Table 1. Crystal data and structure refinement for SPB01 (CCDC 205591).

Empirical formula	$[C_{23}H_{31}N_2O_3]^+Cl^- \cdot C_4H_8O$
Formula weight	491.05
Crystallization Solvent	THF
Crystal Habit	Column
Crystal size	0.37 x 0.07 x 0.04 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 7474 reflections used in lattice determination	2.19 to 28.25°
Unit cell dimensions	a = 6.8505(5) Å b = 18.5579(13) Å β= 103.7750(10)° c = 10.8735(8) Å
Volume	1342.60(17) $Å^3$
Z	2
Crystal system	Monoclinic
Space group	$P2_1$
Density (calculated)	1.215 Mg/m^3
F(000)	528
Data collection program	Bruker SMART v5.054
θ range for data collection	1.93 to 28.43°
Completeness to θ = 28.43°	92.9 %
Index ranges	$-8 \le h \le 9, -23 \le k \le 24, -14 \le l \le 14$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.022
Reflections collected	14842
Independent reflections	5984 [R_{int}= 0.0441]
Absorption coefficient	0.176 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9930 and 0.9377

Table 1 (cont.)

Structure solution and Refinement

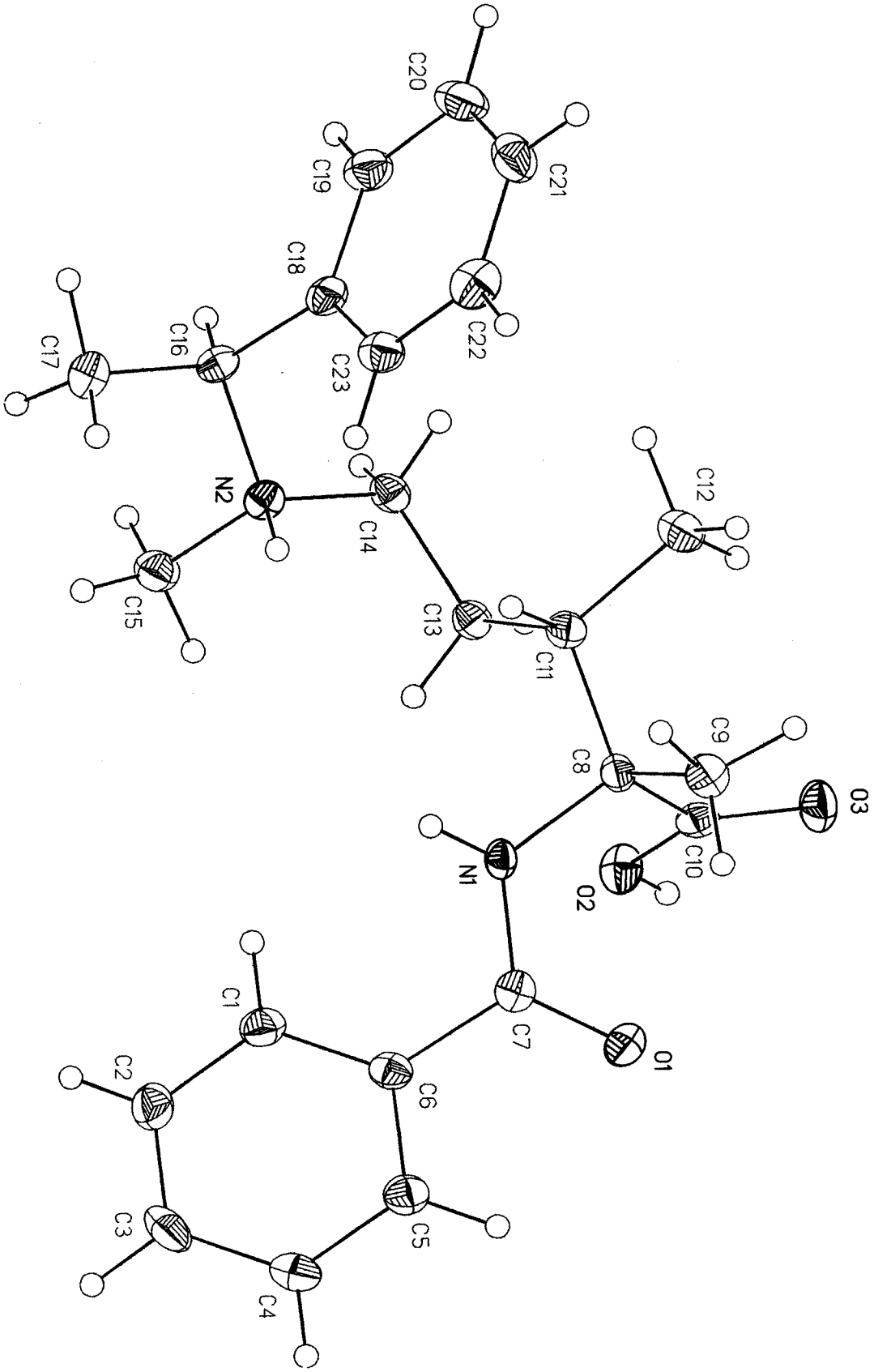
Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	5984 / 1 / 463
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.109
Final R indices [$I>2\sigma(I)$, 5191 reflections]	R1 = 0.0317, *w*R2 = 0.0533
R indices (all data)	R1 = 0.0407, *w*R2 = 0.0551
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Absolute structure parameter	0.05(3)
Largest diff. peak and hole	0.280 and -0.215 e.Å^{-3}

