Heteroaromatic and aniline derivatives of piperidines as potent ligands for vesicular acetylcholine transporter

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Contents of Supporting Information:

Table 2. Elemental Analysis of Analogues

Compounds	Molecular Formulas	Calculated			Found		
		C	H	N	C	H	N
19a	$C_{21}H_{26}N_2O_2 \bullet H_2C_2O_4$	64.47	6.59	6.54	64.26	6.62	6.41
19b	$C_{21}H_{26}N_2O_2 \bullet H_2C_2O_4 \bullet 0.25H_2O$	63.80	6.63	6.47	63.72	6.43	6.38
19c	$C_{21}H_{24}N_2O_2 \bullet H_2C_2O_4 \bullet H_2O$	62.15	6.35	6.30	62.19	6.25	6.29
19d	$C_{22}H_{26}N_2O_2 \bullet 2H_2C_2O_4$	58.86	5.70	5.28	58.35	5.87	4.82
19e	$C_{22}H_{26}N_2O_3 \bullet H_2C_2O_4$	63.15	6.18	6.14	63.06	6.18	6.05
19f	$C_{22}H_{26}N_2O_3 \bullet H_2C_2O_4 \bullet 0.25H_2O$	62.53	6.23	6.08	62.56	6.16	6.04
19g	$C_{21}H_{23}FN_2O_2 \bullet H_2C_2O_4$	62.15	5.67	6.30	61.87	5.53	6.26
19h	$C_{22}H_{26}N_2O_3 \bullet H_2C_2O_4 \bullet 1.5H_2O$	59.62	6.46	5.79	59.71	6.38	5.74
19i	$C_{22}H_{26}N_2O_3 \bullet H_2C_2O_4 \bullet 1.5H_2O$	59.62	6.46	5.79	60.08	6.59	5.72
19j	$C_{21}H_{23}FN_2O_2 \bullet H_2C_2O_4 \bullet 0.5H_2O$	60.92	5.78	6.18	60.62	5.67	6.09
19k	$C_{21}H_{23}FN_2O_2 \bullet H_2C_2O_4 \bullet H_2O$	59.73	5.88	6.06	59.97	5.61	5.80
24a	$C_{24}H_{30}N_2O_2 \bullet H_2C_2O_4$	66.65	6.88	5.98	66.80	6.95	5.92
24b	$C_{23}H_{28}N_2O_2 \bullet H_2C_2O_4$	66.06	6.65	6.16	65.84	6.69	6.14
26a	$C_{24}H_{27}FN_2O_3 \bullet H_2C_2O_4 \bullet H_2O$	60.22	6.03	5.40	60.72	5.98	5.50
26b	$C_{23}H_{27}FN_2O_3 \bullet H_2C_2O_4 \bullet H_2O$	59.28	6.17	5.53	59.14	6.45	5.30
30a	$C_{21}H_{27}N_3O_2 \bullet 2H_2C_2O_4 \bullet 2H_2O$	52.72	6.19	7.38	52.97	5.94	7.21
30c	$C_{21}H_{25}N_3O_2 \bullet 2H_2C_2O_4 \bullet 0.5H_2O$	55.55	5.59	7.77	55.86	5.88	8.04
30d	$C_{24}H_{29}N_3O_4 \bullet H_2C_2O_4$	60.81	6.08	8.18	60.57	6.06	7.96
30e	$C_{22}H_{27}N_3O_3 \bullet 2H_2C_2O_4 \bullet 1.5H_2O$	53.06	5.82	7.14	52.78	5.53	6.86
31a	$C_{21}H_{27}N_3O_2 \bullet 2H_2C_2O_4 \bullet 1.5H_2O$	53.57	6.11	7.50	53.53	6.38	7.64

