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Synthesis and bioactivity of novel (γ-piperidono)dibenzo-33-aza-14-crown-3 ethers

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SUPPLEMENTARY INFORMATION

Table 1. Crystal data and structure refinement for compound **2b**.

Identification code	**2b**	
Empirical formula	$C_{42}H_{37}Cl_2NO_4$	
Formula weight	690.63	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 12.0602(15) Å	α = 90°.
	b = 25.981(3) Å	β = 117.033(3)°.
	c = 12.0826(14) Å	γ = 90°.
Volume	3372.3(7) Å^3	
Z	4	
Density (calculated)	1.360 mg/m^3	
Absorption coefficient	0.239 mm^{-1}	
F(000)	1448	
Crystal size	0.25 × 0.15 × 0.15 mm^3	
Theta range for data collection	3.019 to 25.672°.	
Index ranges	-13≤h≤14, –31≤k≤30, –14≤l≤13	
Reflections collected	34783	
Independent reflections	6323 [R(int) = 0.0579]	
Completeness to θ = 25.242°	99.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.954 and 0.936	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6323 / 6 / 454	
Goodness-of-fit on F^2	1.029	
Final R indices [for 4411 rflns with I>2σ(I)]	R_1 = 0.0540, wR_2 = 0.1216	
R indices (all data)	R_1 = 0.0892, wR_2 = 0.1374	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.467 and –0.635 e.Å^{-3}	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for compound **2b**. *U*(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	*U*(eq)
C(1)	3059(2)	3577(1)	5518(2)	17(1)
C(2)	3104(2)	4021(1)	6365(2)	17(1)
C(3)	4267(2)	4236(1)	7228(2)	19(1)
C(4)	5446(2)	4031(1)	7460(2)	22(1)
C(5)	6535(3)	4255(1)	8294(2)	28(1)
C(6)	6530(3)	4700(1)	8940(2)	32(1)
C(7)	5425(3)	4909(1)	8758(2)	28(1)
C(8)	4277(2)	4686(1)	7919(2)	22(1)
C(9)	3134(3)	4896(1)	7747(2)	23(1)
C(10)	2028(3)	4680(1)	6955(2)	22(1)
C(11)	2018(2)	4243(1)	6252(2)	19(1)
O(12)	932(2)	4021(1)	5402(2)	26(1)
C(13)	–239(2)	4222(1)	5210(2)	23(1)
C(14)	–1207(2)	3955(1)	4081(2)	23(1)
O(15)	–981(2)	4091(1)	3071(2)	23(1)
C(16)	–1539(2)	3761(1)	2030(2)	23(1)
C(17)	–910(2)	3851(1)	1226(2)	21(1)
O(18)	349(2)	3680(1)	1924(2)	22(1)
C(19)	1149(2)	3729(1)	1405(2)	18(1)
C(20)	747(2)	3932(1)	200(2)	23(1)
C(21)	1566(2)	3969(1)	–286(2)	23(1)
C(22)	2808(2)	3809(1)	390(2)	18(1)
C(23)	3643(2)	3839(1)	–139(2)	22(1)
C(24)	4847(2)	3689(1)	507(2)	24(1)
C(25)	5271(2)	3489(1)	1713(2)	27(1)
C(26)	4485(2)	3448(1)	2247(2)	25(1)
C(27)	3220(2)	3609(1)	1614(2)	18(1)
C(28)	2363(2)	3573(1)	2131(2)	16(1)
C(29)	2758(2)	3368(1)	3439(2)	17(1)
C(30)	2106(2)	2846(1)	3442(2)	17(1)
C(31)	2477(2)	2682(1)	4780(2)	19(1)
O(31)	2879(2)	2261(1)	5178(2)	36(1)
C(32)	2311(2)	3094(1)	5585(2)	17(1)
N(33)	2610(2)	3760(1)	4235(2)	18(1)
C(34)	2336(2)	2433(1)	2692(2)	17(1)

