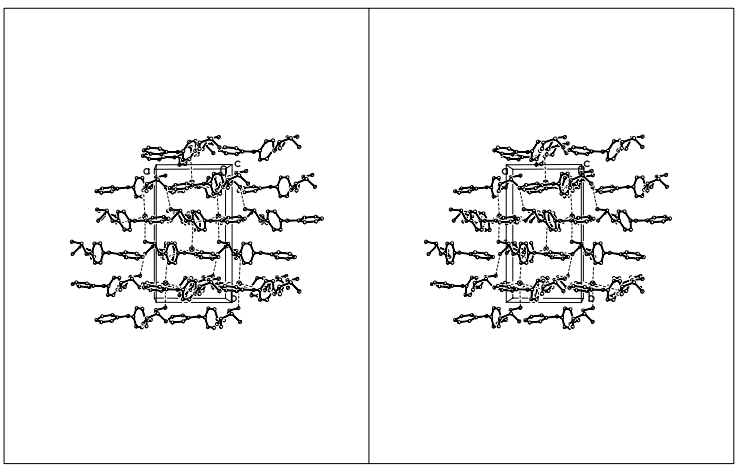
(a)



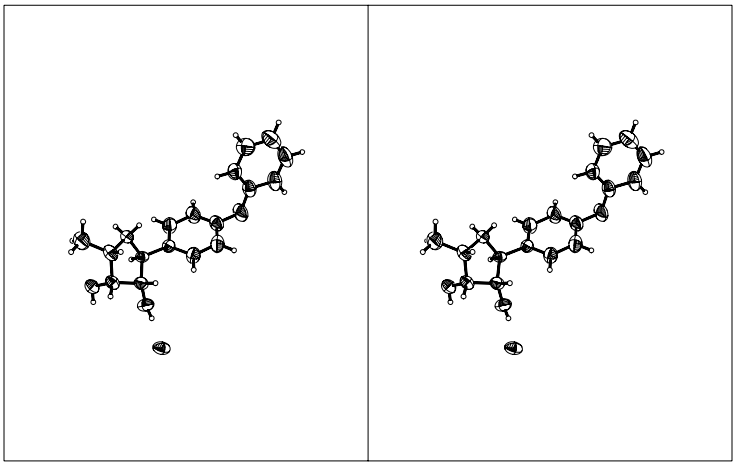
(b)



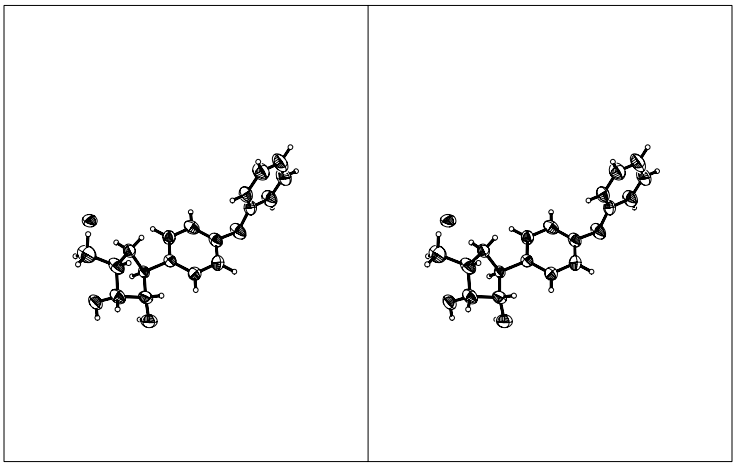
(c)



ORTEP Structure, molecule A



ORTEP Structure, molecule B



Bond lengths [Å] and angles [°]

C(1A)-C(6A)	1.489(6)	C(3A)-H(3A)	0.98
C(1A)-N(1A)	1.530(5)	C(4A)-C(5A)	1.511(7)
C(1A)-C(2A)	1.532(5)	C(4A)-H(4A)	0.98
C(1A)-H(1A)	0.98	C(5A)-H(5A1)	0.96
O(1A)-C(3A)	1.423(5)	C(5A)-H(5A2)	0.96
O(1A)-H(1AA)	0.82	C(5A)-H(5A3)	0.96
N(1A)-C(4A)	1.520(5)	C(6A)-C(11A)	1.384(6)
N(1A)-H(1A1)	0.90	C(6A)-C(7A)	1.388(6)
N(1A)-H(1A2)	0.90	C(7A)-C(8A)	1.388(7)
C(2A)-O(2A)	1.4(5)	C(7A)-H(7A)	0.93
C(2A)-C(3A)	1.525(6)	C(8A)-C(9A)	1.365(7)
C(2A)-H(2A)	0.98	C(8A)-H(8A)	0.93
O(2A)-H(2AA)	0.82	C(9A)-C(10A)	1.361(7)
O(3A)-C(12A)	1.375(6)	C(10A)-C(11A)	1.392(6)
O(3A)-C(9A)	1.411(5)	C(10A)-H(10A)	0.93
C(3A)-C(4A)	1.525(6)	C(11A)-H(11A)	0.93

C(12A)-C(17A)	1.376(7)	C(3A)-O(1A)-H(1AA)	109.5
C(12A)-C(13A)	1.389(7)	C(4A)-N(1A)-C(1A)	109.4(3)
C(13A)-C(14A)	1.363(8)	C(4A)-N(1A)-H(1A1)	109.8
C(13A)-H(13A)	0.93	C(1A)-N(1A)-H(1A1)	109.8
C(14A)-C(15A)	1.364(9)	C(4A)-N(1A)-H(1A2)	109.8
C(14A)-H(14A)	0.93	C(1A)-N(1A)-H(1A2)	109.8
C(15A)-C(16A)	1.384(8)	H(1A1)-N(1A)-H(1A2)	108.2
C(15A)-H(15A)	0.93	O(2A)-C(2A)-C(3A)	114.7(3)
C(16A)-C(17A)	1.376(7)	O(2A)-C(2A)-C(1A)	108.8(3)
C(16A)-H(16A)	0.93	C(3A)-C(2A)-C(1A)	102.7(3)
C(17A)-H(17A)	0.93	O(2A)-C(2A)-H(2A)	110.1
O(1B)-C(3B)	1.427(5)	C(3A)-C(2A)-H(2A)	110.1
O(1B)-H(1BB)	0.82	C(1A)-C(2A)-H(2A)	110.1
C(1B)-N(1B)	1.506(5)	C(2A)-O(2A)-H(2AA)	109.5
C(1B)-C(6B)	1.512(5)	C(12A)-O(3A)-C(9A)	118.6(4)
C(1B)-C(2B)	1.531(5)	O(1A)-C(3A)-C(2A)	111.8(3)
C(1B)-H(1B)	0.98	O(1A)-C(3A)-C(4A)	109.9(3)
N(1B)-C(4B)	1.532(6)	C(2A)-C(3A)-C(4A)	102.4(3)
N(1B)-H(1B1)	0.90	O(1A)-C(3A)-H(3A)	110.8
N(1B)-H(1B2)	0.90	C(2A)-C(3A)-H(3A)	110.8
C(2B)-O(2B)	1.408(5)	C(4A)-C(3A)-H(3A)	110.8
C(2B)-C(3B)	1.530(6)	C(5A)-C(4A)-N(1A)	111.9(4)
C(2B)-H(2B)	0.98	C(5A)-C(4A)-C(3A)	117.1(4)
O(2B)-H(2BB)	0.82	N(1A)-C(4A)-C(3A)	102.9(3)
O(3B)-C(12B)	1.382(5)	C(5A)-C(4A)-H(4A)	108.2
O(3B)-C(9B)	1.401(5)	N(1A)-C(4A)-H(4A)	108.2
C(3B)-C(4B)	1.503(6)	C(3A)-C(4A)-H(4A)	108.2
C(3B)-H(3B)	0.98	C(4A)-C(5A)-H(5A1)	109.5
C(4B)-C(5B)	1.538(7)	C(4A)-C(5A)-H(5A2)	109.5
C(4B)-H(4B)	0.98	H(5A1)-C(5A)-H(5A2)	109.5
C(5B)-H(5B1)	0.96	C(4A)-C(5A)-H(5A3)	109.5
C(5B)-H(5B2)	0.96	H(5A1)-C(5A)-H(5A3)	109.5
C(5B)-H(5B3)	0.96	H(5A2)-C(5A)-H(5A3)	109.5
C(6B)-C(7B)	1.381(5)	C(11A)-C(6A)-C(7A)	117.6(4)
C(6B)-C(11B)	1.389(5)	C(11A)-C(6A)-C(1A)	119.6(4)
C(7B)-C(8B)	1.391(6)	C(7A)-C(6A)-C(1A)	122.8(3)
C(7B)-H(7B)	0.93	C(6A)-C(7A)-C(8A)	120.9(4)
C(8B)-C(9B)	1.369(6)	C(6A)-C(7A)-H(7A)	119.6
C(8B)-H(8B)	0.93	C(8A)-C(7A)-H(7A)	119.6
C(9B)-C(10B)	1.378(6)	C(9A)-C(8A)-C(7A)	119.6(4)
C(10B)-C(11B)	1.378(6)	C(9A)-C(8A)-H(8A)	120.2
C(10B)-H(10B)	0.93	C(7A)-C(8A)-H(8A)	120.2
C(11B)-H(11B)	0.93	C(10A)-C(9A)-C(8A)	121.4(4)
C(12B)-C(17B)	1.358(6)	C(10A)-C(9A)-O(3A)	119.4(4)
C(12B)-C(13B)	1.386(6)	C(8A)-C(9A)-O(3A)	119.0(4)
C(13B)-C(14B)	1.346(7)	C(9A)-C(10A)-C(11A)	118.6(4)
C(13B)-H(13B)	0.93	C(9A)-C(10A)-H(10A)	120.7
C(14B)-C(15B)	1.374(8)	C(11A)-C(10A)-H(10A)	120.7
C(14B)-H(14B)	0.93	C(6A)-C(11A)-C(10A)	121.9(4)
C(15B)-C(16B)	1.380(7)	C(6A)-C(11A)-H(11A)	119.1
C(15B)-H(15B)	0.93	C(10A)-C(11A)-H(11A)	119.1
C(16B)-C(17B)	1.376(6)	O(3A)-C(12A)-C(17A)	124.7(4)
C(16B)-H(16B)	0.93	O(3A)-C(12A)-C(13A)	115.6(4)
C(17B)-H(17B)	0.93	C(17A)-C(12A)-C(13A)	119.6(5)
C(6A)-C(1A)-N(1A)	112.0(3)	C(14A)-C(13A)-C(12A)	119.8(5)
C(6A)-C(1A)-C(2A)	118.7(3)	C(14A)-C(13A)-H(13A)	120.1
N(1A)-C(1A)-C(2A)	101.4(3)	C(12A)-C(13A)-H(13A)	120.1
C(6A)-C(1A)-H(1A)	108.1	C(13A)-C(14A)-C(15A)	121.2(6)
N(1A)-C(1A)-H(1A)	108.1	C(13A)-C(14A)-H(14A)	119.4
C(2A)-C(1A)-H(1A)	108.1	C(15A)-C(14A)-H(14A)	119.4