II. X-Ray Diffraction study of 19i.

Crystals of appropriate dimension were obtained by slow evaporation of methanol solutions at 8°C. A crystal with approximate dimensions 0.31 x 0.24 x 0.03 mm^3 was mounted on glass fibers in a random orientation. Preliminary examination and data collection were performed using a Bruker Kappa Apex II Charge Coupled Device (CCD) Detector system single crystal X-Ray diffractometer equipped with an Oxford Cryostream LT device. All data were collected using graphite monochromated Mo Kα radiation (λ= 0.71073 Å) from a fine focus sealed tube X-Ray source. Preliminary unit cell constants were determined with a set of 36 narrow frame scans. Intensity data set included combinations of ϖ and Φ scan frames with scan width of 0.5° and counting time of 20 seconds/frame at a crystal to detector distance of 4.0 cm. The collected frames were integrated using an orientation matrix determined from the narrow frame scans. Apex II and SAINT software packages (Bruker Analytical X-Ray, Madison, WI, 2006) were used for data collection and data integration. Analysis of the integrated data did not show any decay. Final cell constants were determined by global refinement of xyz centroids of 1393 reflections from the complete data set. Collected data were corrected for systematic errors using SADABS (Blessing, R. H., Acta Cryst. (1995), A51, 33-38) based on the Laue symmetry using equivalent reflections.

Crystal data and intensity data collection parameters are listed in supporting material (Table 3).

Structure solution and refinement were carried out using the SHELXTL- PLUS software package (Sheldrick, G. M., Bruker Analytical X-Ray Division, Madison, WI, 2006). The structure was solved by direct methods and refined successfully in the space group, P21. Full matrix least-squares refinement was carried out by minimizing $\Sigma w(Fo^2-Fc^2)^2$. The non-hydrogen atoms were refined anisotropically to convergence. All hydrogen atoms were treated using appropriate riding model (AFIX m^3). The final residual values and structure refinement parameters are available via the Internet with the supporting information (Table 3).

Complete listings of positional and isotropic displacement coefficients for hydrogen atoms, anisotropic displacement coefficients for the non-hydrogen atoms are available via the Internet with the supporting information (Tables 4-8).

Table 3. Crystal data and structure refinement for **19i**.

Identification code	z8611t5/0 °C/smart/TZ-4-1-148	
Empirical formula	$C_{25}\ H_{32}\ N_2\ O_8$	
Formula weight	488.53	
Temperature	272(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 5.6706(2) Å	α= 98.021(3)°.
	b = 9.0857(3) Å	β= 96.230(3)°.
	c = 25.8454(12) Å	γ = 98.012(2)°.
Volume	1294.53(9) $Å^3$	
Z	2	
Density (calculated)	1.253 Mg/m^3	
Absorption coefficient	0.094 mm^{-1}	
F(000)	520	
Crystal size	0.31 x 0.24 x 0.03 mm^3	
Theta range for data collection	1.61 to 25.38°.	
Index ranges	-6≤h≤6, -10≤k≤10, -10≤l≤31	
Reflections collected	36759	
Independent reflections	4672 [R(int) = 0.0650]	
Completeness to theta = 25.00°	98.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9970 and 0.9713	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4672 / 20 / 411	

Goodness-of-fit on F^2	1.043
Final R indices [I>2sigma(I)]	R1 = 0.0637, wR2 = 0.1450
R indices (all data)	R1 = 0.1172, wR2 = 0.1723
Largest diff. peak and hole	0.446 and -0.200 e.$Å^{-3}$

Table 4. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **19i**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	8003(5)	7787(3)	8213(1)	52(1)
O(2)	-280(8)	3170(5)	6322(2)	103(1)
N(1)	4070(5)	5345(3)	7968(1)	33(1)
C(1)	2759(11)	6332(6)	9844(2)	82(2)
C(2)	3381(14)	7127(8)	10359(2)	103(2)
C(3)	5266(14)	8235(8)	10468(2)	101(2)
C(4)	6657(11)	8550(7)	10082(2)	84(2)
C(5)	6096(8)	7781(5)	9564(2)	54(1)
C(6)	7592(7)	8152(5)	9146(2)	56(1)
C(7)	6389(6)	7648(4)	8592(2)	41(1)
C(8)	5199(6)	6020(4)	8528(2)	36(1)
C(9)	3349(8)	5804(5)	8899(2)	55(1)
C(10)	4116(8)	6660(5)	9450(2)	50(1)
C(11)	5845(6)	4823(4)	7625(2)	36(1)
C(12)	4574(7)	3915(4)	7113(2)	46(1)
C(13)	2994(7)	4841(5)	6812(2)	51(1)
C(14)	1314(7)	5467(5)	7176(2)	48(1)
C(15)	2627(6)	6346(4)	7694(2)	37(1)
C(16)	1635(9)	3880(6)	6332(2)	68(1)
O(3)	1430(20)	2271(13)	4492(3)	156(5)
C(17)	2043(16)	4337(10)	5771(3)	58(2)
C(18)	2330(20)	5768(12)	5678(4)	84(3)

