APPENDIX B

X-ray Crystallographic Data for:

5,5-Bis(trifluoromethyl)-4-oxa-tricyclo[4.3.$0^{1,6}$.$0^{3,7}$]nonane-8,9-diol (**4.6**)

Table 1. Crystal data and structure refinement for DPS01 (CCDC 203516).

Empirical formula	$C_{10}H_{10}F_6O_3$
Formula weight	292.18
Crystallization Solvent	Hexanes/ether
Crystal Habit	Block
Crystal size	0.35 x 0.30 x 0.21 mm^3
Crystal color	Colorless

Data Collection

Preliminary Photos	Rotation	
Type of diffractometer	Bruker SMART 1000	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	98(2) K	
θ range for 14175 reflections used in lattice determination	2.17 to 27.99°	
Unit cell dimensions	a = 7.6162(5) Å b = 11.4014(8) Å c = 13.4278(9) Å	α= 100.3010(10)° β= 98.5160(10)° γ = 100.6610(10)°
Volume	1107.44(13) $Å^3$	
Z	4	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.752 Mg/m^3	
F(000)	592	
Data collection program	Bruker SMART v5.054	
θ range for data collection	1.86 to 28.07°	
Completeness to θ = 28.07°	92.1 %	
Index ranges	$-9 \leq h \leq 10$, $-14 \leq k \leq 14$, $-17 \leq l \leq 17$	
Data collection scan type	ω scans at 7 ϕ settings	
Data reduction program	Bruker SAINT v6.022	
Reflections collected	22015	
Independent reflections	4951 [R_{int}= 0.0398]	
Absorption coefficient	0.191 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9611 and 0.9363	

Table 1 (cont.)

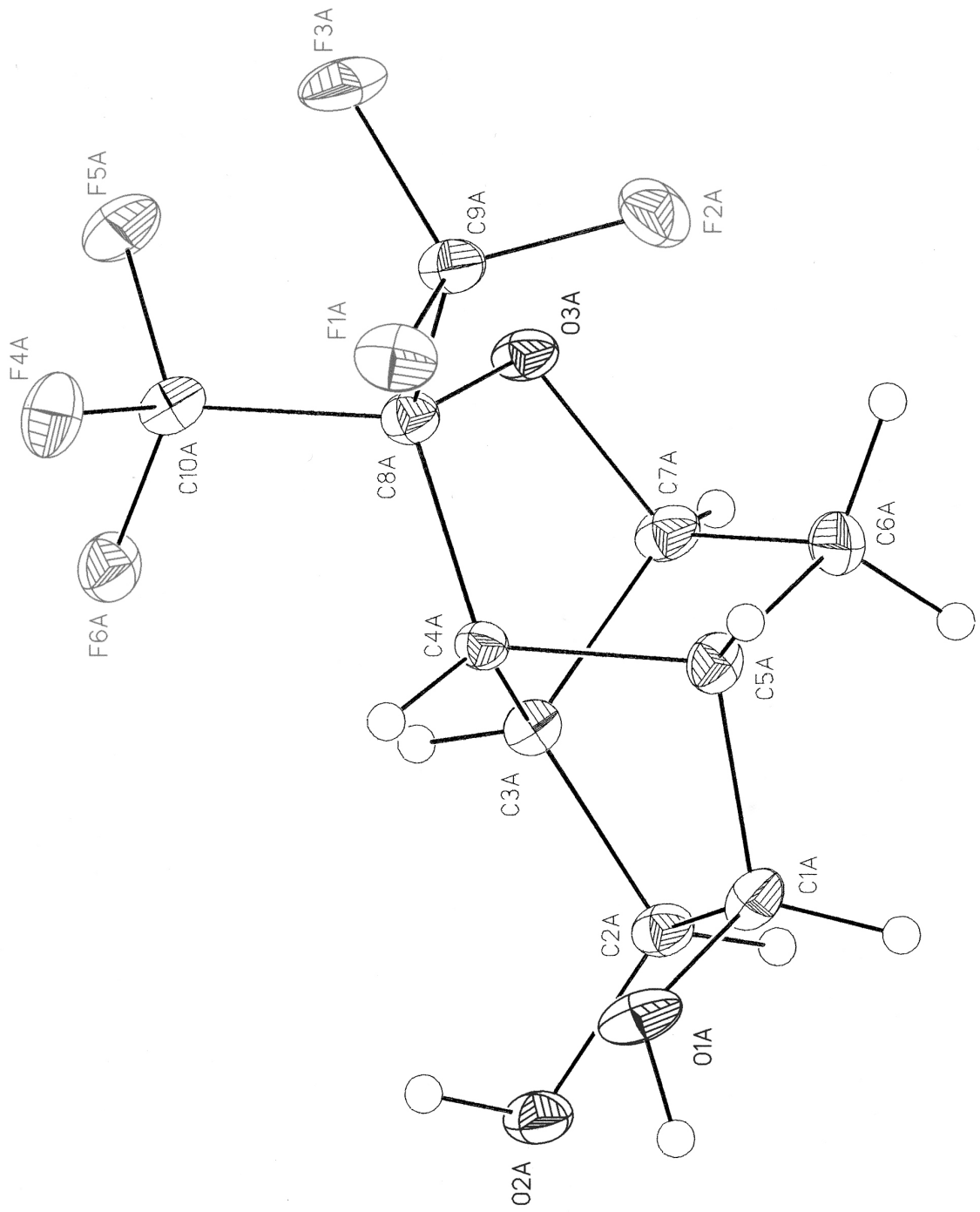
Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	4951 / 0 / 423
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	2.382
Final R indices [$I>2\sigma(I)$, 4252 reflections]	R1 = 0.0317, *w*R2 = 0.0656
R indices (all data)	R1 = 0.0378, *w*R2 = 0.0664
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.003
Average shift/error	0.000
Largest diff. peak and hole	0.416 and -0.320 e.Å^{-3}