C(14A)-C(15A)-C(16A)	119.3(6)
C(14A)-C(15A)-H(15A)	120.4
C(16A)-C(15A)-H(15A)	120.4
C(17A)-C(16A)-C(15A)	120.3(6)
C(17A)-C(16A)-H(16A)	119.8
C(15A)-C(16A)-H(16A)	119.8
C(12A)-C(17A)-C(16A)	119.8(5)
C(12A)-C(17A)-H(17A)	120.1
C(16A)-C(17A)-H(17A)	120.1
C(3B)-O(1B)-H(1BB)	109.5
N(1B)-C(1B)-C(6B)	111.8(3)
N(1B)-C(1B)-C(2B)	103.0(3)
C(6B)-C(1B)-C(2B)	118.2(3)
N(1B)-C(1B)-H(1B)	107.8
C(6B)-C(1B)-H(1B)	107.8
C(2B)-C(1B)-H(1B)	107.8
C(1B)-N(1B)-C(4B)	107.9(3)
C(1B)-N(1B)-H(1B1)	110.1
C(4B)-N(1B)-H(1B1)	110.1
C(1B)-N(1B)-H(1B2)	110.1
C(4B)-N(1B)-H(1B2)	110.1
H(1B1)-N(1B)-H(1B2)	108.4
O(2B)-C(2B)-C(3B)	114.0(3)
O(2B)-C(2B)-C(1B)	110.1(3)
C(3B)-C(2B)-C(1B)	102.9(3)
O(2B)-C(2B)-H(2B)	109.9
C(3B)-C(2B)-H(2B)	109.9
C(1B)-C(2B)-H(2B)	109.9
C(2B)-O(2B)-H(2BB)	109.5
C(12B)-O(3B)-C(9B)	118.9(3)
O(1B)-C(3B)-C(4B)	108.7(4)
O(1B)-C(3B)-C(2B)	110.9(3)
C(4B)-C(3B)-C(2B)	101.4(3)
O(1B)-C(3B)-H(3B)	111.8
C(4B)-C(3B)-H(3B)	111.8
C(2B)-C(3B)-H(3B)	111.8
C(3B)-C(4B)-N(1B)	104.3(3)
C(3B)-C(4B)-C(5B)	116.1(4)
N(1B)-C(4B)-C(5B)	110.5(5)
C(3B)-C(4B)-H(4B)	108.6
N(1B)-C(4B)-H(4B)	108.6
C(5B)-C(4B)-H(4B)	108.6
C(4B)-C(5B)-H(5B1)	109.5
C(4B)-C(5B)-H(5B2)	109.5
H(5B1)-C(5B)-H(5B2)	109.5
C(4B)-C(5B)-H(5B3)	109.5
H(5B1)-C(5B)-H(5B3)	109.5
H(5B2)-C(5B)-H(5B3)	109.5
C(7B)-C(6B)-C(11B)	118.8(4)
C(7B)-C(6B)-C(1B)	121.7(3)
C(11B)-C(6B)-C(1B)	119.5(3)
C(6B)-C(7B)-C(8B)	120.5(4)
C(6B)-C(7B)-H(7B)	119.7
C(8B)-C(7B)-H(7B)	119.7
C(9B)-C(8B)-C(7B)	119.4(4)
C(9B)-C(8B)-H(8B)	120.3
C(7B)-C(8B)-H(8B)	120.3
C(8B)-C(9B)-C(10B)	121.1(4)
C(8B)-C(9B)-O(3B)	118.2(4)
C(10B)-C(9B)-O(3B)	120.6(4)
C(9B)-C(10B)-C(11B)	119.2(4)
C(9B)-C(10B)-H(10B)	120.4
C(11B)-C(10B)-H(10B)	120.4
C(10B)-C(11B)-C(6B)	121.0(4)
C(10B)-C(11B)-H(11B)	119.5
C(6B)-C(11B)-H(11B)	119.5
C(17B)-C(12B)-O(3B)	124.3(4)
C(17B)-C(12B)-C(13B)	119.6(4)
O(3B)-C(12B)-C(13B)	115.9(4)
C(14B)-C(13B)-C(12B)	121.0(5)
C(14B)-C(13B)-H(13B)	119.5
C(12B)-C(13B)-H(13B)	119.5
C(13B)-C(14B)-C(15B)	119.9(5)
C(13B)-C(14B)-H(14B)	120.1
C(15B)-C(14B)-H(14B)	120.1
C(14B)-C(15B)-C(16B)	119.7(5)
C(14B)-C(15B)-H(15B)	120.2
C(16B)-C(15B)-H(15B)	120.2
C(17B)-C(16B)-C(15B)	120.1(5)
C(17B)-C(16B)-H(16B)	119.9
C(15B)-C(16B)-H(16B)	119.9
C(12B)-C(17B)-C(16B)	119.8(4)
C(12B)-C(17B)-H(17B)	120.1
C(16B)-C(17B)-H(17B)	120.1

Torsion angles [°]