C(35)	3487(2)	2192(1)	3108(2)	23(1)
C(36)	3670(3)	1807(1)	2419(2)	27(1)
C(37)	2698(3)	1651(1)	1303(2)	28(1)
C(38)	1567(3)	1894(1)	870(2)	28(1)
C(39)	1385(2)	2286(1)	1547(2)	21(1)
C(40)	2649(2)	2913(1)	6887(2)	18(1)
C(41)	1722(3)	2846(1)	7257(2)	24(1)
C(42)	2014(3)	2671(1)	8451(2)	29(1)
C(43)	3228(3)	2563(1)	9280(2)	26(1)
C(44)	4159(3)	2630(1)	8926(2)	26(1)
C(45)	3870(2)	2803(1)	7738(2)	22(1)
Cl(1)	3844(2)	5422(1)	5169(3)	65(1)
Cl(2)	1565(2)	5056(1)	3059(2)	65(1)
C(46)	3199(5)	4971(5)	3932(11)	55(1)
Cl(1')	3429(4)	5320(2)	5001(5)	55(1)
Cl(2')	1339(4)	5124(1)	2546(3)	55(1)
C(46')	2898(14)	4960(11)	3601(16)	55(1)

Table 3. Bond lengths (Å) and angles (deg) for compound **2b**.

C(1)–N(33)	1.470(3)	C(19)–C(20)	1.410(3)
C(1)–C(2)	1.527(3)	C(20)–C(21)	1.362(4)
C(1)–C(32)	1.567(3)	C(20)–H(20)	0.9500
C(1)–H(1)	1.0000	C(21)–C(22)	1.404(4)
C(2)–C(11)	1.380(3)	C(21)–H(21)	0.9500
C(2)–C(3)	1.427(3)	C(22)–C(23)	1.419(3)
C(3)–C(4)	1.422(4)	C(22)–C(27)	1.427(3)
C(3)–C(8)	1.435(3)	C(23)–C(24)	1.357(4)
C(4)–C(5)	1.369(4)	C(23)–H(23)	0.9500
C(4)–H(4)	0.9500	C(24)–C(25)	1.406(4)
C(5)–C(6)	1.397(4)	C(24)–H(24)	0.9500
C(5)–H(5)	0.9500	C(25)–C(26)	1.372(4)
C(6)–C(7)	1.362(4)	C(25)–H(25)	0.9500
C(6)–H(6)	0.9500	C(26)–C(27)	1.423(3)
C(7)–C(8)	1.415(4)	C(26)–H(26)	0.9500
C(7)–H(7)	0.9500	C(27)–C(28)	1.433(3)
C(8)–C(9)	1.407(4)	C(28)–C(29)	1.524(3)
C(9)–C(10)	1.360(4)	C(29)–N(33)	1.466(3)
C(9)–H(9)	0.9500	C(29)–C(30)	1.570(3)
C(10)–C(11)	1.414(3)	C(29)–H(29)	1.0000
C(10)–H(10)	0.9500	C(30)–C(34)	1.508(3)
C(11)–O(12)	1.372(3)	C(30)–C(31)	1.528(3)
O(12)–C(13)	1.423(3)	C(30)–H(30)	1.0000
C(13)–C(14)	1.502(4)	C(31)–O(31)	1.206(3)
C(13)–H(13A)	0.9900	C(31)–C(32)	1.521(3)
C(13)–H(13B)	0.9900	C(32)–C(40)	1.510(3)
C(14)–O(15)	1.411(3)	C(32)–H(32)	1.0000
C(14)–H(14A)	0.9900	N(33)–H(33)	0.86(3)
C(14)–H(14B)	0.9900	C(34)–C(39)	1.391(3)
O(15)–C(16)	1.415(3)	C(34)–C(35)	1.392(3)
C(16)–C(17)	1.497(3)	C(35)–C(36)	1.381(4)
C(16)–H(16A)	0.9900	C(35)–H(35)	0.9500
C(16)–H(16B)	0.9900	C(36)–C(37)	1.386(4)
C(17)–O(18)	1.432(3)	C(36)–H(36)	0.9500
C(17)–H(17A)	0.9900	C(37)–C(38)	1.373(4)
C(17)–H(17B)	0.9900	C(37)–H(37)	0.9500
O(18)–C(19)	1.375(3)	C(38)–C(39)	1.383(4)
C(19)–C(28)	1.382(3)	C(38)–H(38)	0.9500