C(19)	2210(20)	6036(16)	5143(6)	112(5)
C(20)	1910(30)	4823(19)	4757(4)	108(5)
C(21)	1740(20)	3350(16)	4850(4)	90(4)
N(2)	1880(16)	3173(9)	5369(3)	71(2)
C(22)	1860(60)	1540(30)	5485(12)	140(14)
O(3')	3250(20)	-249(12)	5084(5)	157(5)
C(17')	3105(18)	3144(12)	5928(4)	70(3)
C(18')	4731(19)	4135(13)	5756(4)	72(3)
C(19')	5900(20)	3643(17)	5321(5)	91(4)
C(20')	5290(30)	2176(19)	5077(5)	107(5)
C(21')	3690(30)	1113(15)	5262(5)	95(4)
N(2')	2610(40)	1703(19)	5680(9)	94(6)
C(22')	950(30)	519(16)	5920(7)	139(6)
O(4)	2393(8)	-229(4)	8126(6)	74(3)
O(4')	2270(40)	-70(20)	7841(12)	74(3)
O(5)	4372(4)	2088(3)	8188(1)	44(1)
O(6)	216(4)	3130(3)	8054(1)	49(1)
O(7)	-1697(4)	971(3)	8217(1)	50(1)
C(23)	2430(6)	1108(4)	8126(2)	40(1)
C(24)	118(5)	1822(4)	8131(1)	33(1)
C(1S)	7680(40)	9150(20)	6838(6)	179(9)
O(1S)	7670(30)	7706(16)	6527(6)	225(9)
O(1S')	4560(60)	8410(30)	6354(9)	360(20)
C(1S')	5760(70)	9630(40)	6759(13)	360(20)

Table 5. Bond lengths [Å] and angles [°] for **19i**.

O(1)-C(7)	1.419(4)
O(1)-H(1)	0.8200
O(2)-C(16)	1.179(6)
N(1)-C(11)	1.495(4)
N(1)-C(15)	1.503(4)
N(1)-C(8)	1.525(5)
N(1)-H(1B)	0.9100
C(1)-C(10)	1.382(6)
C(1)-C(2)	1.404(9)
C(1)-H(1A)	0.9300
C(2)-C(3)	1.339(9)
C(2)-H(2A)	0.9300
C(3)-C(4)	1.373(8)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.402(7)
C(4)-H(4A)	0.9300
C(5)-C(10)	1.382(6)
C(5)-C(6)	1.491(6)
C(6)-C(7)	1.497(6)
C(6)-H(6A)	0.9700
C(6)-H(6B)	0.9700
C(7)-C(8)	1.517(5)
C(7)-H(7A)	0.9800
C(8)-C(9)	1.506(5)
C(8)-H(8A)	0.9800

C(9)-C(10)	1.512(6)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(11)-C(12)	1.506(6)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(12)-C(13)	1.536(6)
C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700
C(13)-C(16)	1.482(7)
C(13)-C(14)	1.521(5)
C(13)-H(13A)	0.9800
C(14)-C(15)	1.518(6)
C(14)-H(14A)	0.9700
C(14)-H(14B)	0.9700
C(15)-H(15A)	0.9700
C(15)-H(15B)	0.9700
C(16)-C(17')	1.544(11)
C(16)-C(17)	1.594(10)
O(3)-C(21)	1.230(13)
C(17)-C(18)	1.346(12)
C(17)-N(2)	1.361(11)
C(18)-C(19)	1.433(14)
C(18)-H(18A)	0.9300
C(19)-C(20)	1.358(15)
C(19)-H(19A)	0.9300
C(20)-C(21)	1.384(16)