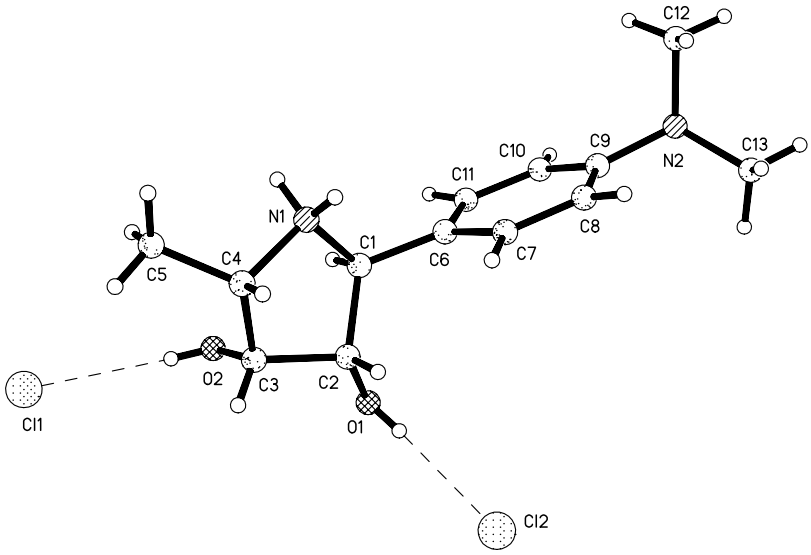
C(6A)-C(1A)-N(1A)-C(4A)	-143.9(3)
C(2A)-C(1A)-N(1A)-C(4A)	-16.3(4)
C(6A)-C(1A)-C(2A)-O(2A)	-76.8(4)
N(1A)-C(1A)-C(2A)-O(2A)	160.1(3)
C(6A)-C(1A)-C(2A)-C(3A)	161.2(3)
N(1A)-C(1A)-C(2A)-C(3A)	38.2(4)
O(2A)-C(2A)-C(3A)-O(1A)	-47.0(4)
C(1A)-C(2A)-C(3A)-O(1A)	70.9(4)
O(2A)-C(2A)-C(3A)-C(4A)	-164.6(3)
C(1A)-C(2A)-C(3A)-C(4A)	-46.7(4)
C(1A)-N(1A)-C(4A)-C(5A)	-138.3(4)
C(1A)-N(1A)-C(4A)-C(3A)	-11.7(4)
O(1A)-C(3A)-C(4A)-C(5A)	39.5(5)
C(2A)-C(3A)-C(4A)-C(5A)	158.5(4)
O(1A)-C(3A)-C(4A)-N(1A)	-83.6(4)
C(2A)-C(3A)-C(4A)-N(1A)	35.4(4)
N(1A)-C(1A)-C(6A)-C(11A)	-73.0(5)
C(2A)-C(1A)-C(6A)-C(11A)	169.4(4)
N(1A)-C(1A)-C(6A)-C(7A)	104.9(4)
C(2A)-C(1A)-C(6A)-C(7A)	-12.7(6)
C(11A)-C(6A)-C(7A)-C(8A)	1.4(7)
C(1A)-C(6A)-C(7A)-C(8A)	-176.6(4)
C(6A)-C(7A)-C(8A)-C(9A)	0.3(8)
C(7A)-C(8A)-C(9A)-C(10A)	-3.2(8)
C(7A)-C(8A)-C(9A)-O(3A)	-178.5(4)
C(12A)-O(3A)-C(9A)-C(10A)	82.7(6)
C(12A)-O(3A)-C(9A)-C(8A)	-101.9(6)
C(8A)-C(9A)-C(10A)-C(11A)	4.1(8)
O(3A)-C(9A)-C(10A)-C(11A)	179.4(5)
C(7A)-C(6A)-C(11A)-C(10A)	-0.3(7)
C(1A)-C(6A)-C(11A)-C(10A)	177.7(5)
C(9A)-C(10A)-C(11A)-C(6A)	-2.4(8)

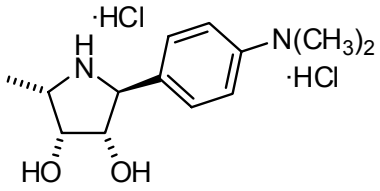
C(9A)-O(3A)-C(12A)-C(17A)	-9.7(7)
C(9A)-O(3A)-C(12A)-C(13A)	171.5(4)
O(3A)-C(12A)-C(13A)-C(14A)	178.9(5)
C(17A)-C(12A)-C(13A)-C(14A)	0.0(7)
C(12A)-C(13A)-C(14A)-C(15A)	-0.3(9)
C(13A)-C(14A)-C(15A)-C(16A)	1.1(10)
C(14A)-C(15A)-C(16A)-C(17A)	-1.6(9)
O(3A)-C(12A)-C(17A)-C(16A)	-179.2(5)
C(13A)-C(12A)-C(17A)-C(16A)	-0.4(7)
C(15A)-C(16A)-C(17A)-C(12A)	1.2(8)
C(6B)-C(1B)-N(1B)-C(4B)	-141.6(3)
C(2B)-C(1B)-N(1B)-C(4B)	-13.7(4)
N(1B)-C(1B)-C(2B)-O(2B)	158.3(3)
C(6B)-C(1B)-C(2B)-O(2B)	-77.9(4)
N(1B)-C(1B)-C(2B)-C(3B)	36.4(3)
C(6B)-C(1B)-C(2B)-C(3B)	160.3(3)
O(2B)-C(2B)-C(3B)-O(1B)	-49.6(5)
C(1B)-C(2B)-C(3B)-O(1B)	69.6(4)
O(2B)-C(2B)-C(3B)-C(4B)	-164.9(4)
C(1B)-C(2B)-C(3B)-C(4B)	-45.8(4)
O(1B)-C(3B)-C(4B)-N(1B)	-80.3(4)
C(2B)-C(3B)-C(4B)-N(1B)	36.7(5)
O(1B)-C(3B)-C(4B)-C(5B)	41.6(6)
C(2B)-C(3B)-C(4B)-C(5B)	158.5(5)
C(1B)-N(1B)-C(4B)-C(3B)	-14.6(4)
C(1B)-N(1B)-C(4B)-C(5B)	-140.0(4)
N(1B)-C(1B)-C(6B)-C(7B)	124.4(4)
C(2B)-C(1B)-C(6B)-C(7B)	5.1(5)
N(1B)-C(1B)-C(6B)-C(11B)	-55.9(4)
C(2B)-C(1B)-C(6B)-C(11B)	-175.3(3)
C(11B)-C(6B)-C(7B)-C(8B)	0.6(7)
C(1B)-C(6B)-C(7B)-C(8B)	-179.8(4)
C(6B)-C(7B)-C(8B)-C(9B)	-0.6(8)
C(7B)-C(8B)-C(9B)-C(10B)	0.4(7)
C(7B)-C(8B)-C(9B)-O(3B)	-175.5(4)
C(12B)-O(3B)-C(9B)-C(8B)	-112.1(5)
C(12B)-O(3B)-C(9B)-C(10B)	72.0(6)
C(8B)-C(9B)-C(10B)-C(11B)	-0.3(7)
O(3B)-C(9B)-C(10B)-C(11B)	175.6(4)
C(9B)-C(10B)-C(11B)-C(6B)	0.3(6)
C(7B)-C(6B)-C(11B)-C(10B)	-0.5(6)
C(1B)-C(6B)-C(11B)-C(10B)	179.9(4)
C(9B)-O(3B)-C(12B)-C(17B)	14.5(7)
C(9B)-O(3B)-C(12B)-C(13B)	-170.7(4)
C(17B)-C(12B)-C(13B)-C(14B)	-0.4(8)
O(3B)-C(12B)-C(13B)-C(14B)	-175.6(5)
C(12B)-C(13B)-C(14B)-C(15B)	0.7(9)
C(13B)-C(14B)-C(15B)-C(16B)	-0.3(9)
C(14B)-C(15B)-C(16B)-C(17B)	-0.5(9)
O(3B)-C(12B)-C(17B)-C(16B)	174.3(5)
C(13B)-C(12B)-C(17B)-C(16B)	-0.4(7)
C(15B)-C(16B)-C(17B)-C(12B)	0.8(8)

H-Bond lengths [Å] and angles [°]

N1A-Br1B_$1	3.2652(0.0031)
H1A1-Br1B_$1	2.3896
N1A-Br1A_$2	3.3023(0.0032)
H1A2-Br1A_$2	2.4363
O1A-Br1B	3.3640(0.0028)
H1AA-Br1B	2.5787
O2A-Br1A	3.3486(0.0031)
H2AA-Br1A	2.5948
N1B-Br1B	3.2798(0.0033)
H1B1-Br1B	2.3959
N1B-Br1A	3.3010(0.0030)
H1B2-Br1A	2.4071
O1B-Br1A_$3	3.3725(0.0029)
H1BB-Br1A_$3	2.5762
O2B-Br1A_$3	3.7229(0.0033)
H2BB-Br1A_$3	3.3621
N1A-H1A1-Br1B_$1	164.32
N1A-H1A2-Br1A_$2	161.55
O1A-H1AA-Br1B	160.84
O2A-H2AA-Br1A	153.47
N1B-H1B1-Br1B	167.25
N1B-H1B2-Br1A	172.17
O1B-H1BB-Br1A_$3	164.18
O2B-H2BB-Br1A_$3	109.98

9.19 (2*S*,3*S*,4*R*,5*S*)-3,4-Dihydroxy-2-(4-*N*,*N*-dimethylanilino)-5-methylpyrrolidine dihydrochloride (104·2HCl)