Special Refinement Details

The crystals contain THF as a solvent of crystallization. It was included in refinement without restraints. The hydrogen bonds formed with it are listed in Table 6. The chloride anion forms hydrogen bonds with both nitrogen atoms, see Table 6.

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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C21
C19
C18
C22
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C17
C23
N2
C14
C12
C15
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C11
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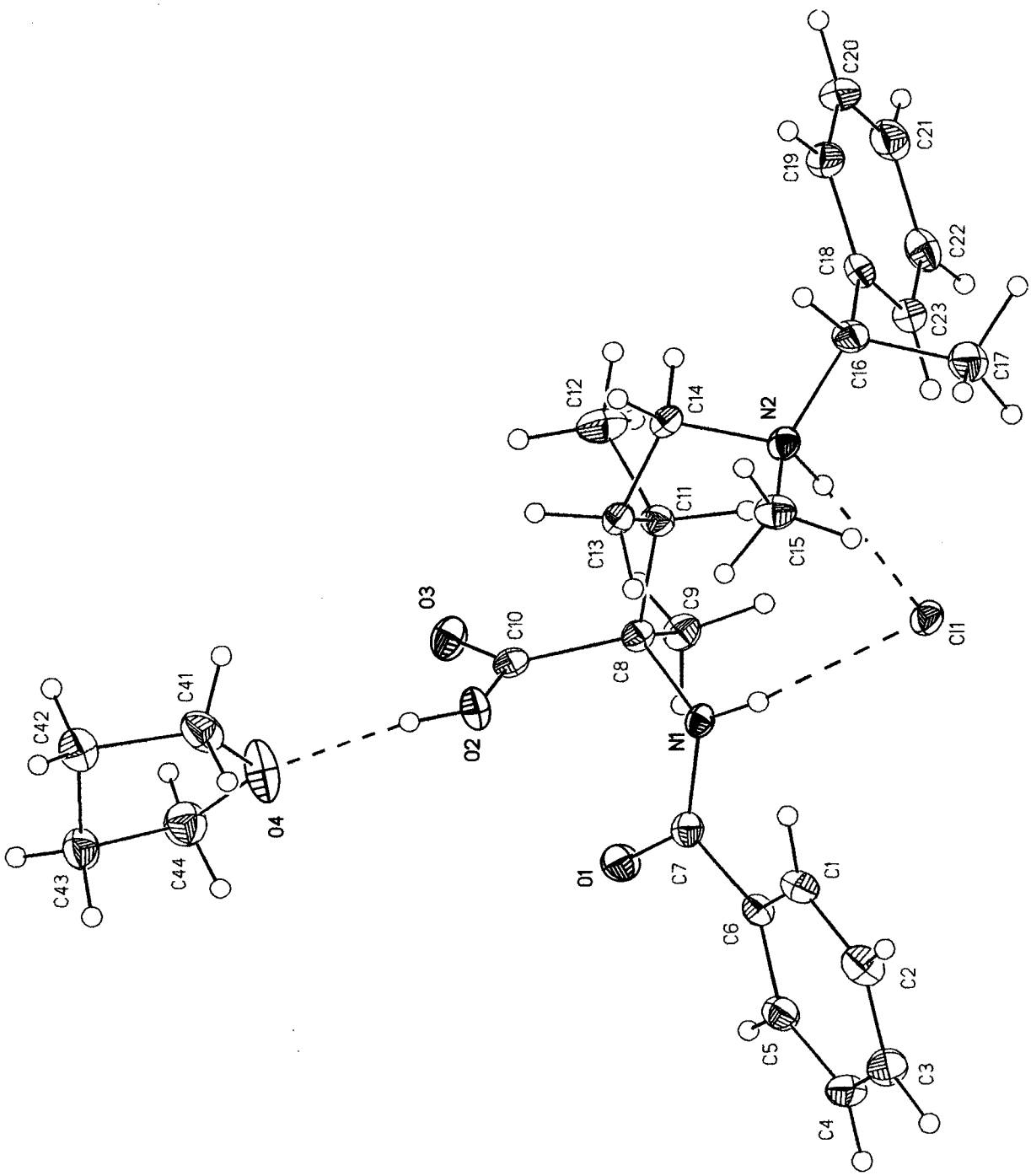
C1
C7
O1
C2
C6
C3
C5
C4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C9
C8
Cl1
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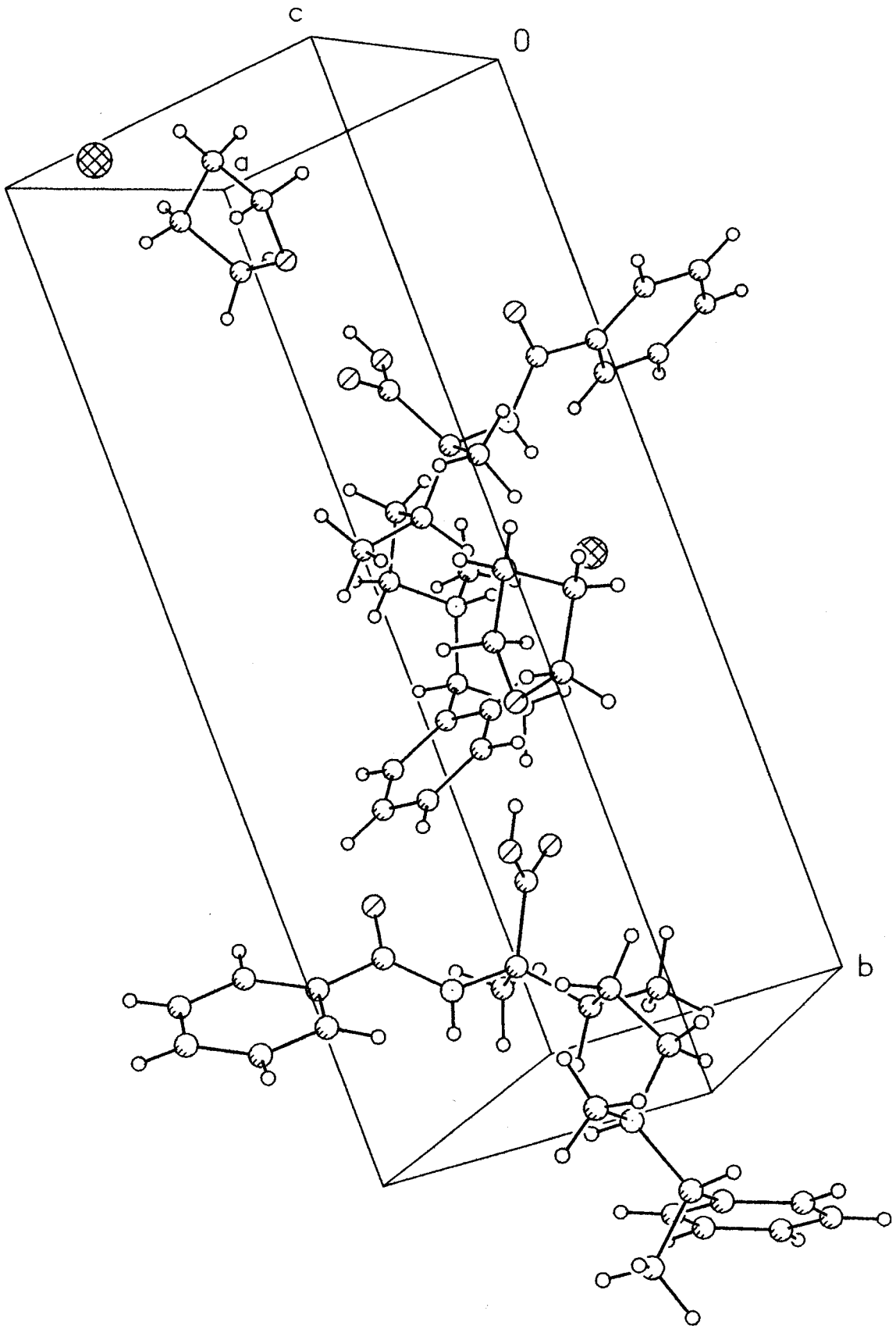
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0
a
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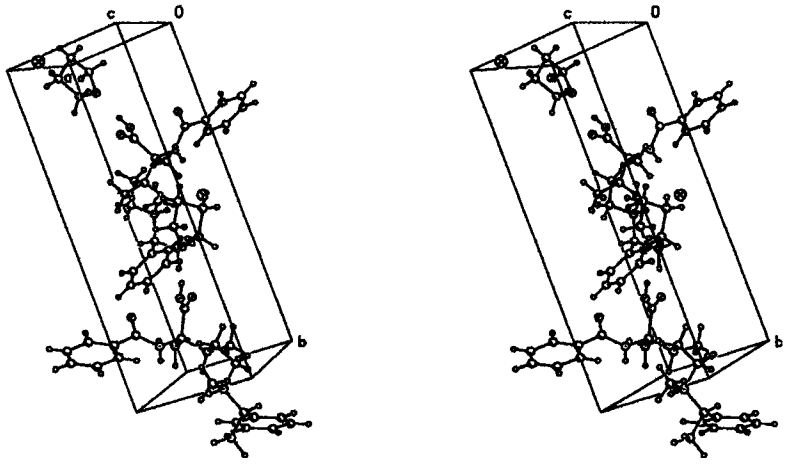
c
0
b
c
0
b