C(39)–H(39)	0.9500	C(44)–H(44)	0.9500
C(40)–C(45)	1.389(3)	C(45)–H(45)	0.9500
C(40)–C(41)	1.391(4)	Cl(1)–C(46)	1.776(3)
C(41)–C(42)	1.397(4)	Cl(2)–C(46)	1.776(3)
C(41)–H(41)	0.9500	C(46)–H(46A)	0.9900
C(42)–C(43)	1.376(4)	C(46)–H(46B)	0.9900
C(42)–H(42)	0.9500	Cl(1')–C(46')	1.778(3)
C(43)–C(44)	1.383(4)	Cl(2')–C(46')	1.778(3)
C(43)–H(43)	0.9500	C(46')–H(46C)	0.9900
C(44)–C(45)	1.389(3)	C(46')–H(46D)	0.9900

N(33)–C(1)–C(2)	110.23(19)	C(8)–C(9)–H(9)	119.1
N(33)–C(1)–C(32)	111.68(19)	C(9)–C(10)–C(11)	119.4(2)
C(2)–C(1)–C(32)	114.53(19)	C(9)–C(10)–H(10)	120.3
N(33)–C(1)–H(1)	106.6	C(11)–C(10)–H(10)	120.3
C(2)–C(1)–H(1)	106.6	O(12)–C(11)–C(2)	116.0(2)
C(32)–C(1)–H(1)	106.6	O(12)–C(11)–C(10)	122.1(2)
C(11)–C(2)–C(3)	118.8(2)	C(2)–C(11)–C(10)	121.9(2)
C(11)–C(2)–C(1)	120.5(2)	C(11)–O(12)–C(13)	120.46(19)
C(3)–C(2)–C(1)	120.6(2)	O(12)–C(13)–C(14)	106.4(2)
C(4)–C(3)–C(2)	124.1(2)	O(12)–C(13)–H(13A)	110.5
C(4)–C(3)–C(8)	116.6(2)	C(14)–C(13)–H(13A)	110.5
C(2)–C(3)–C(8)	119.3(2)	O(12)–C(13)–H(13B)	110.5
C(5)–C(4)–C(3)	121.7(2)	C(14)–C(13)–H(13B)	110.5
C(5)–C(4)–H(4)	119.1	H(13A)–C(13)–H(13B)	108.6
C(3)–C(4)–H(4)	119.1	O(15)–C(14)–C(13)	107.1(2)
C(4)–C(5)–C(6)	121.1(3)	O(15)–C(14)–H(14A)	110.3
C(4)–C(5)–H(5)	119.5	C(13)–C(14)–H(14A)	110.3
C(6)–C(5)–H(5)	119.5	O(15)–C(14)–H(14B)	110.3
C(7)–C(6)–C(5)	119.5(3)	C(13)–C(14)–H(14B)	110.3
C(7)–C(6)–H(6)	120.3	H(14A)–C(14)–H(14B)	108.6
C(5)–C(6)–H(6)	120.3	C(14)–O(15)–C(16)	114.60(19)
C(6)–C(7)–C(8)	121.3(2)	O(15)–C(16)–C(17)	107.8(2)
C(6)–C(7)–H(7)	119.3	O(15)–C(16)–H(16A)	110.1
C(8)–C(7)–H(7)	119.3	C(17)–C(16)–H(16A)	110.1
C(9)–C(8)–C(7)	121.4(2)	O(15)–C(16)–H(16B)	110.1
C(9)–C(8)–C(3)	118.8(2)	C(17)–C(16)–H(16B)	110.1
C(7)–C(8)–C(3)	119.8(2)	H(16A)–C(16)–H(16B)	108.5
C(10)–C(9)–C(8)	121.8(2)	O(18)–C(17)–C(16)	106.50(19)
C(10)–C(9)–H(9)	119.1	O(18)–C(17)–H(17A)	110.4