C(20)-H(20A)	0.9300
C(21)-N(2)	1.369(12)
N(2)-C(22)	1.55(3)
C(22)-H(22A)	0.9600
C(22)-H(22B)	0.9600
C(22)-H(22C)	0.9600
O(3')-C(21')	1.238(13)
C(17')-N(2')	1.352(16)
C(17')-C(18')	1.352(13)
C(18')-C(19')	1.414(13)
C(18')-H(18C)	0.9300
C(19')-C(20')	1.372(16)
C(19')-H(19B)	0.9300
C(20')-C(21')	1.401(16)
C(20')-H(20B)	0.9300
C(21')-N(2')	1.382(18)
N(2')-C(22')	1.57(3)
C(22')-H(22D)	0.9600
C(22')-H(22E)	0.9600
C(22')-H(22F)	0.9600
O(4)-C(23)	1.212(6)
O(4')-C(23)	1.200(16)
O(5)-C(23)	1.296(4)
O(5)-H(5)	0.8200
O(6)-C(24)	1.226(4)
O(7)-C(24)	1.259(4)
C(23)-C(24)	1.542(5)

C(1S)-O(1S)	1.435(17)
C(1S)-H(1S1)	0.9600
C(1S)-H(1S2)	0.9600
C(1S)-H(1S3)	0.9600
O(1S)-H(1S)	0.8200
O(1S')-C(1S')	1.45(3)
O(1S')-H(1S')	0.8200
C(1S')-H(1S4)	0.9600
C(1S')-H(1S5)	0.9600
C(1S')-H(1S6)	0.9600

C(7)-O(1)-H(1)	109.5
C(11)-N(1)-C(15)	109.3(3)
C(11)-N(1)-C(8)	113.3(3)
C(15)-N(1)-C(8)	114.4(3)
C(11)-N(1)-H(1B)	106.4
C(15)-N(1)-H(1B)	106.4
C(8)-N(1)-H(1B)	106.4
C(10)-C(1)-C(2)	120.5(6)
C(10)-C(1)-H(1A)	119.8
C(2)-C(1)-H(1A)	119.8
C(3)-C(2)-C(1)	120.1(6)
C(3)-C(2)-H(2A)	120.0
C(1)-C(2)-H(2A)	120.0
C(2)-C(3)-C(4)	119.9(6)
C(2)-C(3)-H(3A)	120.0
C(4)-C(3)-H(3A)	120.0

C(3)-C(4)-C(5)	121.6(6)
C(3)-C(4)-H(4A)	119.2
C(5)-C(4)-H(4A)	119.2
C(10)-C(5)-C(4)	118.2(4)
C(10)-C(5)-C(6)	120.6(4)
C(4)-C(5)-C(6)	121.2(4)
C(5)-C(6)-C(7)	115.2(3)
C(5)-C(6)-H(6A)	108.5
C(7)-C(6)-H(6A)	108.5
C(5)-C(6)-H(6B)	108.5
C(7)-C(6)-H(6B)	108.5
H(6A)-C(6)-H(6B)	107.5
O(1)-C(7)-C(6)	112.8(3)
O(1)-C(7)-C(8)	108.9(3)
C(6)-C(7)-C(8)	109.8(3)
O(1)-C(7)-H(7A)	108.4
C(6)-C(7)-H(7A)	108.4
C(8)-C(7)-H(7A)	108.4
C(9)-C(8)-C(7)	111.8(3)
C(9)-C(8)-N(1)	109.2(3)
C(7)-C(8)-N(1)	114.5(3)
C(9)-C(8)-H(8A)	107.0
C(7)-C(8)-H(8A)	107.0
N(1)-C(8)-H(8A)	107.0
C(8)-C(9)-C(10)	114.2(3)
C(8)-C(9)-H(9A)	108.7
C(10)-C(9)-H(9A)	108.7

C(8)-C(9)-H(9B)	108.7
C(10)-C(9)-H(9B)	108.7
H(9A)-C(9)-H(9B)	107.6
C(1)-C(10)-C(5)	119.6(4)
C(1)-C(10)-C(9)	118.8(4)
C(5)-C(10)-C(9)	121.6(4)
N(1)-C(11)-C(12)	110.6(3)
N(1)-C(11)-H(11A)	109.5
C(12)-C(11)-H(11A)	109.5
N(1)-C(11)-H(11B)	109.5
C(12)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	108.1
C(11)-C(12)-C(13)	111.2(3)
C(11)-C(12)-H(12A)	109.4
C(13)-C(12)-H(12A)	109.4
C(11)-C(12)-H(12B)	109.4
C(13)-C(12)-H(12B)	109.4
H(12A)-C(12)-H(12B)	108.0
C(16)-C(13)-C(14)	111.4(4)
C(16)-C(13)-C(12)	109.7(4)
C(14)-C(13)-C(12)	108.5(3)
C(16)-C(13)-H(13A)	109.1
C(14)-C(13)-H(13A)	109.1
C(12)-C(13)-H(13A)	109.1
C(15)-C(14)-C(13)	113.1(3)
C(15)-C(14)-H(14A)	109.0
C(13)-C(14)-H(14A)	109.0