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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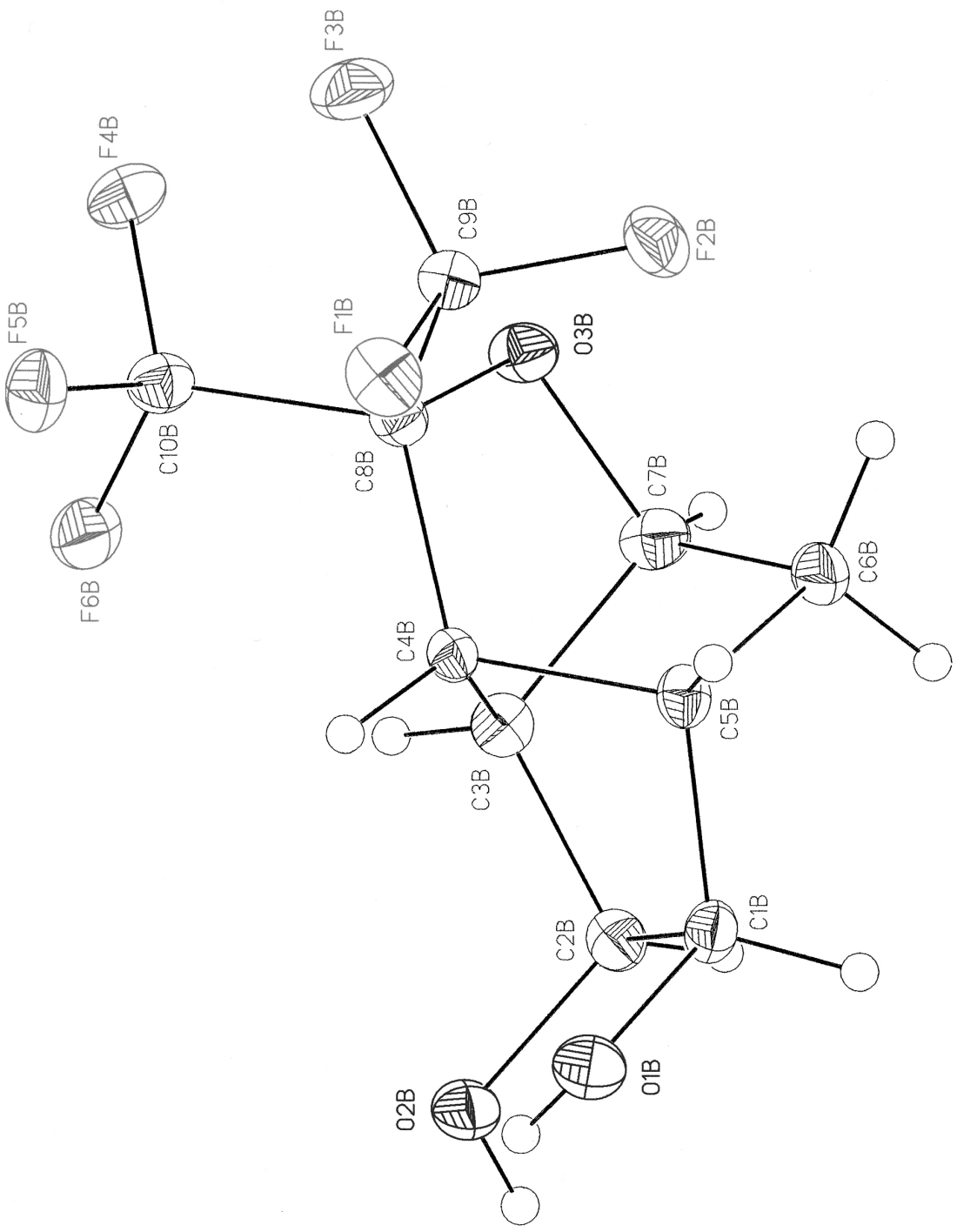
F5A
C9A
F2A
F1A
O3A
F4A
C10A
C8A
C7A
C6A
F6A
C4A
C5A
C3A
C1A
C2A
O1A