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for SPB01 (CCDC 205591). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Cl(1)	7412(1)	93(1)	9582(1)	19(1)
O(1)	8741(2)	7568(1)	7781(1)	22(1)
O(2)	4375(2)	7499(1)	7535(1)	20(1)
O(3)	4361(2)	7642(1)	5491(1)	23(1)
N(1)	7095(2)	8550(1)	8228(1)	14(1)
N(2)	2841(2)	10119(1)	9203(1)	15(1)
C(1)	9250(3)	8371(1)	10873(2)	20(1)
C(2)	10539(3)	8361(1)	12068(2)	23(1)
C(3)	12428(3)	8057(1)	12262(2)	25(1)
C(4)	13028(3)	7751(1)	11247(2)	23(1)
C(5)	11738(3)	7742(1)	10055(2)	19(1)
C(6)	9846(2)	8060(1)	9855(2)	17(1)
C(7)	8522(2)	8037(1)	8534(2)	16(1)
C(8)	5784(2)	8589(1)	6944(2)	15(1)
C(9)	7021(3)	8807(1)	6004(2)	19(1)
C(10)	4803(2)	7854(1)	6572(2)	17(1)
C(11)	4119(3)	9160(1)	6947(2)	16(1)
C(12)	2753(3)	9272(1)	5630(2)	27(1)
C(13)	2842(2)	8988(1)	7899(2)	16(1)
C(14)	1638(2)	9628(1)	8193(2)	16(1)
C(15)	2920(3)	9808(1)	10483(2)	21(1)
C(16)	2025(3)	10887(1)	9087(2)	17(1)
C(17)	3241(3)	11362(1)	10142(2)	23(1)
C(18)	2008(3)	11194(1)	7797(2)	18(1)
C(19)	228(3)	11479(1)	7063(2)	23(1)
C(20)	192(3)	11809(1)	5913(2)	27(1)
C(21)	1942(3)	11858(1)	5488(2)	26(1)
C(22)	3708(3)	11567(1)	6200(2)	24(1)
C(23)	3758(3)	11243(1)	7355(2)	20(1)
O(4)	2896(2)	6196(1)	7053(1)	37(1)
C(41)	1090(3)	6073(1)	7499(2)	27(1)
C(42)	199(3)	5385(1)	6862(2)	25(1)
C(43)	2036(3)	4985(1)	6672(2)	24(1)
C(44)	3280(3)	5594(1)	6338(2)	28(1)

Table 3. Bond lengths [Å] and angles [°] for SPB01 (CCDC 205591).