104·2HCl

$C_{13}H_{22}Cl_2N_2O_2$

orthorhombic, $P2_12_12_1$

a = 7.2733(9) Å

b = 8.3574(14) Å

c = 26.118(3) Å

α = 90 °

β = 90 °

γ = 90 °

V = 1587.6(4) Å^3

Z = 4, $R(F)$ = 0.0422

$R_w(F^2)$ = 0.0992

Crystal size: 1.00 x 0.80 x 0.40 mm

Calculated density: 1.294 g/cm^3

2θ-Range for data collection: 2.56– 29.99 °

Independent reflections: 5229

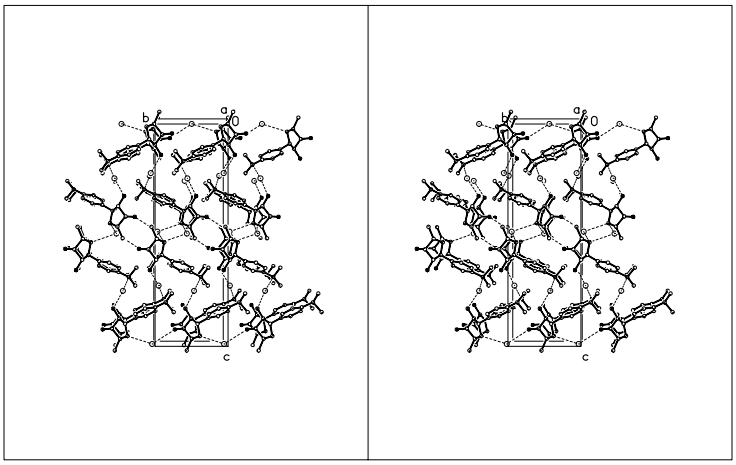
Observed reflections: 4629

Contributed reflections to refinement: 4429

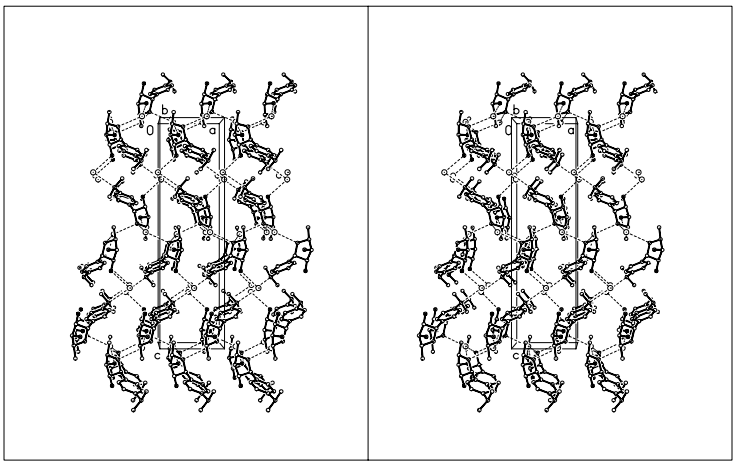
Refined parameters: 261

Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

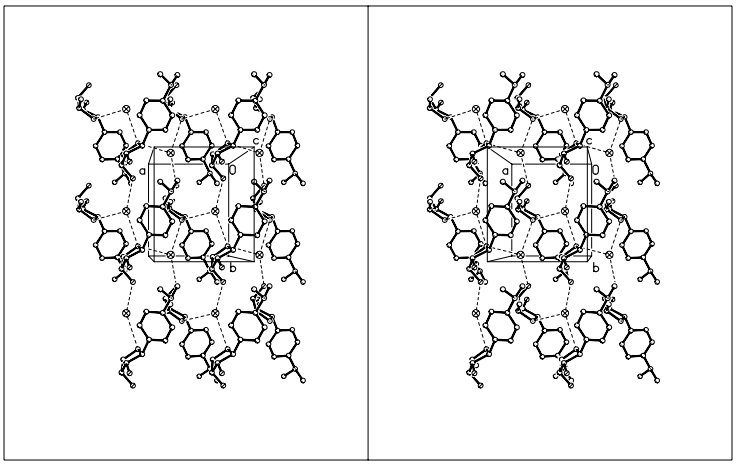
(a)



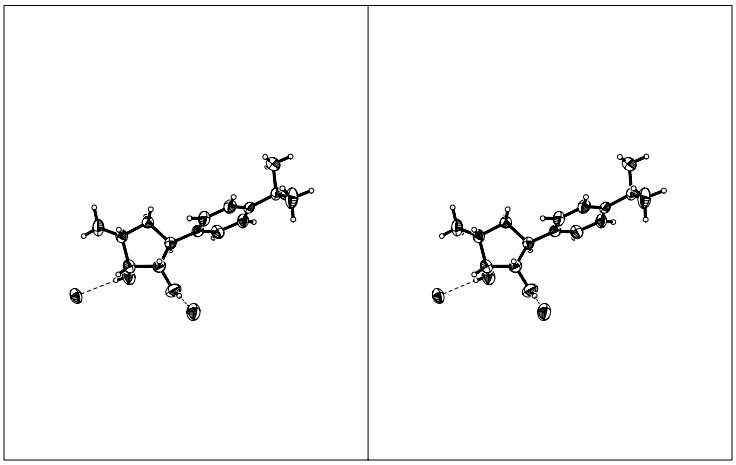
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

C(1)-C(2)	1.398(3)	C(12)-N(2)-H(2A)	104.8(18)
O(1)-H(1C)	0.76(3)	C(3)-O(2)-H(2B)	111(3)
N(1)-C(1)	1.508(2)	O(1)-C(2)-C(1)	110.34(18)
N(1)-C(4)	1.523(2)	O(1)-C(2)-C(3)	114.41(18)
N(1)-H(1A)	0.83(3)	C(1)-C(2)-C(3)	101.12(16)
N(1)-H(1B)	0.91(3)	O(1)-C(2)-H(2)	113.5(16)
C(1)-C(6)	1.506(3)	C(1)-C(2)-H(2)	110.4(16)
C(1)-C(2)	1.533(3)	C(3)-C(2)-H(2)	106.4(16)
C(1)-H(1)	0.96(2)	O(2)-C(3)-C(4)	112.77(19)
N(2)-C(9)	1.470(2)	O(2)-C(3)-C(2)	107.06(18)
N(2)-C(13)	1.488(3)	C(4)-C(3)-C(2)	102.18(16)
N(2)-C(12)	1.491(3)	O(2)-C(3)-H(3)	113.7(19)
N(2)-H(2A)	0.90(3)	C(4)-C(3)-H(3)	106.4(18)
O(2)-C(3)	1.404(3)	C(2)-C(3)-H(3)	114.2(18)
O(2)-H(2B)	0.75(3)	C(5)-C(4)-N(1)	111.55(18)
C(2)-C(3)	1.534(3)	C(5)-C(4)-C(3)	116.8(2)
C(2)-H(2)	0.98(3)	N(1)-C(4)-C(3)	103.22(15)
C(3)-C(4)	1.528(3)	C(5)-C(4)-H(4)	109.1(14)
C(3)-H(3)	0.89(3)	N(1)-C(4)-H(4)	102.9(15)
C(4)-C(5)	1.506(3)	C(3)-C(4)-H(4)	112.3(14)
C(4)-H(4)	0.93(3)	C(4)-C(5)-H(5A)	110(2)
C(5)-H(5A)	0.95(4)	C(4)-C(5)-H(5B)	107(2)
C(5)-H(5B)	0.91(4)	H(5A)-C(5)-H(5B)	113(3)
C(5)-H(5C)	0.99(4)	C(4)-C(5)-H(5C)	109(2)
C(6)-C(11)	1.389(3)	H(5A)-C(5)-H(5C)	116(3)
C(6)-C(7)	1.391(3)	H(5B)-C(5)-H(5C)	102(3)
C(7)-C(8)	1.390(3)	C(11)-C(6)-C(7)	118.99(18)
C(7)-H(7)	1.02(3)	C(11)-C(6)-C(1)	119.01(17)
C(8)-C(9)	1.389(3)	C(7)-C(6)-C(1)	122.00(17)
C(8)-H(8)	0.92(3)	C(8)-C(7)-C(6)	120.92(19)
C(9)-C(10)	1.380(3)	C(8)-C(7)-H(7)	118.7(15)
C(10)-C(11)	1.384(3)	C(6)-C(7)-H(7)	120.4(15)
C(10)-H(10)	0.90(3)	C(9)-C(8)-C(7)	118.52(18)
C(11)-H(11)	0.93(3)	C(9)-C(8)-H(8)	119.7(19)
C(12)-H(12A)	0.93(4)	C(7)-C(8)-H(8)	121.7(19)
C(12)-H(12B)	0.96(5)	C(10)-C(9)-C(8)	121.54(18)
C(12)-H(12C)	0.92(4)	C(10)-C(9)-N(2)	118.12(17)
C(13)-H(13A)	0.92(4)	C(8)-C(9)-N(2)	120.30(18)
C(13)-H(13B)	1.04(4)	C(9)-C(10)-C(11)	119.01(19)
C(13)-H(13C)	0.97(4)	C(9)-C(10)-H(10)	119.9(18)
C(2)-O(1)-H(1C)	111(3)	C(11)-C(10)-H(10)	121.1(19)
C(1)-N(1)-C(4)	108.58(15)	C(10)-C(11)-C(6)	120.96(19)
C(1)-N(1)-H(1A)	112.0(17)	C(10)-C(11)-H(11)	117.8(19)
C(4)-N(1)-H(1A)	110.7(17)	C(6)-C(11)-H(11)	120.7(19)
C(1)-N(1)-H(1B)	104.5(18)	N(2)-C(12)-H(12A)	101(3)
C(4)-N(1)-H(1B)	111.9(18)	N(2)-C(12)-H(12B)	98(3)
H(1A)-N(1)-H(1B)	109(3)	H(12A)-C(12)-H(12B)	133(4)
C(6)-C(1)-N(1)	112.66(15)	N(2)-C(12)-H(12C)	106(2)
C(6)-C(1)-C(2)	118.46(16)	H(12A)-C(12)-H(12C)	105(4)
N(1)-C(1)-C(2)	102.88(15)	H(12B)-C(12)-H(12C)	110(4)
C(6)-C(1)-H(1)	109.1(13)	N(2)-C(13)-H(13A)	109(2)
N(1)-C(1)-H(1)	108.1(13)	N(2)-C(13)-H(13B)	105(2)
C(2)-C(1)-H(1)	105.0(13)	H(13A)-C(13)-H(13B)	118(3)
C(9)-N(2)-C(13)	114.3(2)	N(2)-C(13)-H(13C)	112(2)
C(9)-N(2)-C(12)	110.05(17)	H(13A)-C(13)-H(13C)	108(3)
C(13)-N(2)-C(12)	112.0(3)	H(13B)-C(13)-H(13C)	105(3)
C(9)-N(2)-H(2A)	109.3(18)		
C(13)-N(2)-H(2A)	105.9(18)		