O2A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F4B
C9B
F2B
F5B
F1B
O3B
C10B
C8B
C7B
C6B
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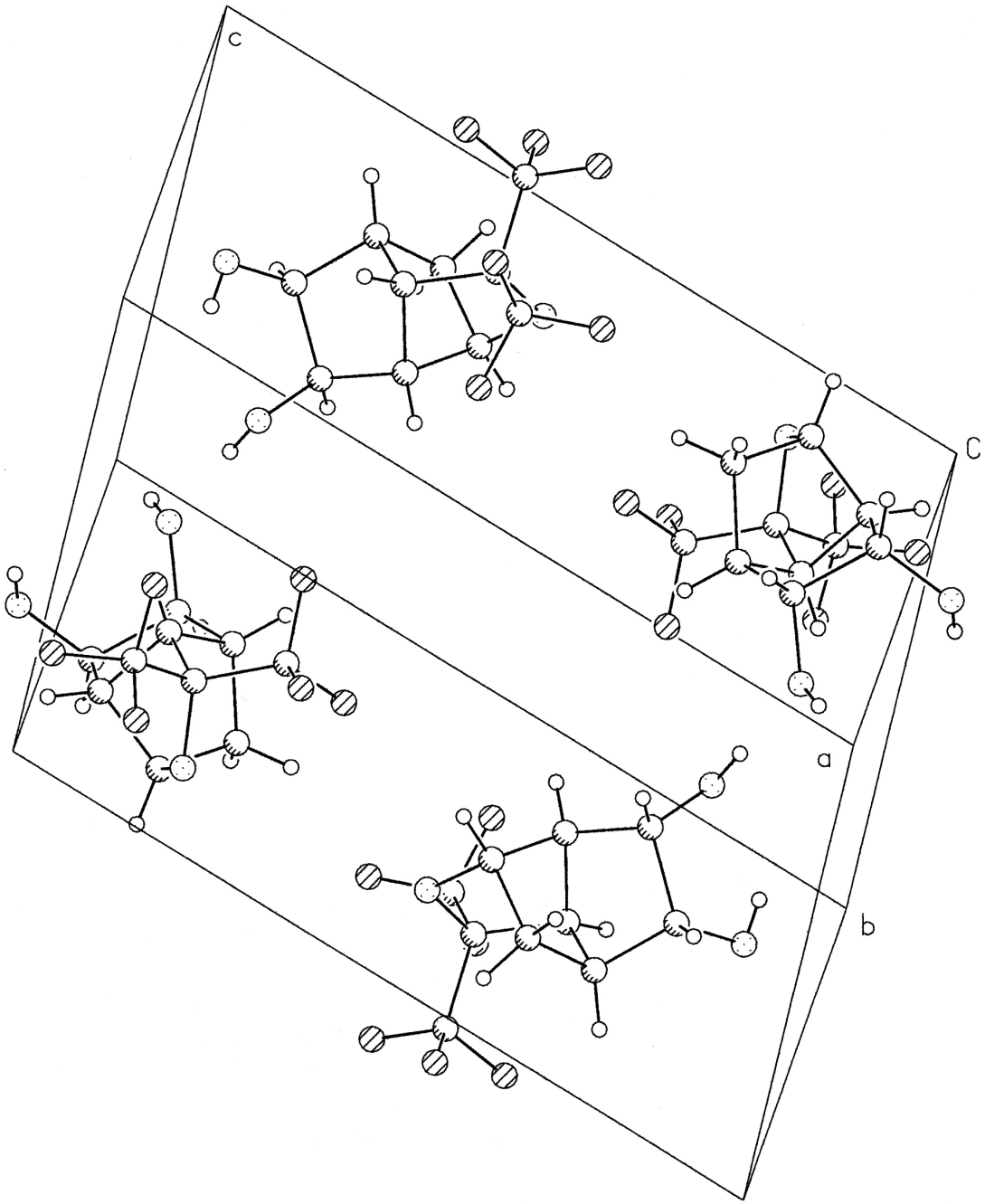
C5B
C3B
C2B
C1B
O1B
O2B