O(1)-C(7)	1.2286(18)	C(19)-C(20)	1.388(3)
O(2)-C(10)	1.3281(19)	C(19)-H(19)	0.947(18)
O(2)-H(2)	0.80(2)	C(20)-C(21)	1.386(3)
O(3)-C(10)	1.2074(19)	C(20)-H(20)	0.933(19)
N(1)-C(7)	1.348(2)	C(21)-C(22)	1.381(3)
N(1)-C(8)	1.471(2)	C(21)-H(21)	0.929(18)
N(1)-H(1)	0.834(18)	C(22)-C(23)	1.386(2)
N(2)-C(15)	1.495(2)	C(22)-H(22)	1.000(19)
N(2)-C(14)	1.511(2)	C(23)-H(23)	0.921(17)
N(2)-C(16)	1.526(2)	O(4)-C(44)	1.420(2)
N(2)-H(2A)	0.879(17)	O(4)-C(41)	1.451(2)
C(1)-C(2)	1.386(2)	C(41)-C(42)	1.510(3)
C(1)-C(6)	1.394(2)	C(41)-H(41A)	0.95(2)
C(1)-H(1A)	0.941(17)	C(41)-H(41B)	1.02(2)
C(2)-C(3)	1.381(3)	C(42)-C(43)	1.517(3)
C(2)-H(2B)	0.917(17)	C(42)-H(42A)	0.944(19)
C(3)-C(4)	1.388(2)	C(42)-H(42B)	0.93(2)
C(3)-H(3)	0.962(16)	C(43)-C(44)	1.511(3)
C(4)-C(5)	1.384(2)	C(43)-H(43A)	0.989(18)
C(4)-H(4)	0.954(18)	C(43)-H(43B)	0.955(19)
C(5)-C(6)	1.393(2)	C(44)-H(44A)	0.95(2)
C(5)-H(5)	0.985(16)	C(44)-H(44B)	0.962(18)
C(6)-C(7)	1.506(2)		
C(8)-C(9)	1.530(2)	C(10)-O(2)-H(2)	110.0(15)
C(8)-C(10)	1.532(2)	C(7)-N(1)-C(8)	120.77(13)
C(8)-C(11)	1.558(2)	C(7)-N(1)-H(1)	117.8(12)
C(9)-H(9A)	1.028(19)	C(8)-N(1)-H(1)	118.5(12)
C(9)-H(9B)	0.996(17)	C(15)-N(2)-C(14)	109.68(14)
C(9)-H(9C)	0.932(18)	C(15)-N(2)-C(16)	111.61(13)
C(11)-C(12)	1.527(2)	C(14)-N(2)-C(16)	112.19(12)
C(11)-C(13)	1.541(2)	C(15)-N(2)-H(2A)	104.4(11)
C(11)-H(11)	1.062(16)	C(14)-N(2)-H(2A)	110.8(11)
C(12)-H(12A)	1.00(2)	C(16)-N(2)-H(2A)	107.9(13)
C(12)-H(12B)	1.027(18)	C(2)-C(1)-C(6)	119.84(17)
C(12)-H(12C)	0.998(19)	C(2)-C(1)-H(1A)	121.0(10)
C(13)-C(14)	1.523(2)	C(6)-C(1)-H(1A)	119.1(10)
C(13)-H(13A)	0.986(16)	C(3)-C(2)-C(1)	120.87(17)
C(13)-H(13B)	0.977(16)	C(3)-C(2)-H(2B)	121.7(11)
C(14)-H(14A)	0.941(16)	C(1)-C(2)-H(2B)	117.4(11)
C(14)-H(14B)	1.013(17)	C(2)-C(3)-C(4)	119.36(16)
C(15)-H(15A)	0.963(17)	C(2)-C(3)-H(3)	120.9(9)
C(15)-H(15B)	0.979(19)	C(4)-C(3)-H(3)	119.7(9)
C(15)-H(15C)	0.966(17)	C(5)-C(4)-C(3)	120.34(17)
C(16)-C(18)	1.511(2)	C(5)-C(4)-H(4)	120.0(10)
C(16)-C(17)	1.527(3)	C(3)-C(4)-H(4)	119.6(10)
C(16)-H(16)	0.953(17)	C(4)-C(5)-C(6)	120.31(17)
C(17)-H(17A)	0.98(2)	C(4)-C(5)-H(5)	120.9(9)
C(17)-H(17B)	0.944(19)	C(6)-C(5)-H(5)	118.7(9)
C(17)-H(17C)	1.08(2)	C(5)-C(6)-C(1)	119.25(15)
C(18)-C(19)	1.393(2)	C(5)-C(6)-C(7)	117.66(15)
C(18)-C(23)	1.397(2)	C(1)-C(6)-C(7)	123.08(15)