C(16)–C(17)–H(17A)	110.4	C(30)–C(29)–H(29)	106.5
O(18)–C(17)–H(17B)	110.4	C(34)–C(30)–C(31)	113.2(2)
C(16)–C(17)–H(17B)	110.4	C(34)–C(30)–C(29)	112.56(19)
H(17A)–C(17)–H(17B)	108.6	C(31)–C(30)–C(29)	109.66(18)
C(19)–O(18)–C(17)	118.59(18)	C(34)–C(30)–H(30)	107.0
O(18)–C(19)–C(28)	116.3(2)	C(31)–C(30)–H(30)	107.0
O(18)–C(19)–C(20)	121.5(2)	C(29)–C(30)–H(30)	107.0
C(28)–C(19)–C(20)	122.2(2)	O(31)–C(31)–C(32)	122.0(2)
C(21)–C(20)–C(19)	119.6(2)	O(31)–C(31)–C(30)	123.1(2)
C(21)–C(20)–H(20)	120.2	C(32)–C(31)–C(30)	114.9(2)
C(19)–C(20)–H(20)	120.2	C(40)–C(32)–C(31)	113.2(2)
C(20)–C(21)–C(22)	121.3(2)	C(40)–C(32)–C(1)	114.52(19)
C(20)–C(21)–H(21)	119.4	C(31)–C(32)–C(1)	106.47(19)
C(22)–C(21)–H(21)	119.4	C(40)–C(32)–H(32)	107.4
C(21)–C(22)–C(23)	120.7(2)	C(31)–C(32)–H(32)	107.4
C(21)–C(22)–C(27)	119.2(2)	C(1)–C(32)–H(32)	107.4
C(23)–C(22)–C(27)	120.2(2)	C(29)–N(33)–C(1)	111.75(19)
C(24)–C(23)–C(22)	121.2(2)	C(29)–N(33)–H(33)	109.1(18)
C(24)–C(23)–H(23)	119.4	C(1)–N(33)–H(33)	109.7(17)
C(22)–C(23)–H(23)	119.4	C(39)–C(34)–C(35)	118.1(2)
C(23)–C(24)–C(25)	119.5(2)	C(39)–C(34)–C(30)	120.0(2)
C(23)–C(24)–H(24)	120.3	C(35)–C(34)–C(30)	121.9(2)
C(25)–C(24)–H(24)	120.3	C(36)–C(35)–C(34)	121.0(2)
C(26)–C(25)–C(24)	120.9(2)	C(36)–C(35)–H(35)	119.5
C(26)–C(25)–H(25)	119.6	C(34)–C(35)–H(35)	119.5
C(24)–C(25)–H(25)	119.6	C(35)–C(36)–C(37)	120.1(3)
C(25)–C(26)–C(27)	121.7(2)	C(35)–C(36)–H(36)	119.9
C(25)–C(26)–H(26)	119.2	C(37)–C(36)–H(36)	119.9
C(27)–C(26)–H(26)	119.2	C(38)–C(37)–C(36)	119.4(2)
C(26)–C(27)–C(22)	116.6(2)	C(38)–C(37)–H(37)	120.3
C(26)–C(27)–C(28)	123.8(2)	C(36)–C(37)–H(37)	120.3
C(22)–C(27)–C(28)	119.7(2)	C(37)–C(38)–C(39)	120.6(2)
C(19)–C(28)–C(27)	118.0(2)	C(37)–C(38)–H(38)	119.7
C(19)–C(28)–C(29)	120.2(2)	C(39)–C(38)–H(38)	119.7
C(27)–C(28)–C(29)	121.8(2)	C(38)–C(39)–C(34)	120.7(2)
N(33)–C(29)–C(28)	111.35(19)	C(38)–C(39)–H(39)	119.6
N(33)–C(29)–C(30)	112.90(19)	C(34)–C(39)–H(39)	119.6
C(28)–C(29)–C(30)	112.57(19)	C(45)–C(40)–C(41)	118.3(2)
N(33)–C(29)–H(29)	106.5	C(45)–C(40)–C(32)	121.9(2)
C(28)–C(29)–H(29)	106.5	C(41)–C(40)–C(32)	119.8(2)