Torsion angles [°]

C(4)-N(1)-C(1)-C(6)	-147.13(16)
C(4)-N(1)-C(1)-C(2)	-18.43(19)
C(6)-C(1)-C(2)-O(1)	-73.7(2)
N(1)-C(1)-C(2)-O(1)	161.34(17)
C(6)-C(1)-C(2)-C(3)	164.86(17)
N(1)-C(1)-C(2)-C(3)	39.87(18)
O(1)-C(2)-C(3)-O(2)	-46.8(3)
C(1)-C(2)-C(3)-O(2)	71.8(2)
O(1)-C(2)-C(3)-C(4)	-165.52(18)
C(1)-C(2)-C(3)-C(4)	-46.95(19)
C(1)-N(1)-C(4)-C(5)	-136.7(2)
C(1)-N(1)-C(4)-C(3)	-10.5(2)
O(2)-C(3)-C(4)-C(5)	43.4(3)
C(2)-C(3)-C(4)-C(5)	158.0(2)
O(2)-C(3)-C(4)-N(1)	-79.3(2)
C(2)-C(3)-C(4)-N(1)	35.3(2)
N(1)-C(1)-C(6)-C(11)	-118.3(2)
C(2)-C(1)-C(6)-C(11)	121.6(2)
N(1)-C(1)-C(6)-C(7)	61.2(3)
C(2)-C(1)-C(6)-C(7)	-58.9(3)
C(11)-C(6)-C(7)-C(8)	1.9(3)
C(1)-C(6)-C(7)-C(8)	-177.7(2)
C(6)-C(7)-C(8)-C(9)	-1.0(3)
C(7)-C(8)-C(9)-C(10)	-1.4(3)
C(7)-C(8)-C(9)-N(2)	176.39(19)
C(13)-N(2)-C(9)-C(10)	-136.2(2)
C(12)-N(2)-C(9)-C(10)	96.8(3)
C(13)-N(2)-C(9)-C(8)	46.0(3)
C(12)-N(2)-C(9)-C(8)	-81.1(3)
C(8)-C(9)-C(10)-C(11)	2.8(3)
N(2)-C(9)-C(10)-C(11)	-175.02(19)
C(9)-C(10)-C(11)-C(6)	-1.9(3)
C(7)-C(6)-C(11)-C(10)	-0.4(3)
C(1)-C(6)-C(11)-C(10)	179.1(2)

H-Bond lengths [Å] and angles [°]

O2-Cl1	3.1761(0.0019)
H2B-Cl1	2.4413(0.0349)
O1-Cl2	3.0865(0.0023)
H1C-Cl2	2.3363(0.0358)
N1-Cl1_$1	3.1732(0.0019)
H1A-Cl1_$1	2.3610(0.0276)
N1-Cl1_$2	3.0414(0.0018)
H1B-Cl1_$2	2.1449(0.0315)
N2-Cl2_$3	3.0171(0.0020)
H2A-Cl2_$3	2.1232(0.0313)
O2-H2B-Cl1	166.41(3.54)
O1-H1C-Cl2	169.47(3.63)
N1-H1A-Cl1_$1	164.62(2.30)
N1-H1B-Cl1_$2	170.07(2.58)

9.20 (2*S*,3*S*,4*R*,5*S*)-2-*tert*-Butyl-3,4-dihydroxy-5-methyl-pyrrolidine hydrobromide (110·HBr)

$C_9H_{20}BrNO_2$

tetragonal, $P4_12_12$

a = 8.0533(6) Å

b = 8.0533(6) Å

c = 37.121(4) Å

α = 90 °

β = 90 °

γ = 90 °

V = 2407.5(4) Å^3

Z = 8, $R(F)$ = 0.0534

$R_w(F^2)$ = 0.1361

Crystal size: 0.5 x 0.5 x 0.5 mm

Calculated density: 1.402 g/cm^3

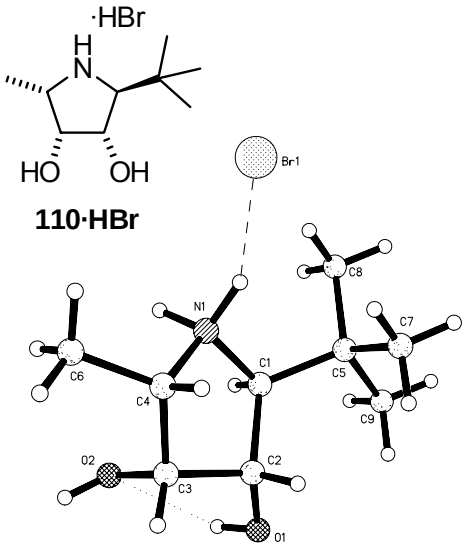
2θ-Range for data collection: 4.76 – 67.99 °

Independent reflections: 2085

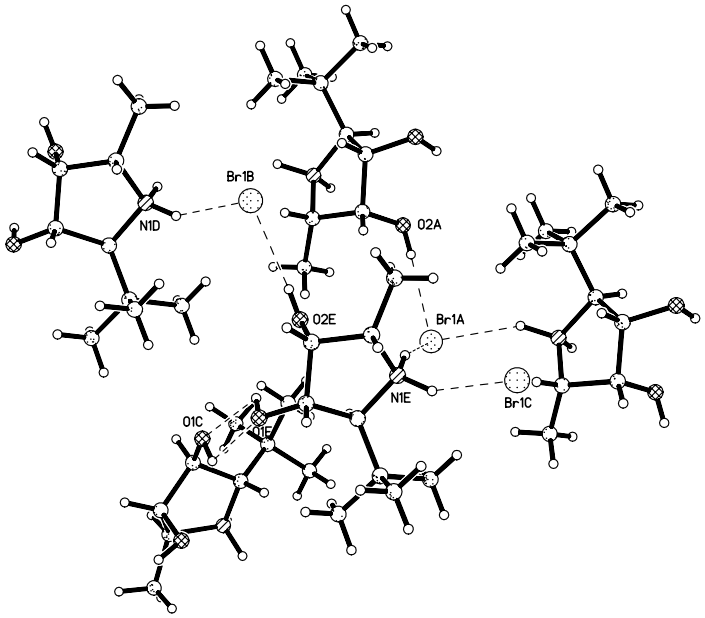
Observed reflections: 4186

Contributed reflections to refinement: 2085

Refined parameters: 119

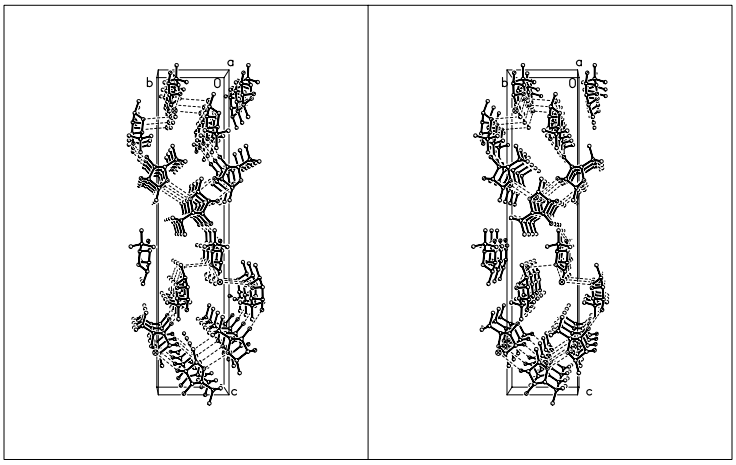


110·HBr

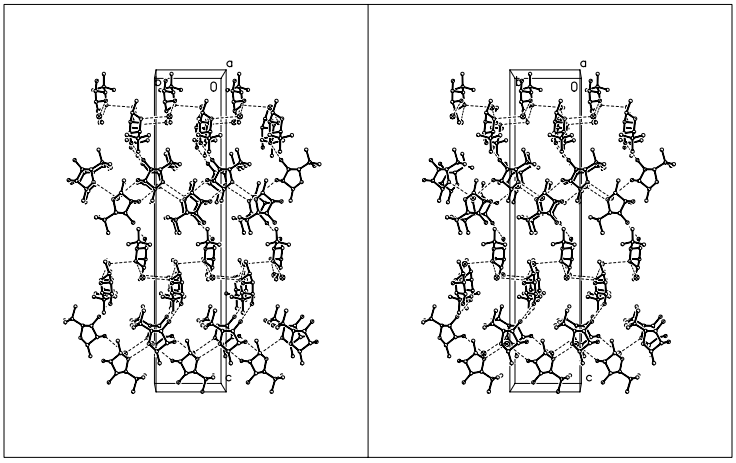


Unit cell; view along the a-axis (a), b-axis (b) and c-axis (c):