O(1)-C(7)-N(1)	122.14(14)	N(2)-C(16)-H(16)	102.5(10)
O(1)-C(7)-C(6)	120.96(14)	C(17)-C(16)-H(16)	112.8(10)
N(1)-C(7)-C(6)	116.91(14)	C(16)-C(17)-H(17A)	112.5(11)
N(1)-C(8)-C(9)	109.72(13)	C(16)-C(17)-H(17B)	111.7(12)
N(1)-C(8)-C(10)	109.72(13)	H(17A)-C(17)-H(17B)	107.7(16)
C(9)-C(8)-C(10)	109.72(13)	C(16)-C(17)-H(17C)	108.4(11)
N(1)-C(8)-C(11)	108.26(12)	H(17A)-C(17)-H(17C)	105.0(16)
C(9)-C(8)-C(11)	110.03(13)	H(17B)-C(17)-H(17C)	111.5(16)
C(10)-C(8)-C(11)	109.37(13)	C(19)-C(18)-C(23)	118.92(16)
C(8)-C(9)-H(9A)	111.4(10)	C(19)-C(18)-C(16)	119.20(16)
C(8)-C(9)-H(9B)	114.7(10)	C(23)-C(18)-C(16)	121.75(16)
H(9A)-C(9)-H(9B)	105.3(14)	C(20)-C(19)-C(18)	120.65(18)
C(8)-C(9)-H(9C)	106.8(10)	C(20)-C(19)-H(19)	120.4(11)
H(9A)-C(9)-H(9C)	108.1(15)	C(18)-C(19)-H(19)	118.9(11)
H(9B)-C(9)-H(9C)	110.4(14)	C(21)-C(20)-C(19)	119.86(18)
O(3)-C(10)-O(2)	123.75(15)	C(21)-C(20)-H(20)	121.0(11)
O(3)-C(10)-C(8)	122.64(15)	C(19)-C(20)-H(20)	119.1(11)
O(2)-C(10)-C(8)	113.48(14)	C(22)-C(21)-C(20)	119.92(18)
C(12)-C(11)-C(13)	109.83(15)	C(22)-C(21)-H(21)	118.1(11)
C(12)-C(11)-C(8)	112.08(14)	C(20)-C(21)-H(21)	121.9(11)
C(13)-C(11)-C(8)	113.13(13)	C(21)-C(22)-C(23)	120.46(18)
C(12)-C(11)-H(11)	108.9(8)	C(21)-C(22)-H(22)	121.8(11)
C(13)-C(11)-H(11)	109.1(8)	C(23)-C(22)-H(22)	117.5(11)
C(8)-C(11)-H(11)	103.5(8)	C(22)-C(23)-C(18)	120.16(17)
C(11)-C(12)-H(12A)	110.9(12)	C(22)-C(23)-H(23)	121.2(10)
C(11)-C(12)-H(12B)	110.5(9)	C(18)-C(23)-H(23)	118.6(10)
H(12A)-C(12)-H(12B)	109.6(15)	C(44)-O(4)-C(41)	110.08(14)
C(11)-C(12)-H(12C)	108.3(10)	O(4)-C(41)-C(42)	105.26(15)
H(12A)-C(12)-H(12C)	111.0(16)	O(4)-C(41)-H(41A)	108.8(11)
H(12B)-C(12)-H(12C)	106.4(14)	C(42)-C(41)-H(41A)	113.7(12)
C(14)-C(13)-C(11)	113.69(14)	O(4)-C(41)-H(41B)	105.8(11)
C(14)-C(13)-H(13A)	107.0(9)	C(42)-C(41)-H(41B)	110.2(12)
C(11)-C(13)-H(13A)	110.0(9)	H(41A)-C(41)-H(41B)	112.5(16)
C(14)-C(13)-H(13B)	110.3(9)	C(41)-C(42)-C(43)	102.60(16)
C(11)-C(13)-H(13B)	112.5(9)	C(41)-C(42)-H(42A)	108.5(12)
H(13A)-C(13)-H(13B)	102.7(13)	C(43)-C(42)-H(42A)	107.0(12)
N(2)-C(14)-C(13)	113.16(13)	C(41)-C(42)-H(42B)	110.9(12)
N(2)-C(14)-H(14A)	105.3(10)	C(43)-C(42)-H(42B)	112.5(12)
C(13)-C(14)-H(14A)	111.0(9)	H(42A)-C(42)-H(42B)	114.7(17)
N(2)-C(14)-H(14B)	106.5(9)	C(44)-C(43)-C(42)	101.56(16)
C(13)-C(14)-H(14B)	114.0(9)	C(44)-C(43)-H(43A)	110.9(10)
H(14A)-C(14)-H(14B)	106.2(13)	C(42)-C(43)-H(43A)	108.2(10)
N(2)-C(15)-H(15A)	110.0(10)	C(44)-C(43)-H(43B)	111.4(11)
N(2)-C(15)-H(15B)	109.0(10)	C(42)-C(43)-H(43B)	115.1(11)
H(15A)-C(15)-H(15B)	110.4(13)	H(43A)-C(43)-H(43B)	109.5(15)
N(2)-C(15)-H(15C)	109.5(9)	O(4)-C(44)-C(43)	105.57(15)
H(15A)-C(15)-H(15C)	106.9(14)	O(4)-C(44)-H(44A)	109.0(12)
H(15B)-C(15)-H(15C)	111.0(14)	C(43)-C(44)-H(44A)	113.5(12)
C(18)-C(16)-N(2)	110.52(13)	O(4)-C(44)-H(44B)	108.1(10)
C(18)-C(16)-C(17)	111.21(15)	C(43)-C(44)-H(44B)	111.0(11)
N(2)-C(16)-C(17)	110.77(14)	H(44A)-C(44)-H(44B)	109.5(15)
C(18)-C(16)-H(16)	108.7(10)		