C(40)–C(41)–C(42)	120.7(3)	C(44)–C(45)–H(45)	119.5
C(40)–C(41)–H(41)	119.6	Cl(2)–C(46)–Cl(1)	111.2(2)
C(42)–C(41)–H(41)	119.6	Cl(2)–C(46)–H(46A)	109.4
C(43)–C(42)–C(41)	120.2(3)	Cl(1)–C(46)–H(46A)	109.4
C(43)–C(42)–H(42)	119.9	Cl(2)–C(46)–H(46B)	109.4
C(41)–C(42)–H(42)	119.9	Cl(1)–C(46)–H(46B)	109.4
C(42)–C(43)–C(44)	119.6(2)	H(46A)–C(46)–H(46B)	108.0
C(42)–C(43)–H(43)	120.2	Cl(2')–C(46')–Cl(1')	111.2(2)
C(44)–C(43)–H(43)	120.2	Cl(2')–C(46')–H(46C)	109.4
C(43)–C(44)–C(45)	120.2(3)	Cl(1')–C(46')–H(46C)	109.4
C(43)–C(44)–H(44)	119.9	Cl(2')–C(46')–H(46D)	109.4
C(45)–C(44)–H(44)	119.9	Cl(1')–C(46')–H(46D)	109.4
C(40)–C(45)–C(44)	121.0(2)	H(46C)–C(46')–H(46D)	108.0
C(40)–C(45)–H(45)	119.5		

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for compound **2b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	19(1)	17(1)	16(1)	2(1)	8(1)	1(1)
C(2)	25(1)	14(1)	14(1)	1(1)	10(1)	1(1)
C(3)	25(1)	18(1)	13(1)	4(1)	10(1)	0(1)
C(4)	24(2)	20(1)	21(1)	–1(1)	11(1)	–2(1)
C(5)	22(2)	31(2)	28(1)	–1(1)	10(1)	–1(1)
C(6)	29(2)	36(2)	24(1)	–6(1)	6(1)	–9(1)
C(7)	37(2)	23(2)	22(1)	–7(1)	12(1)	–7(1)
C(8)	31(2)	17(1)	17(1)	1(1)	11(1)	–1(1)
C(9)	35(2)	16(1)	19(1)	–1(1)	13(1)	2(1)
C(10)	30(2)	19(1)	20(1)	4(1)	14(1)	8(1)
C(11)	24(1)	18(1)	15(1)	2(1)	8(1)	0(1)
O(12)	18(1)	24(1)	30(1)	–7(1)	6(1)	3(1)
C(13)	24(1)	21(1)	27(1)	1(1)	14(1)	3(1)
C(14)	20(1)	25(1)	28(1)	4(1)	14(1)	2(1)
O(15)	24(1)	22(1)	25(1)	–1(1)	14(1)	–1(1)
C(16)	16(1)	25(1)	25(1)	–2(1)	7(1)	–2(1)
C(17)	17(1)	23(1)	20(1)	2(1)	6(1)	2(1)
O(18)	16(1)	31(1)	22(1)	8(1)	10(1)	7(1)
C(19)	21(1)	19(1)	18(1)	0(1)	10(1)	1(1)
C(20)	20(1)	26(2)	20(1)	3(1)	6(1)	4(1)
C(21)	27(2)	25(1)	14(1)	4(1)	7(1)	0(1)
C(22)	22(1)	16(1)	17(1)	–2(1)	9(1)	–4(1)
C(23)	30(2)	21(1)	17(1)	0(1)	13(1)	–4(1)
C(24)	25(2)	27(2)	26(1)	–4(1)	16(1)	–7(1)
C(25)	19(1)	38(2)	24(1)	2(1)	10(1)	–1(1)
C(26)	22(2)	33(2)	21(1)	5(1)	9(1)	0(1)
C(27)	21(1)	15(1)	16(1)	–2(1)	8(1)	–2(1)
C(28)	20(1)	15(1)	16(1)	–1(1)	8(1)	0(1)
C(29)	17(1)	17(1)	17(1)	0(1)	8(1)	1(1)
C(30)	16(1)	18(1)	16(1)	–2(1)	6(1)	0(1)
C(31)	21(1)	15(1)	21(1)	–1(1)	10(1)	–2(1)
O(31)	68(2)	19(1)	26(1)	5(1)	26(1)	14(1)
C(32)	19(1)	16(1)	17(1)	1(1)	9(1)	2(1)
N(33)	23(1)	17(1)	13(1)	1(1)	7(1)	2(1)
C(34)	21(1)	15(1)	17(1)	1(1)	10(1)	–2(1)