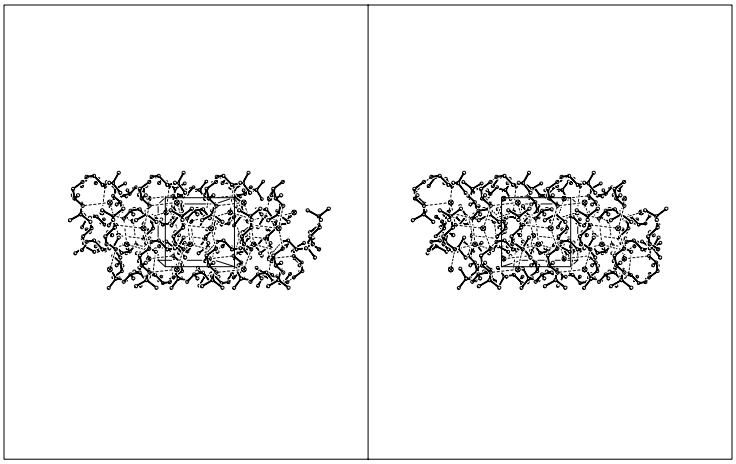
(a)



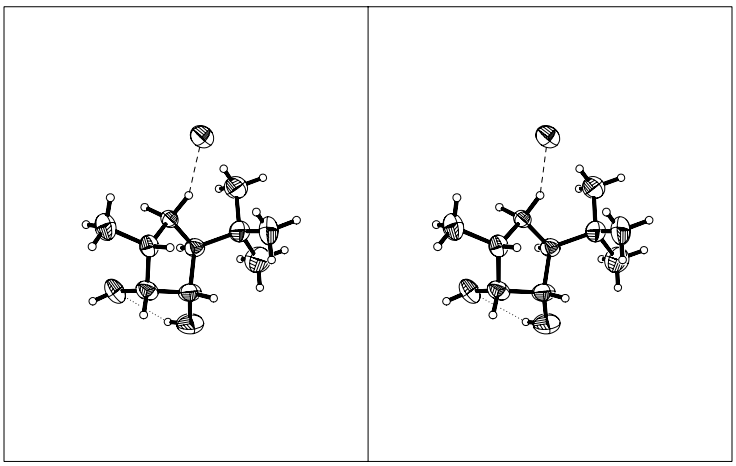
(b)



(c)



ORTEP Structure



Bond lengths [Å] and angles [°]

N(1)-C(1)	1.513(7)	C(5)-C(8)	1.535(10)
N(1)-C(4)	1.514(7)	C(5)-C(9)	1.541(9)
N(1)-H(1A)	1.0523	C(6)-H(6A)	0.9600
N(1)-H(1B)	1.0420	C(6)-H(6B)	0.9600
C(1)-C(5)	1.540(9)	C(6)-H(6C)	0.9600
C(1)-C(2)	1.547(8)	C(7)-H(7A)	0.9600
C(1)-H(1)	0.9800	C(7)-H(7B)	0.9600
O(1)-C(2)	1.410(8)	C(7)-H(7C)	0.9600
O(1)-H(1C)	0.8593	C(8)-H(8A)	0.9600
O(2)-C(3)	1.428(8)	C(8)-H(8B)	0.9600
O(2)-H(2A)	0.8196	C(8)-H(8C)	0.9600
C(2)-C(3)	1.523(9)	C(9)-H(9A)	0.9600
C(2)-H(2)	0.9800	C(9)-H(9B)	0.9600
C(3)-C(4)	1.523(9)	C(9)-H(9C)	0.9600
C(3)-H(3)	0.9800	C(1)-N(1)-C(4)	109.3(4)
C(4)-C(6)	1.508(8)	C(1)-N(1)-H(1A)	105.5
C(4)-H(4)	0.9800	C(4)-N(1)-H(1A)	107.7
C(5)-C(7)	1.519(11)	C(1)-N(1)-H(1B)	112.6

C(4)-N(1)-H(1B)	106.6
H(1A)-N(1)-H(1B)	115.1
N(1)-C(1)-C(5)	112.4(5)
N(1)-C(1)-C(2)	103.7(5)
C(5)-C(1)-C(2)	117.5(5)
N(1)-C(1)-H(1)	107.6
C(5)-C(1)-H(1)	107.6
C(2)-C(1)-H(1)	107.6
C(2)-O(1)-H(1C)	110.1
C(3)-O(2)-H(2A)	109.5
O(1)-C(2)-C(3)	112.8(5)
O(1)-C(2)-C(1)	112.9(5)
C(3)-C(2)-C(1)	103.7(5)
O(1)-C(2)-H(2)	109.1
C(3)-C(2)-H(2)	109.1
C(1)-C(2)-H(2)	109.1
O(2)-C(3)-C(2)	106.4(5)
O(2)-C(3)-C(4)	111.5(5)
C(2)-C(3)-C(4)	102.8(5)
O(2)-C(3)-H(3)	111.9
C(2)-C(3)-H(3)	111.9
C(4)-C(3)-H(3)	111.9
C(6)-C(4)-N(1)	113.2(5)
C(6)-C(4)-C(3)	116.7(5)
N(1)-C(4)-C(3)	102.0(5)
C(6)-C(4)-H(4)	108.2
N(1)-C(4)-H(4)	108.2
C(3)-C(4)-H(4)	108.2
C(7)-C(5)-C(8)	109.5(7)
C(7)-C(5)-C(1)	112.9(6)
C(8)-C(5)-C(1)	108.1(5)
C(7)-C(5)-C(9)	110.3(8)
C(8)-C(5)-C(9)	109.0(7)
C(1)-C(5)-C(9)	107.0(6)
C(4)-C(6)-H(6A)	109.5
C(4)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
C(4)-C(6)-H(6C)	109.5
H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5
C(5)-C(7)-H(7A)	109.5
C(5)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
C(5)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(5)-C(8)-H(8A)	109.5
C(5)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(5)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(5)-C(9)-H(9A)	109.5
C(5)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(5)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5

Torsion angles [°]

C(4)-N(1)-C(1)-C(5)	-128.7(5)
C(4)-N(1)-C(1)-C(2)	-0.8(6)
N(1)-C(1)-C(2)-O(1)	148.6(5)
C(5)-C(1)-C(2)-O(1)	-86.7(7)
N(1)-C(1)-C(2)-C(3)	26.2(6)
C(5)-C(1)-C(2)-C(3)	150.8(5)
O(1)-C(2)-C(3)-O(2)	-47.1(7)
C(1)-C(2)-C(3)-O(2)	75.4(6)
O(1)-C(2)-C(3)-C(4)	-164.4(5)
C(1)-C(2)-C(3)-C(4)	-41.9(6)
C(1)-N(1)-C(4)-C(6)	-150.8(5)
C(1)-N(1)-C(4)-C(3)	-24.6(6)
O(2)-C(3)-C(4)-C(6)	50.8(7)
C(2)-C(3)-C(4)-C(6)	164.4(5)
O(2)-C(3)-C(4)-N(1)	-73.1(6)
C(2)-C(3)-C(4)-N(1)	40.5(5)
N(1)-C(1)-C(5)-C(7)	64.0(7)
C(2)-C(1)-C(5)-C(7)	-56.2(8)
N(1)-C(1)-C(5)-C(8)	-57.3(7)
C(2)-C(1)-C(5)-C(8)	-177.4(6)
N(1)-C(1)-C(5)-C(9)	-174.5(6)
C(2)-C(1)-C(5)-C(9)	65.3(8)

10 Acknowledgments

Special thanks go to:

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11 Curriculum Vitae

Personal Information

Name:	Redcliffe, James Leo
Born, Birthplace:	25.2.1979 in Whiston, Merseyside, England
Parents:	Eric (✞ 20.7.2010) and Ruth Redcliffe, neé English
Nationality:	British
Marital status:	single