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for SPB01 (CCDC 205591). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	126(2)	192(2)	238(2)	-46(2)	35(2)	-1(2)
O(1)	229(6)	202(7)	211(6)	-46(5)	45(5)	35(5)
O(2)	250(7)	150(7)	210(6)	-21(5)	67(5)	-66(5)
O(3)	245(7)	263(7)	172(6)	-68(5)	22(5)	-33(5)
N(1)	145(7)	160(8)	118(7)	-37(6)	17(6)	-19(6)
N(2)	114(6)	172(7)	179(7)	-8(7)	49(5)	-8(7)
C(1)	213(9)	163(9)	222(9)	19(7)	58(7)	29(7)
C(2)	320(11)	212(10)	179(10)	9(7)	81(8)	21(8)
C(3)	303(11)	235(10)	166(9)	55(8)	-39(8)	-12(8)
C(4)	210(10)	209(9)	245(10)	51(7)	15(8)	34(7)
C(5)	206(9)	163(9)	211(9)	21(7)	51(7)	11(7)
C(6)	173(9)	129(8)	188(9)	26(7)	30(7)	-7(7)
C(7)	145(9)	172(9)	177(9)	1(7)	61(7)	-24(7)
C(8)	147(8)	168(9)	122(8)	-7(7)	29(7)	3(6)
C(9)	163(9)	231(10)	191(10)	7(7)	56(8)	-12(7)
C(10)	101(8)	191(9)	203(9)	1(7)	40(7)	25(6)
C(11)	162(9)	152(9)	163(9)	18(7)	37(7)	5(7)
C(12)	255(11)	336(12)	195(10)	28(9)	26(8)	100(9)
C(13)	142(9)	173(9)	159(9)	-1(7)	22(7)	-10(7)
C(14)	122(8)	194(9)	160(9)	4(7)	13(7)	-9(7)
C(15)	211(10)	226(10)	195(9)	32(7)	50(8)	28(8)
C(16)	147(9)	177(9)	205(9)	-6(7)	61(7)	28(7)
C(17)	270(11)	214(10)	214(10)	-45(8)	54(8)	14(8)
C(18)	203(9)	115(8)	205(9)	-25(7)	34(7)	2(7)
C(19)	185(10)	214(10)	276(10)	-27(8)	31(8)	23(7)
C(20)	245(10)	229(10)	273(11)	30(8)	-49(8)	37(8)
C(21)	358(11)	199(10)	190(10)	17(8)	6(8)	-15(8)
C(22)	276(11)	170(9)	289(11)	8(8)	109(9)	-45(8)
C(23)	182(9)	166(9)	229(10)	-4(8)	21(8)	-4(7)
O(4)	481(9)	246(7)	416(8)	-99(6)	187(7)	-173(7)
C(41)	382(12)	191(10)	219(10)	-15(8)	63(9)	27(8)
C(42)	289(11)	226(10)	235(11)	-7(8)	77(9)	-23(8)
C(43)	279(10)	199(10)	238(10)	-36(8)	38(8)	-20(8)
C(44)	275(12)	280(11)	298(12)	-4(9)	76(9)	-18(9)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for SPB01 (CCDC 205591).