c
C
a
b

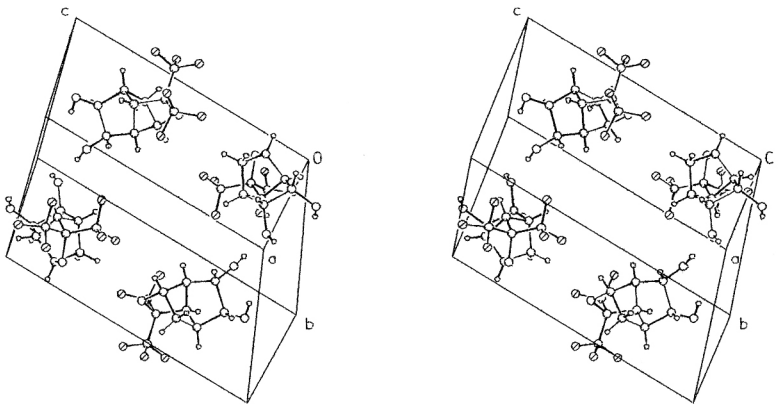

c
O
a
b
c
C
a
b

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DPS01 (CCDC 203516). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
F(1A)	5587(1)	2914(1)	2740(1)	21(1)
F(2A)	3625(1)	2357(1)	3658(1)	22(1)
F(3A)	5154(1)	1093(1)	3042(1)	25(1)
F(4A)	5320(1)	1230(1)	997(1)	23(1)
F(5A)	3567(1)	-377(1)	1238(1)	25(1)
F(6A)	2639(1)	559(1)	82(1)	19(1)
O(1A)	1899(1)	4943(1)	1118(1)	19(1)
O(2A)	-633(1)	3299(1)	-335(1)	17(1)
O(3A)	1222(1)	847(1)	2020(1)	15(1)
C(1A)	615(2)	4189(1)	1531(1)	16(1)
C(2A)	-599(2)	3099(1)	686(1)	14(1)
C(3A)	259(2)	2014(1)	856(1)	13(1)
C(4A)	2238(2)	2607(1)	1421(1)	11(1)
C(5A)	1639(2)	3525(1)	2236(1)	14(1)
C(6A)	164(2)	2681(1)	2630(1)	18(1)
C(7A)	-209(2)	1522(1)	1792(1)	15(1)
C(8A)	2841(2)	1551(1)	1838(1)	13(1)
C(9A)	4305(2)	1977(1)	2831(1)	17(1)
C(10A)	3592(2)	729(1)	1036(1)	16(1)
F(1B)	12913(1)	8455(1)	3099(1)	21(1)
F(2B)	11792(1)	9781(1)	4008(1)	21(1)
F(3B)	13603(1)	8842(1)	4758(1)	25(1)
F(4B)	11998(1)	6859(1)	5331(1)	23(1)
F(5B)	12727(1)	6376(1)	3838(1)	22(1)
F(6B)	10107(1)	5572(1)	4104(1)	23(1)
O(1B)	7355(1)	6884(1)	836(1)	18(1)
O(2B)	5517(1)	5473(1)	1748(1)	19(1)
O(3B)	9542(1)	8092(1)	4688(1)	17(1)
C(1B)	6913(2)	7580(1)	1720(1)	15(1)
C(2B)	5972(2)	6692(1)	2343(1)	15(1)
C(3B)	7447(2)	6803(1)	3284(1)	15(1)
C(4B)	9266(2)	7239(1)	2936(1)	12(1)
C(5B)	8610(2)	8294(1)	2499(1)	14(1)
C(6B)	7876(2)	8956(1)	3417(1)	17(1)
C(7B)	7727(2)	7992(1)	4077(1)	17(1)
C(8B)	10601(2)	7673(1)	3969(1)	14(1)
C(9B)	12237(2)	8702(1)	3963(1)	16(1)
C(10B)	11364(2)	6615(1)	4320(1)	17(1)