Primary and Secondary Education

1984-1990	Nutgrove Primary School, St. Helens
1990-1995	Rainhill High School Comprehensive, Prescott, Merseyside
Sept. 1995-May 1997	Rainhill High School, Sixth From College
May 1997	'A' Levels (ger. Abitur)

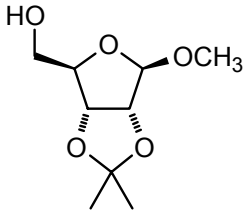
Tertiary Education

Oct. 1997	Began chemistry studies at the University of Reading, UK
Oct. 2000-March 2001	Masters research project under the supervision of Dr. H.M.I. Osborn. Title: *Carbohydrate derived β-amino acids*
Juli 2001	Masters (MChem) degree awarded
since Oct. 2001	Doctoral student with Prof. Dr. V. Jäger at the Institut für Organische Chemie der Universität Stuttgart

Employment

Nov. 2001–Sept. 2002	"Wissenschaftliche Hilfskraft" at the Institut für Organische Chemie der Universität Stuttgart
Sept. 2002–March. 2004	"Wissenschaftlicher Angestellter" (BAT IIa/2), dto.
April 2004–Dec 2004	"Wissenschaftlicher Angestellter" (BAT IIa), dto.
Sept. 2004–Dec. 2005	"Wissenschaftlicher Angestellter" (BAT IIa/2), dto.
May 2007–June 2007	Internship, Bayer Healthcare, Wuppertal, Germany
August 2007–Nov. 2007	"Wissenschaftlicher Angestellter" (TVL 13/2), at the Institut für Organische Chemie der Universität Stuttgart
May 2009–July 2009	"Wissenschaftlicher Angestellter" (TVL 13/2), dto.

12 Formula Register



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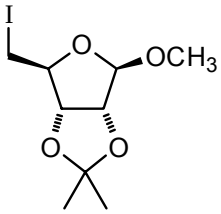

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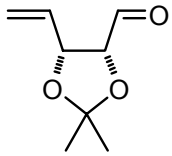

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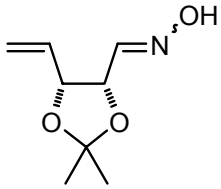

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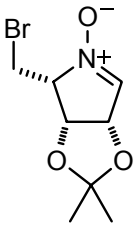
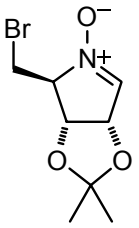


5 L-*lyxo* (E 5, lit.[2])



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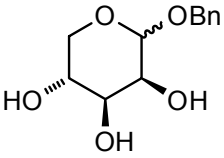
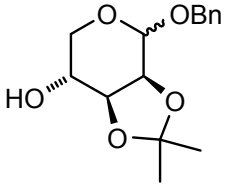
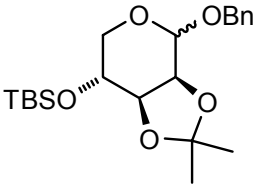


7 (E 6, lit.[208])



8 (E 7, lit.[208])



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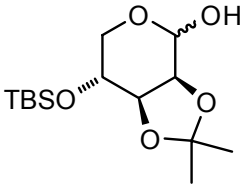

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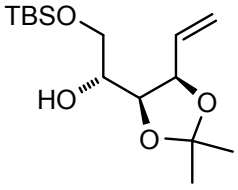
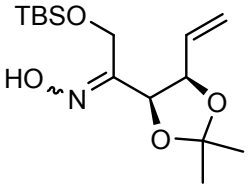
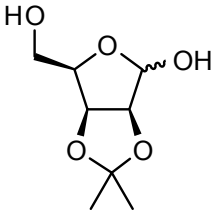


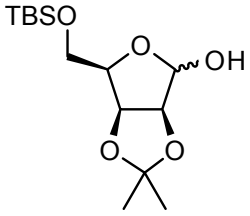
11 (E 10, 14)



12-(*E*/*Z*) (E 17)



13 (E 12, lit.[223])



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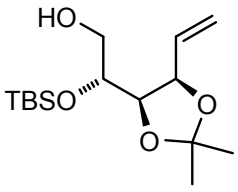

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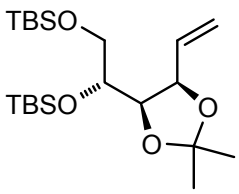

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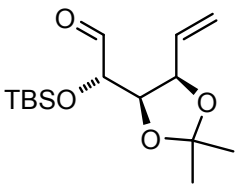
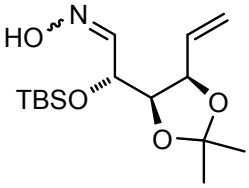


17 (E 17)



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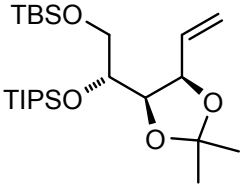
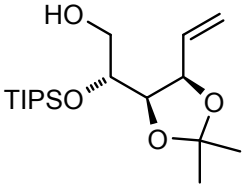
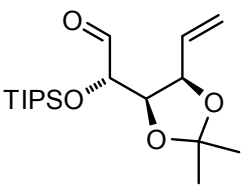


19 (E 19)



20 (E 20)



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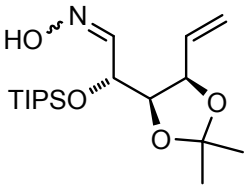

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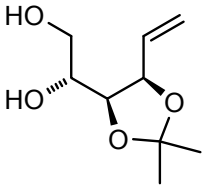

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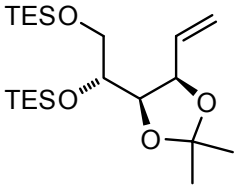

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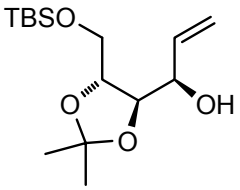
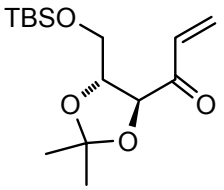


25 (E 25)



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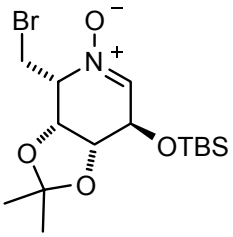
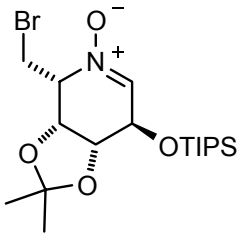
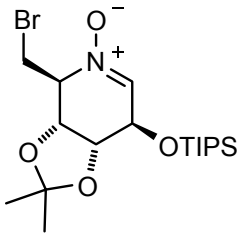


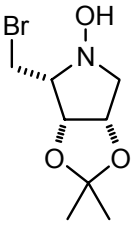
27 L-*fuco* (E 26)



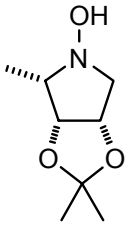
28 L-*fuco* (E 27)



29 D-*altro* (E 27)



30 (E 28, lit.[2])



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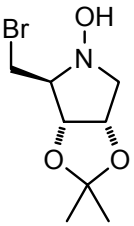
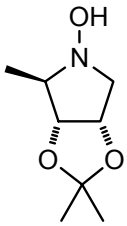
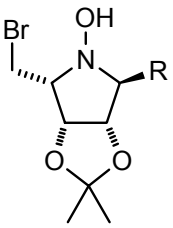


31 (E 30)

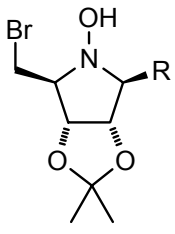


33 (E 31, lit.[2])



34-50

34	Methyl	(E 32)
35	*n*-Propyl	(E 33)
36	*i*-Propyl	(E 34)
37	*t*-Butyl	(E 35)
38	Allyl	(E 36)
39	4-Penten-1-yl	(E 37)
40	Phenyl	(E 38)
41	*p*-Methoxyphenyl	(E 39)
42	[1,1'-Biphenyl]-4-yl	(E 50)
43	*p*-Fluorophenyl	(E 41)
44	*p*-Chlorophenyl	(E 42)
45	*p*-Bromophenyl	(E 43)
46	*p*-Methoxybenzyl	(E 44)
47	*p*-Phenoxyphenyl	(E 45)
48	*p*-Methylthiophenyl	(E 46)
49	2-Furyl	(E 47)
50	*p*-*N*,*N*-Dimethylanilino	(E 48)
51	*p*-*N*,*N*-Dimethylanilino	(E 49)



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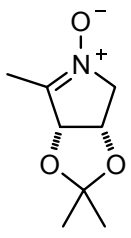
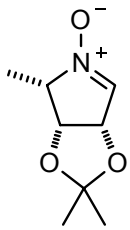
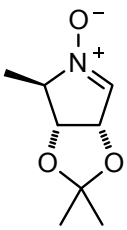