	x	y	z	U_{iso}
H(2)	3880(30)	7118(12)	7299(19)	34(6)
H(1)	7210(20)	8915(10)	8687(16)	16(5)
H(2A)	4100(30)	10136(10)	9158(14)	21(5)
H(1A)	8000(30)	8606(9)	10728(15)	18(5)
H(2B)	10060(30)	8541(10)	12725(16)	20(5)
H(3)	13310(20)	8042(9)	13094(15)	13(4)
H(4)	14330(30)	7540(10)	11377(15)	21(5)
H(5)	12130(20)	7506(9)	9338(14)	11(4)
H(9A)	7530(30)	9328(10)	6155(16)	26(5)
H(9B)	6280(30)	8791(9)	5098(16)	16(4)
H(9C)	8130(30)	8502(9)	6144(15)	19(5)
H(11)	4940(20)	9642(9)	7230(14)	10(4)
H(12A)	3540(30)	9447(11)	5020(20)	46(6)
H(12B)	1630(30)	9634(10)	5664(15)	19(4)
H(12C)	2070(30)	8805(11)	5341(17)	29(5)
H(13A)	1870(20)	8603(9)	7563(14)	11(4)
H(13B)	3640(20)	8777(9)	8683(15)	9(4)
H(14A)	1200(20)	9922(9)	7474(14)	12(4)
H(14B)	390(30)	9490(8)	8480(14)	14(4)
H(15A)	1620(30)	9837(9)	10666(14)	13(4)
H(15B)	3910(30)	10074(11)	11117(15)	27(5)
H(15C)	3260(20)	9303(10)	10487(14)	13(4)
H(16)	670(30)	10821(9)	9149(15)	13(4)
H(17A)	4690(30)	11355(10)	10173(18)	35(6)
H(17B)	3080(30)	11220(10)	10946(18)	22(5)
H(17C)	2790(30)	11915(11)	9938(18)	36(6)
H(19)	-970(30)	11421(9)	7340(16)	21(5)
H(20)	-1010(30)	12007(10)	5448(17)	26(5)
H(21)	1970(30)	12077(10)	4723(17)	23(5)
H(22)	5010(30)	11625(10)	5951(17)	28(5)
H(23)	4920(30)	11042(9)	7833(15)	13(4)
H(41A)	230(30)	6479(11)	7290(17)	26(5)
H(41B)	1560(30)	5996(11)	8450(20)	40(6)
H(42A)	-590(30)	5500(11)	6049(18)	31(5)
H(42B)	-480(30)	5131(12)	7374(17)	40(6)
H(43A)	2720(30)	4762(10)	7487(17)	22(5)
H(43B)	1770(30)	4625(11)	6025(17)	25(5)
H(44A)	4680(30)	5498(11)	6547(16)	28(5)
H(44B)	2850(30)	5715(9)	5455(18)	20(5)

Table 6. Hydrogen bonds for SPB01 (CCDC 205591) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(2)-H(2)...O(4)	0.80(2)	1.84(2)	2.626(2)	170(2)
N(1)-H(1)...Cl(1)#1	0.84(2)	2.38(2)	3.204(2)	168(2)
N(2)-H(2A)...Cl(1)#1	0.88(2)	2.20(2)	3.060(1)	164(1)

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