C(35)	24(2)	25(1)	19(1)	0(1)	7(1)	2(1)
C(36)	32(2)	26(2)	28(1)	3(1)	19(1)	6(1)
C(37)	42(2)	23(1)	29(1)	–8(1)	25(1)	–7(1)
C(38)	30(2)	38(2)	20(1)	–10(1)	13(1)	–15(1)
C(39)	18(1)	27(1)	19(1)	–1(1)	9(1)	–4(1)
C(40)	26(1)	13(1)	20(1)	–2(1)	13(1)	–1(1)
C(41)	27(2)	25(2)	21(1)	–1(1)	12(1)	–4(1)
C(42)	37(2)	31(2)	27(1)	–3(1)	21(1)	–8(1)
C(43)	42(2)	20(1)	18(1)	4(1)	15(1)	1(1)
C(44)	32(2)	24(1)	22(1)	2(1)	11(1)	6(1)
C(45)	26(2)	21(1)	20(1)	4(1)	13(1)	5(1)
Cl(1)	82(1)	50(1)	85(1)	12(1)	57(1)	14(1)
Cl(2)	82(1)	50(1)	85(1)	12(1)	57(1)	14(1)
C(46)	75(5)	34(2)	80(6)	12(4)	57(3)	13(4)
Cl(1')	83(2)	46(1)	55(1)	15(1)	49(2)	10(1)
Cl(2')	83(2)	46(1)	55(1)	15(1)	49(2)	10(1)
C(46')	75(5)	34(2)	80(6)	12(4)	57(3)	13(4)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for compound **2b**.

Atom	*x*	*y*	*z*	*U*(iso)
H(1)	3938	3461	5804	21
H(4)	5480	3732	7025	26
H(5)	7307	4104	8436	33
H(6)	7293	4856	9503	38
H(7)	5424	5211	9202	34
H(9)	3135	5196	8196	28
H(10)	1268	4821	6875	27
H(13A)	-270	4599	5071	27
H(13B)	-381	4154	5942	27
H(14A)	-1143	3577	4205	28
H(14B)	-2051	4065	3924	28
H(16A)	-2440	3836	1560	28
H(16B)	-1438	3397	2304	28
H(17A)	-1332	3654	442	25
H(17B)	-932	4221	1022	25
H(20)	-90	4042	-270	28
H(21)	1291	4105	-1098	27
H(23)	3353	3966	-961	26
H(24)	5400	3719	148	29
H(25)	6112	3381	2163	33
H(26)	4793	3310	3061	30
H(29)	3669	3294	3801	20
H(30)	1189	2912	3037	21
H(32)	1410	3191	5187	20
H(33)	1830(30)	3844(10)	3930(20)	22
H(35)	4156	2292	3878	28
H(36)	4464	1650	2710	32
H(37)	2814	1378	842	33
H(38)	902	1793	97	34
H(39)	602	2455	1227	25
H(41)	881	2919	6690	29
H(42)	1372	2627	8692	35
H(43)	3426	2442	10091	31
H(44)	4999	2557	9496	31
H(45)	4517	2848	7504	26
H(46A)	3596	5015	3379	66

H(46B)	3375	4618	4273	66
H(46C)	3446	5028	3208	66
H(46D)	2945	4588	3795	66

Table 6. Torsion angles (deg) for compound **2b**.