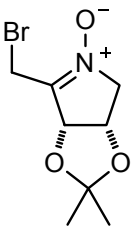
54 (E 51-54, lit.[4a])



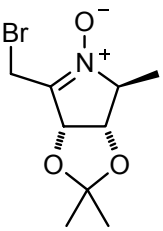
55 (E 51, 52)



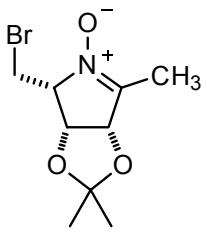
56 (E 53, 54, lit.[4a])



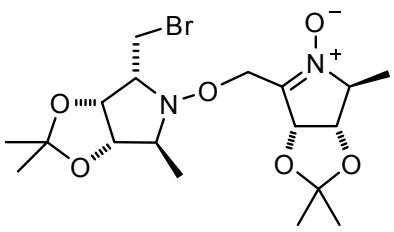
57 (E 55-58)



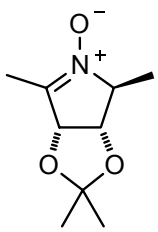
58 (E 59, 60)



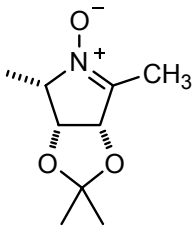
59 (E 59, 60)



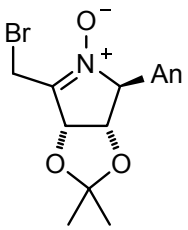
60 (E 59)



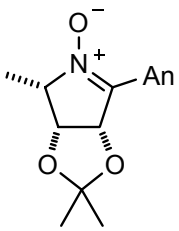
61 (E 61, 62)



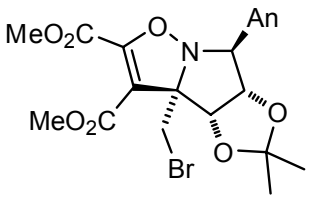
62 (E 61, 62)



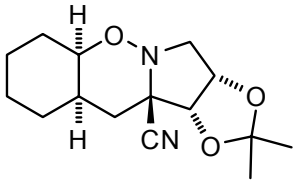
63 (E 63, 64)



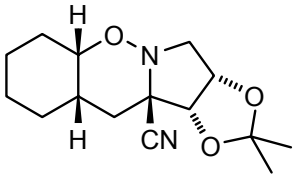
64 (E 65, 66)



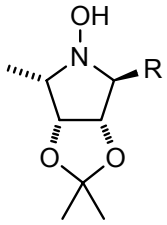
65 (E 67)



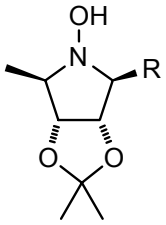
66 (E 68)



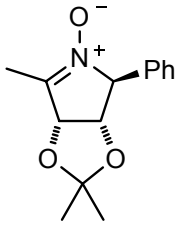
67 (E 68)



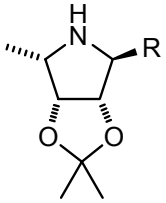
68-80



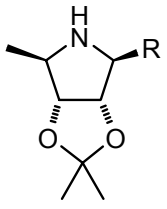
81



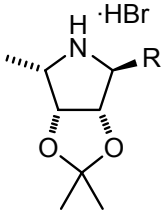
82 (E 83)



86-94

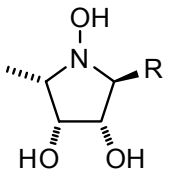


95

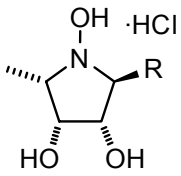


109, 111

68	Methyl	(E 69)
69	*i*-Propyl	(E 70)
70	Allyl	(E 71)
71	*p*-Methoxyphenyl	(E 72)
72	[1,1'-Biphenyl]-4-yl	(E 73)
73	*p*-Fluorophenyl	(E 74)
74	*p*-Chlorophenyl	(E 75)
75	*p*-Bromophenyl	(E 76)
76	*p*-Methylthiophenyl	(E 77)
77	*p*-Methoxybenzyl	(E 78)
78	*p*-Phenoxyphenyl	(E 79)
79	2-Furyl	(E 80)
80	*p*-*N*,*N*-Dimethylanilino	(E 81)
81	*p*-*N*,*N*-Dimethylanilino	(E 82)
86	*p*-Methoxyphenyl	(E 84, 88)
87	[1,1'-Biphenyl]-4-yl	(E 89)
88	*p*-Fluorophenyl	(E 90)
89	*p*-Chlorophenyl	(E 91)
90	*p*-Bromophenyl	(E 92)
91	*p*-Thiomethylphenyl	(E 93)
92	*p*-Phenoxyphenyl	(E 94)
93	2-Furyl	(E 95)
94	*p*-*N*,*N*-Dimethylanilino	(E 96)
95	*p*-*N*,*N*-Dimethylanilino	(E 97)
109	*t*-Butyl	(E 112)
111	*p*-Methoxybenzyl	(E 114)

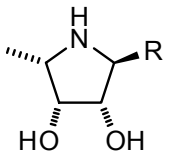


83

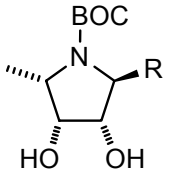


84, 85

83	*p*-Methoxyphenyl	(E 85)
84	[1,1'-Biphenyl]-4-yl	(E 86)
85	*p*-Methylthiophenyl	(E 87)

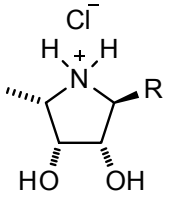


105, 106, 107

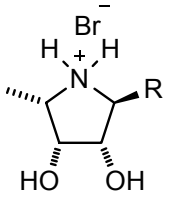


108

105	*p*-Methoxyphenyl	(E 108)
106	*p*-Chlorophenyl	(E 109)
107	*p*-Phenoxyphenyl	(E 110)
108	*p*-Methoxyphenyl	(E 111)



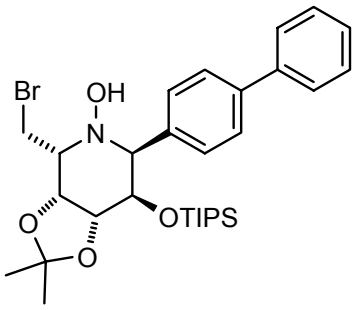
96, 97, 98, 99 100, 101, 102, 103, 104 (·2HCl)



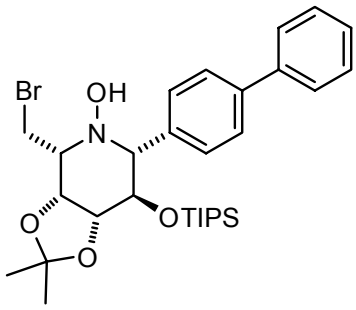
102, 110, 112

96	*p*-Methoxyphenyl	(E 98)
97	[1,1'-Biphenyl]-4-yl	(E 99)
98	*p*-Fluorophenyl	(E 100)
99	*p*-Chlorophenyl	(E 101)
100	*p*-Bromophenyl	(E 102)
101	*p*-Methylthiophenyl	(E 103)
102	*p*-Phenoxyphenyl	(E 104)
103	2-Furyl	(E 106)
104	*p*-*N*,*N*-Dimethylanilino	(E 107)

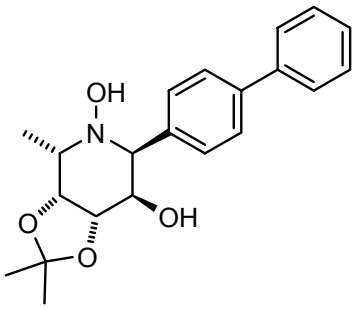
102	*p*-Phenoxyphenyl	(E 105)
110	*t*-Butyl	(E 113)
112	*p*-Methoxybenzyl	(E 115)



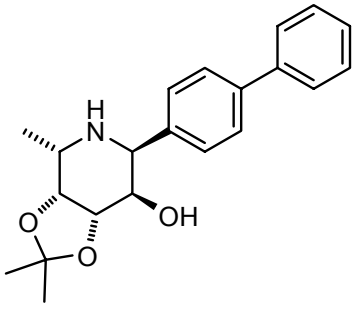
52 (E 50)



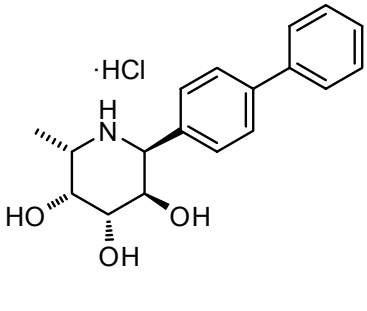
53 (E 50)



113 (E 116)



114 (E 117)



115·HCl (E 118)

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