C(15)-C(14)-H(14B)	109.0
C(13)-C(14)-H(14B)	109.0
H(14A)-C(14)-H(14B)	107.8
N(1)-C(15)-C(14)	109.2(3)
N(1)-C(15)-H(15A)	109.8
C(14)-C(15)-H(15A)	109.8
N(1)-C(15)-H(15B)	109.8
C(14)-C(15)-H(15B)	109.8
H(15A)-C(15)-H(15B)	108.3
O(2)-C(16)-C(13)	123.7(5)
O(2)-C(16)-C(17')	112.3(6)
C(13)-C(16)-C(17')	117.3(5)
O(2)-C(16)-C(17)	111.7(5)
C(13)-C(16)-C(17)	118.7(5)
C(17')-C(16)-C(17)	52.9(5)
C(18)-C(17)-N(2)	120.5(8)
C(18)-C(17)-C(16)	123.6(8)
N(2)-C(17)-C(16)	115.7(7)
C(17)-C(18)-C(19)	118.8(10)
C(17)-C(18)-H(18A)	120.6
C(19)-C(18)-H(18A)	120.6
C(20)-C(19)-C(18)	117.7(11)
C(20)-C(19)-H(19A)	121.1
C(18)-C(19)-H(19A)	121.1
C(19)-C(20)-C(21)	123.9(10)
C(19)-C(20)-H(20A)	118.0
C(21)-C(20)-H(20A)	118.0

O(3)-C(21)-N(2)	122.1(12)
O(3)-C(21)-C(20)	122.5(10)
N(2)-C(21)-C(20)	115.4(10)
C(17)-N(2)-C(21)	123.3(9)
C(17)-N(2)-C(22)	120.5(13)
C(21)-N(2)-C(22)	116.2(13)
N(2')-C(17')-C(18')	119.5(12)
N(2')-C(17')-C(16)	125.4(11)
C(18')-C(17')-C(16)	114.0(9)
C(17')-C(18')-C(19')	119.4(11)
C(17')-C(18')-H(18C)	120.3
C(19')-C(18')-H(18C)	120.3
C(20')-C(19')-C(18')	118.8(12)
C(20')-C(19')-H(19B)	120.6
C(18')-C(19')-H(19B)	120.6
C(19')-C(20')-C(21')	122.8(11)
C(19')-C(20')-H(20B)	118.6
C(21')-C(20')-H(20B)	118.6
O(3')-C(21')-N(2')	120.7(14)
O(3')-C(21')-C(20')	125.3(12)
N(2')-C(21')-C(20')	114.0(12)
C(17')-N(2')-C(21')	125.2(16)
C(17')-N(2')-C(22')	119.2(14)
C(21')-N(2')-C(22')	115.0(13)
N(2')-C(22')-H(22D)	109.5
N(2')-C(22')-H(22E)	109.5
H(22D)-C(22')-H(22E)	109.5

N(2')-C(22')-H(22F)	109.5
H(22D)-C(22')-H(22F)	109.5
H(22E)-C(22')-H(22F)	109.5
C(23)-O(5)-H(5)	109.5
O(4')-C(23)-O(4)	37.1(12)
O(4')-C(23)-O(5)	120.6(12)
O(4)-C(23)-O(5)	124.4(4)
O(4')-C(23)-C(24)	116.1(12)
O(4)-C(23)-C(24)	121.6(3)
O(5)-C(23)-C(24)	113.5(3)
O(6)-C(24)-O(7)	126.8(3)
O(6)-C(24)-C(23)	118.3(3)
O(7)-C(24)-C(23)	114.9(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
C(1S)-O(1S)-H(1S)	109.5
C(1S')-O(1S')-H(1S')	109.5
O(1S')-C(1S')-H(1S4)	109.5
O(1S')-C(1S')-H(1S5)	109.5
H(1S4)-C(1S')-H(1S5)	109.5
O(1S')-C(1S')-H(1S6)	109.5
H(1S4)-C(1S')-H(1S6)	109.5
H(1S5)-C(1S')-H(1S6)	109.5