Table 3. Bond lengths [Å] and angles [°] for DPS01 (CCDC 203516).

F(1A)-C(9A)	1.3436(14)	C(4B)-C(8B)	1.5347(17)
F(2A)-C(9A)	1.3304(15)	C(4B)-C(5B)	1.5574(16)
F(3A)-C(9A)	1.3423(14)	C(4B)-H(4B)	0.966(12)
F(4A)-C(10A)	1.3463(14)	C(5B)-C(6B)	1.5590(18)
F(5A)-C(10A)	1.3339(14)	C(5B)-H(5B)	0.940(13)
F(6A)-C(10A)	1.3386(14)	C(6B)-C(7B)	1.5292(19)
O(1A)-C(1A)	1.4223(15)	C(6B)-H(6B1)	0.959(12)
O(1A)-H(1A)	0.801(16)	C(6B)-H(6B2)	0.961(14)
O(2A)-C(2A)	1.4267(15)	C(7B)-H(7B)	0.966(13)
O(2A)-H(2A)	0.820(17)	C(8B)-C(9B)	1.5476(17)
O(3A)-C(8A)	1.4207(14)	C(8B)-C(10B)	1.5479(17)
O(3A)-C(7A)	1.4725(14)		
C(1A)-C(5A)	1.5275(17)	C(1A)-O(1A)-H(1A)	113.6(11)
C(1A)-C(2A)	1.5641(17)	C(2A)-O(2A)-H(2A)	107.7(12)
C(1A)-H(1A1)	0.958(12)	C(8A)-O(3A)-C(7A)	105.82(9)
C(2A)-C(3A)	1.5379(17)	O(1A)-C(1A)-C(5A)	108.60(10)
C(2A)-H(2A1)	0.987(12)	O(1A)-C(1A)-C(2A)	111.93(10)
C(3A)-C(7A)	1.5272(18)	C(5A)-C(1A)-C(2A)	101.77(10)
C(3A)-C(4A)	1.5508(16)	O(1A)-C(1A)-H(1A1)	110.5(8)
C(3A)-H(3A)	0.954(14)	C(5A)-C(1A)-H(1A1)	112.7(8)
C(4A)-C(8A)	1.5299(16)	C(2A)-C(1A)-H(1A1)	111.1(8)
C(4A)-C(5A)	1.5547(17)	O(2A)-C(2A)-C(3A)	112.11(10)
C(4A)-H(4A)	0.902(13)	O(2A)-C(2A)-C(1A)	113.23(10)
C(5A)-C(6A)	1.5575(18)	C(3A)-C(2A)-C(1A)	103.28(9)
C(5A)-H(5A)	0.949(13)	O(2A)-C(2A)-H(2A1)	105.0(7)
C(6A)-C(7A)	1.5254(19)	C(3A)-C(2A)-H(2A1)	112.6(7)
C(6A)-H(6A1)	0.992(14)	C(1A)-C(2A)-H(2A1)	110.8(7)
C(6A)-H(6A2)	0.950(14)	C(7A)-C(3A)-C(4A)	91.89(10)
C(7A)-H(7A)	0.948(13)	C(7A)-C(3A)-C(2A)	113.25(10)
C(8A)-C(10A)	1.5439(17)	C(4A)-C(3A)-C(2A)	104.61(9)
C(8A)-C(9A)	1.5452(17)	C(7A)-C(3A)-H(3A)	113.2(8)
F(1B)-C(9B)	1.3441(15)	C(4A)-C(3A)-H(3A)	117.1(8)
F(2B)-C(9B)	1.3291(14)	C(2A)-C(3A)-H(3A)	114.6(8)
F(3B)-C(9B)	1.3401(14)	C(8A)-C(4A)-C(5A)	113.61(10)
F(4B)-C(10B)	1.3321(14)	C(8A)-C(4A)-C(3A)	102.18(10)
F(5B)-C(10B)	1.3456(14)	C(5A)-C(4A)-C(3A)	93.49(9)
F(6B)-C(10B)	1.3368(15)	C(8A)-C(4A)-H(4A)	112.9(8)
O(1B)-C(1B)	1.4261(15)	C(5A)-C(4A)-H(4A)	116.4(8)
O(1B)-H(1B)	0.777(17)	C(3A)-C(4A)-H(4A)	116.0(8)
O(2B)-C(2B)	1.4262(15)	C(1A)-C(5A)-C(4A)	100.16(10)
O(2B)-H(2B)	0.815(15)	C(1A)-C(5A)-C(6A)	106.24(10)
O(3B)-C(8B)	1.4204(14)	C(4A)-C(5A)-C(6A)	102.61(10)
O(3B)-C(7B)	1.4723(15)	C(1A)-C(5A)-H(5A)	112.0(8)
C(1B)-C(2B)	1.5646(17)	C(4A)-C(5A)-H(5A)	117.5(8)
C(1B)-C(5B)	1.5271(17)	C(6A)-C(5A)-H(5A)	116.4(8)
C(1B)-H(1B1)	0.968(13)	C(7A)-C(6A)-C(5A)	100.57(10)
C(2B)-C(3B)	1.5318(17)	C(7A)-C(6A)-H(6A1)	113.4(8)
C(2B)-H(2B1)	0.948(13)	C(5A)-C(6A)-H(6A1)	112.7(8)
C(3B)-C(4B)	1.5508(17)	C(7A)-C(6A)-H(6A2)	110.6(8)
C(3B)-C(7B)	1.5261(18)	C(5A)-C(6A)-H(6A2)	111.9(8)
C(3B)-H(3B)	0.951(14)	H(6A1)-C(6A)-H(6A2)	107.7(11)