N(33)–C(1)–C(2)–C(11)	68.7(3)	O(18)–C(19)–C(20)–C(21)	–179.2(2)
C(32)–C(1)–C(2)–C(11)	–58.2(3)	C(28)–C(19)–C(20)–C(21)	1.1(4)
N(33)–C(1)–C(2)–C(3)	–107.1(2)	C(19)–C(20)–C(21)–C(22)	0.0(4)
C(32)–C(1)–C(2)–C(3)	126.0(2)	C(20)–C(21)–C(22)–C(23)	178.6(2)
C(11)–C(2)–C(3)–C(4)	177.7(2)	C(20)–C(21)–C(22)–C(27)	–0.5(4)
C(1)–C(2)–C(3)–C(4)	–6.4(3)	C(21)–C(22)–C(23)–C(24)	179.6(2)
C(11)–C(2)–C(3)–C(8)	–2.4(3)	C(27)–C(22)–C(23)–C(24)	–1.3(4)
C(1)–C(2)–C(3)–C(8)	173.5(2)	C(22)–C(23)–C(24)–C(25)	1.5(4)
C(2)–C(3)–C(4)–C(5)	179.6(2)	C(23)–C(24)–C(25)–C(26)	–0.6(4)
C(8)–C(3)–C(4)–C(5)	–0.3(3)	C(24)–C(25)–C(26)–C(27)	–0.5(4)
C(3)–C(4)–C(5)–C(6)	–1.0(4)	C(25)–C(26)–C(27)–C(22)	0.6(4)
C(4)–C(5)–C(6)–C(7)	1.4(4)	C(25)–C(26)–C(27)–C(28)	179.9(2)
C(5)–C(6)–C(7)–C(8)	–0.4(4)	C(21)–C(22)–C(27)–C(26)	179.4(2)
C(6)–C(7)–C(8)–C(9)	178.7(2)	C(23)–C(22)–C(27)–C(26)	0.3(3)
C(6)–C(7)–C(8)–C(3)	–0.9(4)	C(21)–C(22)–C(27)–C(28)	0.1(3)
C(4)–C(3)–C(8)–C(9)	–178.4(2)	C(23)–C(22)–C(27)–C(28)	–179.0(2)
C(2)–C(3)–C(8)–C(9)	1.8(3)	O(18)–C(19)–C(28)–C(27)	178.7(2)
C(4)–C(3)–C(8)–C(7)	1.3(3)	C(20)–C(19)–C(28)–C(27)	–1.6(4)
C(2)–C(3)–C(8)–C(7)	–178.6(2)	O(18)–C(19)–C(28)–C(29)	–1.1(3)
C(7)–C(8)–C(9)–C(10)	–179.1(2)	C(20)–C(19)–C(28)–C(29)	178.6(2)
C(3)–C(8)–C(9)–C(10)	0.5(4)	C(26)–C(27)–C(28)–C(19)	–178.3(2)
C(8)–C(9)–C(10)–C(11)	–2.1(4)	C(22)–C(27)–C(28)–C(19)	1.0(3)
C(3)–C(2)–C(11)–O(12)	179.7(2)	C(26)–C(27)–C(28)–C(29)	1.5(4)
C(1)–C(2)–C(11)–O(12)	3.8(3)	C(22)–C(27)–C(28)–C(29)	–179.2(2)
C(3)–C(2)–C(11)–C(10)	0.8(3)	C(19)–C(28)–C(29)–N(33)	–61.8(3)
C(1)–C(2)–C(11)–C(10)	–175.1(2)	C(27)–C(28)–C(29)–N(33)	118.3(2)
C(9)–C(10)–C(11)–O(12)	–177.3(2)	C(19)–C(28)–C(29)–C(30)	66.1(3)
C(9)–C(10)–C(11)–C(2)	1.5(4)	C(27)–C(28)–C(29)–C(30)	–113.7(2)
C(2)–C(11)–O(12)–C(13)	–179.7(2)	N(33)–C(29)–C(30)–C(34)	–176.04(19)
C(10)–C(11)–O(12)–C(13)	–0.8(3)	C(28)–C(29)–C(30)–C(34)	56.8(3)
C(11)–O(12)–C(13)–C(14)	170.2(2)	N(33)–C(29)–C(30)–C(31)	–49.1(3)
O(12)–C(13)–C(14)–O(15)	–63.0(2)	C(28)–C(29)–C(30)–C(31)	–176.3(2)
C(13)–C(14)–O(15)–C(16)	161.6(2)	C(34)–C(30)–C(31)–O(31)	–2.0(4)
C(14)–O(15)–C(16)–C(17)	–162.7(2)	C(29)–C(30)–C(31)–O(31)	–128.6(3)
O(15)–C(16)–C(17)–O(18)	66.0(2)	C(34)–C(30)–C(31)–C(32)	177.5(2)
C(16)–C(17)–O(18)–C(19)	–178.6(2)	C(29)–C(30)–C(31)–C(32)	51.0(3)
C(17)–O(18)–C(19)–C(28)	178.2(2)	O(31)–C(31)–C(32)–C(40)	–2.2(3)
C(17)–O(18)–C(19)–C(20)	–1.6(3)	C(30)–C(31)–C(32)–C(40)	178.2(2)