Table 6. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **19i**. 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}
O(1)	52(2)	31(1)	72(2)	8(1)	26(2)	-6(1)
O(2)	97(3)	125(3)	64(2)	-19(2)	18(2)	-36(2)
N(1)	27(2)	26(1)	47(2)	9(1)	11(1)	2(1)
C(1)	101(4)	93(4)	53(3)	15(3)	29(3)	-1(3)
C(2)	143(6)	122(6)	49(3)	23(4)	32(4)	20(5)
C(3)	150(7)	96(5)	50(4)	4(3)	4(4)	16(4)
C(4)	100(4)	87(4)	54(3)	-3(3)	-4(3)	-2(3)
C(5)	64(3)	48(2)	48(3)	5(2)	-1(2)	14(2)
C(6)	48(3)	48(2)	63(3)	-2(2)	-1(2)	-4(2)
C(7)	35(2)	32(2)	55(3)	10(2)	10(2)	1(2)
C(8)	33(2)	33(2)	41(2)	9(2)	6(2)	-1(1)
C(9)	53(2)	58(3)	52(3)	13(2)	17(2)	-8(2)
C(10)	51(2)	55(2)	45(3)	9(2)	5(2)	8(2)
C(11)	26(2)	30(2)	55(2)	9(2)	12(2)	6(1)
C(12)	37(2)	50(2)	52(3)	0(2)	17(2)	8(2)
C(13)	46(2)	61(3)	47(3)	1(2)	14(2)	8(2)
C(14)	39(2)	57(3)	51(3)	12(2)	8(2)	16(2)
C(15)	31(2)	35(2)	49(2)	7(2)	11(2)	12(2)
C(16)	62(3)	87(4)	52(3)	1(3)	9(3)	3(3)
O(3)	246(14)	164(10)	55(5)	-14(6)	38(7)	43(9)

C(17)	66(6)	65(6)	46(5)	8(5)	7(4)	22(5)
C(18)	113(9)	82(8)	66(7)	29(6)	8(6)	25(6)
C(19)	127(12)	117(11)	113(12)	65(10)	28(10)	33(9)
C(20)	133(12)	165(15)	39(6)	28(8)	20(7)	53(10)
C(21)	112(10)	121(10)	50(7)	29(8)	27(7)	33(8)
N(2)	101(6)	69(5)	47(5)	8(4)	13(4)	32(4)
C(22)	180(30)	92(15)	150(30)	-8(15)	40(20)	24(16)
O(3')	197(11)	111(8)	152(10)	-54(8)	28(8)	53(8)
C(17')	74(7)	98(9)	45(6)	22(6)	4(5)	26(6)
C(18')	82(7)	97(8)	45(6)	9(6)	23(6)	33(6)
C(19')	93(9)	128(12)	61(7)	21(8)	22(7)	39(8)
C(20')	101(11)	152(14)	74(9)	-2(10)	22(8)	50(10)
C(21')	109(11)	102(11)	79(9)	-10(8)	14(8)	60(9)
N(2')	103(11)	60(8)	116(14)	-23(8)	31(11)	28(7)
C(22')	162(15)	96(11)	150(15)	4(11)	41(12)	-8(10)
O(4)	31(2)	24(2)	172(9)	37(3)	8(3)	9(1)
O(4')	31(2)	24(2)	172(9)	37(3)	8(3)	9(1)
O(5)	17(1)	35(1)	81(2)	12(1)	8(1)	4(1)
O(6)	25(1)	30(1)	97(2)	24(1)	8(1)	7(1)
O(7)	22(1)	37(1)	97(2)	26(2)	14(1)	4(1)
C(23)	22(2)	30(2)	71(3)	11(2)	8(2)	2(1)
C(24)	23(2)	26(2)	52(2)	11(2)	4(2)	5(1)
C(1S)	220(20)	170(17)	92(11)	-81(11)	-14(12)	-17(14)
O(1S)	360(30)	156(12)	174(13)	51(10)	29(14)	63(13)
O(1S')	600(50)	460(40)	190(20)	140(20)	220(30)	400(40)
C(1S')	600(50)	460(40)	190(20)	140(20)	220(30)	400(40)