O(3A)-C(7A)-C(6A)	108.21(10)	C(2B)-C(3B)-H(3B)	110.0(8)
O(3A)-C(7A)-C(3A)	103.04(9)	C(8B)-C(4B)-C(5B)	113.80(10)
C(6A)-C(7A)-C(3A)	102.66(10)	C(8B)-C(4B)-C(3B)	101.88(10)
O(3A)-C(7A)-H(7A)	105.7(7)	C(5B)-C(4B)-C(3B)	93.75(9)
C(6A)-C(7A)-H(7A)	116.2(8)	C(8B)-C(4B)-H(4B)	113.9(7)
C(3A)-C(7A)-H(7A)	119.9(8)	C(5B)-C(4B)-H(4B)	113.8(7)
O(3A)-C(8A)-C(4A)	104.64(9)	C(3B)-C(4B)-H(4B)	117.8(7)
O(3A)-C(8A)-C(10A)	107.95(10)	C(6B)-C(5B)-C(4B)	102.24(10)
C(4A)-C(8A)-C(10A)	111.95(10)	C(6B)-C(5B)-C(1B)	104.95(10)
O(3A)-C(8A)-C(9A)	111.07(10)	C(4B)-C(5B)-C(1B)	100.74(10)
C(4A)-C(8A)-C(9A)	113.35(10)	C(6B)-C(5B)-H(5B)	115.3(8)
C(10A)-C(8A)-C(9A)	107.77(9)	C(4B)-C(5B)-H(5B)	116.7(8)
F(3A)-C(9A)-F(2A)	106.67(10)	C(1B)-C(5B)-H(5B)	114.9(8)
F(3A)-C(9A)-F(1A)	107.15(9)	C(5B)-C(6B)-C(7B)	100.75(10)
F(2A)-C(9A)-F(1A)	107.15(10)	C(5B)-C(6B)-H(6B1)	110.4(8)
F(3A)-C(9A)-C(8A)	112.70(10)	C(7B)-C(6B)-H(6B1)	109.2(8)
F(2A)-C(9A)-C(8A)	112.58(10)	C(5B)-C(6B)-H(6B2)	114.0(8)
F(1A)-C(9A)-C(8A)	110.27(10)	C(7B)-C(6B)-H(6B2)	111.1(8)
F(6A)-C(10A)-F(4A)	106.17(10)	H(6B1)-C(6B)-H(6B2)	111.0(11)
F(6A)-C(10A)-F(5A)	106.65(10)	O(3B)-C(7B)-C(3B)	103.01(10)
F(4A)-C(10A)-F(5A)	107.59(10)	O(3B)-C(7B)-C(6B)	108.23(10)
F(6A)-C(10A)-C(8A)	112.20(9)	C(3B)-C(7B)-C(6B)	102.74(10)
F(4A)-C(10A)-C(8A)	111.38(10)	O(3B)-C(7B)-H(7B)	105.9(7)
F(5A)-C(10A)-C(8A)	112.48(10)	C(3B)-C(7B)-H(7B)	118.4(8)
C(1B)-O(1B)-H(1B)	103.8(12)	C(6B)-C(7B)-H(7B)	117.4(7)
C(2B)-O(2B)-H(2B)	108.1(11)	O(3B)-C(8B)-C(4B)	104.71(9)
C(8B)-O(3B)-C(7B)	105.68(9)	O(3B)-C(8B)-C(9B)	110.45(10)
O(1B)-C(1B)-C(2B)	109.26(10)	C(4B)-C(8B)-C(9B)	113.74(10)
O(1B)-C(1B)-C(5B)	112.02(10)	O(3B)-C(8B)-C(10B)	108.05(10)
C(2B)-C(1B)-C(5B)	102.26(10)	C(4B)-C(8B)-C(10B)	112.01(10)
O(1B)-C(1B)-H(1B1)	109.4(8)	C(9B)-C(8B)-C(10B)	107.75(10)
C(2B)-C(1B)-H(1B1)	113.4(8)	F(1B)-C(9B)-F(3B)	106.96(10)
C(5B)-C(1B)-H(1B1)	110.4(8)	F(1B)-C(9B)-F(2B)	107.29(10)
O(2B)-C(2B)-C(3B)	109.98(10)	F(3B)-C(9B)-F(2B)	106.64(10)
O(2B)-C(2B)-C(1B)	109.92(10)	F(1B)-C(9B)-C(8B)	110.58(10)
C(3B)-C(2B)-C(1B)	103.43(10)	F(3B)-C(9B)-C(8B)	112.49(10)
O(2B)-C(2B)-H(2B1)	111.2(8)	F(2B)-C(9B)-C(8B)	112.55(10)
C(3B)-C(2B)-H(2B1)	110.1(8)	F(4B)-C(10B)-F(5B)	107.76(10)
C(1B)-C(2B)-H(2B1)	112.0(8)	F(4B)-C(10B)-F(6B)	106.82(10)
C(4B)-C(3B)-C(7B)	91.91(10)	F(5B)-C(10B)-F(6B)	106.22(10)
C(4B)-C(3B)-C(2B)	104.83(10)	F(4B)-C(10B)-C(8B)	112.23(10)
C(7B)-C(3B)-C(2B)	113.24(11)	F(5B)-C(10B)-C(8B)	111.20(10)
C(4B)-C(3B)-H(3B)	120.4(8)	F(6B)-C(10B)-C(8B)	112.27(10)
C(7B)-C(3B)-H(3B)	115.3(8)		