O(31)–C(31)–C(32)–C(1)	124.5(3)	C(35)–C(36)–C(37)–C(38)	2.3(4)
C(30)–C(31)–C(32)–C(1)	–55.1(3)	C(36)–C(37)–C(38)–C(39)	–1.2(4)
N(33)–C(1)–C(32)–C(40)	–175.0(2)	C(37)–C(38)–C(39)–C(34)	–1.3(4)
C(2)–C(1)–C(32)–C(40)	–48.9(3)	C(35)–C(34)–C(39)–C(38)	2.6(4)
N(33)–C(1)–C(32)–C(31)	59.0(2)	C(30)–C(34)–C(39)–C(38)	–177.5(2)
C(2)–C(1)–C(32)–C(31)	–174.81(19)	C(31)–C(32)–C(40)–C(45)	68.8(3)
C(28)–C(29)–N(33)–C(1)	–176.6(2)	C(1)–C(32)–C(40)–C(45)	–53.6(3)
C(30)–C(29)–N(33)–C(1)	55.7(3)	C(31)–C(32)–C(40)–C(41)	–110.5(3)
C(2)–C(1)–N(33)–C(29)	170.1(2)	C(1)–C(32)–C(40)–C(41)	127.1(2)
C(32)–C(1)–N(33)–C(29)	–61.4(3)	C(45)–C(40)–C(41)–C(42)	–0.2(4)
C(31)–C(30)–C(34)–C(39)	128.1(2)	C(32)–C(40)–C(41)–C(42)	179.1(2)
C(29)–C(30)–C(34)–C(39)	–106.9(3)	C(40)–C(41)–C(42)–C(43)	0.0(4)
C(31)–C(30)–C(34)–C(35)	–52.0(3)	C(41)–C(42)–C(43)–C(44)	0.3(4)
C(29)–C(30)–C(34)–C(35)	73.0(3)	C(42)–C(43)–C(44)–C(45)	–0.3(4)
C(39)–C(34)–C(35)–C(36)	–1.5(4)	C(41)–C(40)–C(45)–C(44)	0.2(4)
C(30)–C(34)–C(35)–C(36)	178.6(2)	C(32)–C(40)–C(45)–C(44)	–179.1(2)
C(34)–C(35)–C(36)–C(37)	–0.9(4)	C(43)–C(44)–C(45)–C(40)	0.1(4)

Table 7. Hydrogen bonds for compound **2b** (Å and deg).

D–H···A	*d*(D–H)	*d*(H···A)	*d*(D···A)	<(DHA)
C(17)–H(17A)···O(31)#1	0.99	2.52	3.223(3)	127
N(33)–H(33)···O(12)	0.86(3)	2.51(3)	3.023(3)	119(2)
N(33)–H(33)···O(18)	0.86(3)	2.31(3)	2.890(3)	125(2)
C(46)–H(46B)···N(33)	0.99	2.40	3.283(17)	148
C(46')–H(46D)···N(33)	0.99	2.29	3.27(4)	167

Symmetry transformations used to generate equivalent atoms:

#1 *x*–1/2, –*y*+1/2, *z*–1/2