Table 7. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **19i**.

	x	y	z	U(eq)
H(1)	8567	8676	8228	77
H(1B)	3024	4511	7994	40
H(1A)	1425	5579	9768	99
H(2A)	2482	6882	10624	123
H(3A)	5634	8791	10805	121
H(4A)	8005	9291	10166	101
H(6A)	9035	7697	9190	67
H(6B)	8084	9234	9197	67
H(7A)	5142	8269	8524	49
H(8A)	6457	5433	8625	43
H(9A)	2983	4740	8919	66
H(9B)	1886	6120	8753	66
H(11A)	6827	4213	7807	43
H(11B)	6894	5687	7552	43
H(12A)	3582	3027	7186	55
H(12B)	5753	3585	6897	55
H(13A)	4024	5679	6708	62
H(14A)	403	6120	6998	57
H(14B)	184	4641	7249	57
H(15A)	3683	7217	7629	45

H(15B)	1478	6696	7916	45
H(18A)	2608	6567	5955	101
H(19A)	2328	7008	5063	135
H(20A)	1821	4991	4409	129
H(22A)	3104	1526	5766	210
H(22B)	2121	902	5174	210
H(22C)	327	1173	5586	210
H(18C)	5080	5131	5921	86
H(19B)	7048	4302	5203	109
H(20B)	5978	1875	4777	129
H(22D)	-608	303	5716	208
H(22E)	810	917	6277	208
H(22F)	1643	-389	5914	208
H(5)	5547	1656	8211	66
H(1S1)	7959	9066	7205	268
H(1S2)	6164	9474	6762	268
H(1S3)	8940	9864	6754	268
H(1S)	7730	7808	6218	338
H(1S')	3136	8227	6392	546
H(1S4)	6337	10481	6597	546
H(1S5)	7090	9314	6952	546
H(1S6)	4649	9918	6995	546

Table 8. Torsion angles [°] for **19i**.

C(10)-C(1)-C(2)-C(3)	1.6(10)
C(1)-C(2)-C(3)-C(4)	-2.9(11)
C(2)-C(3)-C(4)-C(5)	2.9(10)
C(3)-C(4)-C(5)-C(10)	-1.4(8)
C(3)-C(4)-C(5)-C(6)	178.8(5)
C(10)-C(5)-C(6)-C(7)	20.0(6)
C(4)-C(5)-C(6)-C(7)	-160.3(4)
C(5)-C(6)-C(7)-O(1)	-169.5(3)
C(5)-C(6)-C(7)-C(8)	-47.9(5)
O(1)-C(7)-C(8)-C(9)	-176.7(3)
C(6)-C(7)-C(8)-C(9)	59.4(4)
O(1)-C(7)-C(8)-N(1)	-51.8(4)
C(6)-C(7)-C(8)-N(1)	-175.7(3)
C(11)-N(1)-C(8)-C(9)	-152.2(3)
C(15)-N(1)-C(8)-C(9)	81.6(4)
C(11)-N(1)-C(8)-C(7)	81.5(4)
C(15)-N(1)-C(8)-C(7)	-44.7(4)
C(7)-C(8)-C(9)-C(10)	-42.1(5)
N(1)-C(8)-C(9)-C(10)	-169.9(3)
C(2)-C(1)-C(10)-C(5)	-0.1(8)
C(2)-C(1)-C(10)-C(9)	-178.5(5)
C(4)-C(5)-C(10)-C(1)	0.1(7)
C(6)-C(5)-C(10)-C(1)	179.8(5)
C(4)-C(5)-C(10)-C(9)	178.4(4)
C(6)-C(5)-C(10)-C(9)	-1.8(6)