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for DPS01 (CCDC 203516). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
F(1A)	153(4)	222(4)	211(4)	58(3)	-27(3)	-13(3)
F(2A)	247(4)	299(5)	126(4)	46(3)	19(3)	65(3)
F(3A)	259(4)	250(4)	250(4)	102(4)	-42(3)	119(4)
F(4A)	138(4)	263(4)	282(5)	27(4)	70(3)	62(3)
F(5A)	346(5)	143(4)	316(5)	72(3)	72(4)	124(3)
F(6A)	219(4)	191(4)	156(4)	-10(3)	12(3)	70(3)
O(1A)	146(5)	132(5)	293(6)	99(4)	-9(4)	21(4)
O(2A)	146(5)	197(5)	184(5)	87(4)	12(4)	56(4)
O(3A)	120(4)	143(5)	201(5)	88(4)	31(4)	27(4)
C(1A)	147(6)	130(6)	208(7)	35(5)	41(5)	52(5)
C(2A)	105(6)	147(6)	170(7)	60(5)	16(5)	28(5)
C(3A)	117(6)	106(6)	141(7)	20(5)	12(5)	12(5)
C(4A)	114(6)	117(6)	117(6)	41(5)	24(5)	22(5)
C(5A)	148(6)	116(6)	139(7)	-3(5)	14(5)	28(5)
C(6A)	155(7)	226(7)	177(7)	67(6)	67(5)	71(6)
C(7A)	100(6)	163(7)	213(7)	80(5)	29(5)	34(5)
C(8A)	115(6)	123(6)	149(6)	47(5)	23(5)	20(5)
C(9A)	164(6)	168(7)	173(7)	65(5)	18(5)	50(5)
C(10A)	151(6)	144(7)	196(7)	47(5)	14(5)	49(5)
F(1B)	195(4)	220(4)	233(4)	48(3)	105(3)	29(3)
F(2B)	219(4)	128(4)	261(4)	21(3)	36(3)	20(3)
F(3B)	179(4)	250(4)	259(4)	44(4)	-56(3)	-12(3)
F(4B)	272(4)	286(4)	137(4)	60(3)	-19(3)	73(4)
F(5B)	231(4)	233(4)	251(4)	69(3)	79(3)	119(3)
F(6B)	243(4)	167(4)	258(4)	94(3)	-17(3)	9(3)
O(1B)	197(5)	203(5)	147(5)	29(4)	44(4)	28(4)
O(2B)	138(5)	161(5)	225(5)	7(4)	0(4)	1(4)
O(3B)	149(4)	213(5)	134(5)	8(4)	38(4)	43(4)
C(1B)	154(6)	156(7)	152(7)	41(5)	33(5)	67(5)
C(2B)	130(6)	147(7)	178(7)	23(5)	42(5)	27(5)
C(3B)	151(6)	150(7)	149(7)	54(5)	49(5)	19(5)
C(4B)	135(6)	116(6)	119(6)	16(5)	25(5)	41(5)
C(5B)	142(6)	119(6)	166(7)	52(5)	43(5)	29(5)
C(6B)	152(7)	162(7)	194(7)	0(6)	25(5)	59(5)
C(7B)	125(6)	229(7)	153(7)	14(6)	39(5)	38(5)
C(8B)	147(6)	144(6)	115(6)	15(5)	35(5)	36(5)
C(9B)	171(6)	158(7)	154(7)	20(5)	16(5)	36(5)
C(10B)	175(7)	180(7)	146(7)	38(5)	19(5)	28(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for DPS01 (CCDC 203516).

	x	y	z	U_{iso}
H(1A)	1490(20)	5438(15)	858(12)	35(5)
H(2A)	330(20)	3195(15)	-501(13)	40(5)
H(1A1)	-115(17)	4665(12)	1878(10)	12(3)
H(2A1)	-1880(17)	2972(11)	772(9)	11(3)
H(3A)	101(17)	1389(12)	254(11)	16(3)
H(4A)	2993(16)	2933(11)	1036(10)	9(3)
H(5A)	2565(17)	4074(12)	2748(10)	15(3)
H(6A1)	597(17)	2557(12)	3329(11)	16(3)
H(6A2)	-905(19)	2996(12)	2649(10)	20(4)
H(7A)	-1326(17)	963(12)	1733(10)	12(3)
H(1B)	6820(20)	6224(16)	803(13)	34(5)
H(2B)	4420(20)	5288(14)	1545(12)	29(5)
H(1B1)	6177(17)	8130(12)	1506(10)	17(3)
H(2B1)	4935(18)	6932(12)	2555(10)	14(3)
H(3B)	7247(18)	6079(13)	3549(10)	18(4)
H(4B)	9649(16)	6668(11)	2435(9)	6(3)
H(5B)	9458(17)	8799(12)	2231(10)	13(3)
H(6B1)	6685(17)	9081(11)	3184(10)	11(3)
H(6B2)	8676(18)	9709(13)	3795(11)	19(4)
H(7B)	6919(17)	8041(11)	4566(10)	11(3)

Table 6. Hydrogen bonds for DPS01 (CCDC 203516) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1A)-H(1A)...O(2A)#1	0.801(16)	1.888(17)	2.6873(13)	175.4(17)
O(2A)-H(2A)...O(1B)#2	0.820(17)	1.897(17)	2.7143(14)	174.5(17)
O(1B)-H(1B)...O(2B)	0.777(17)	1.943(17)	2.5346(14)	132.7(16)
O(2B)-H(2B)...O(1A)	0.815(15)	1.867(16)	2.6803(13)	177.0(16)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z #2 -x+1,-y+1,-z