C(8)-C(9)-C(10)-C(1)	-168.3(4)
C(8)-C(9)-C(10)-C(5)	13.4(6)
C(15)-N(1)-C(11)-C(12)	-61.7(4)
C(8)-N(1)-C(11)-C(12)	169.4(3)
N(1)-C(11)-C(12)-C(13)	59.2(4)
C(11)-C(12)-C(13)-C(16)	-175.8(4)
C(11)-C(12)-C(13)-C(14)	-53.9(4)
C(16)-C(13)-C(14)-C(15)	174.9(4)
C(12)-C(13)-C(14)-C(15)	54.1(5)
C(11)-N(1)-C(15)-C(14)	59.9(4)
C(8)-N(1)-C(15)-C(14)	-171.9(3)
C(13)-C(14)-C(15)-N(1)	-57.9(4)
C(14)-C(13)-C(16)-O(2)	-30.3(7)
C(12)-C(13)-C(16)-O(2)	89.9(6)
C(14)-C(13)-C(16)-C(17')	-179.1(6)
C(12)-C(13)-C(16)-C(17')	-59.0(7)
C(14)-C(13)-C(16)-C(17)	120.3(5)
C(12)-C(13)-C(16)-C(17)	-119.5(5)
O(2)-C(16)-C(17)-C(18)	115.0(10)
C(13)-C(16)-C(17)-C(18)	-38.9(12)
C(17')-C(16)-C(17)-C(18)	-142.9(12)
O(2)-C(16)-C(17)-N(2)	-59.6(9)
C(13)-C(16)-C(17)-N(2)	146.5(7)
C(17')-C(16)-C(17)-N(2)	42.5(8)
N(2)-C(17)-C(18)-C(19)	6.6(16)
C(16)-C(17)-C(18)-C(19)	-167.7(9)
C(17)-C(18)-C(19)-C(20)	-3.0(19)

C(18)-C(19)-C(20)-C(21)	0(2)
C(19)-C(20)-C(21)-O(3)	178.8(15)
C(19)-C(20)-C(21)-N(2)	0(2)
C(18)-C(17)-N(2)-C(21)	-7.1(16)
C(16)-C(17)-N(2)-C(21)	167.7(9)
C(18)-C(17)-N(2)-C(22)	172.6(17)
C(16)-C(17)-N(2)-C(22)	-12.7(18)
O(3)-C(21)-N(2)-C(17)	-175.1(12)
C(20)-C(21)-N(2)-C(17)	3.5(17)
O(3)-C(21)-N(2)-C(22)	5(2)
C(20)-C(21)-N(2)-C(22)	-176.2(17)
O(2)-C(16)-C(17')-N(2')	-15.9(17)
C(13)-C(16)-C(17')-N(2')	136.4(15)
C(17)-C(16)-C(17')-N(2')	-116.9(17)
O(2)-C(16)-C(17')-C(18')	152.1(8)
C(13)-C(16)-C(17')-C(18')	-55.5(10)
C(17)-C(16)-C(17')-C(18')	51.1(8)
N(2')-C(17')-C(18')-C(19')	0(2)
C(16)-C(17')-C(18')-C(19')	-169.2(9)
C(17')-C(18')-C(19')-C(20')	1.6(17)
C(18')-C(19')-C(20')-C(21')	-4(2)
C(19')-C(20')-C(21')-O(3')	-173.4(14)
C(19')-C(20')-C(21')-N(2')	6(2)
C(18')-C(17')-N(2')-C(21')	2(3)
C(16)-C(17')-N(2')-C(21')	169.4(16)
C(18')-C(17')-N(2')-C(22')	173.0(15)
C(16)-C(17')-N(2')-C(22')	-20(3)

O(3')-C(21')-N(2')-C(17')	174.6(18)
C(20')-C(21')-N(2')-C(17')	-4(3)
O(3')-C(21')-N(2')-C(22')	3(3)
C(20')-C(21')-N(2')-C(22')	-175.7(16)
O(4')-C(23)-C(24)-O(6)	-127.7(18)
O(4)-C(23)-C(24)-O(6)	-169.8(9)
O(5)-C(23)-C(24)-O(6)	18.4(6)
O(4')-C(23)-C(24)-O(7)	52.6(19)
O(4)-C(23)-C(24)-O(7)	10.5(10)
O(5)-C(23)-C(24)-O(7)	-161.3(3)

Projection view with 30% thermal ellipsoids- disorder components omitted for clarity: