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4-Chloro-6-methoxy-*N*-(2,2,6,6-tetramethylpiperidin-4-yl)-1,3,5-triazin-2-aminium chloride

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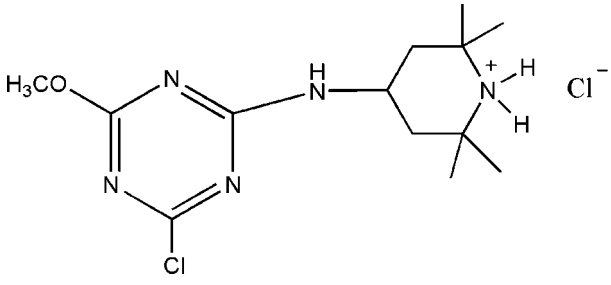
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.100; data-to-parameter ratio = 16.1.

The crystal structure of the title compound, $C_{13}H_{23}ClN_5O^+\cdot Cl^-$, is stabilized by intermolecular N—H···Cl hydrogen bonds which help to establish the crystal packing. The piperidine ring adopts a chair conformation.

Related literature

For general background, see Borzatta & Carrozza (1991). For related structures, see Deng *et al.* (2006).

For related literature, see: Kaiser & Thurston (1951).



Experimental

Crystal data

$C_{13}H_{23}ClN_5O^+\cdot Cl^-$
M_r = 336.26
Monoclinic, $P2_1/n$
a = 7.7987 (16) Å
b = 8.9425 (18) Å
c = 24.472 (5) Å
β = 90.36 (3)°
V = 1706.7 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 113 (2) K
0.30 × 0.26 × 0.22 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.893, T_{max} = 0.920
13533 measured reflections
3338 independent reflections
3184 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.100
S = 1.08
3338 reflections
207 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···Cl2^{i}	0.82 (3)	2.45 (3)	3.2639 (19)	171 (2)
N5—H5A···Cl2	0.86 (2)	2.33 (2)	3.1821 (17)	173.2 (19)
N5—H5B···Cl2^{ii}	0.85 (3)	2.32 (3)	3.1714 (18)	177 (2)

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2461).

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supplementary materials

Acta Cryst. (2007). E**63**, o3716 [**doi:10.1107/S1600536807037877**]

4-Chloro-6-methoxy-*N*-(2,2,6,6-tetramethylpiperidin-4-yl)-1,3,5-triazin-2-aminium chloride

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Comment

The title compound is an intermediate for the synthesis of hindered light stabilizers (Borzatta & Carrozza, 1991). Its derivatives have many applications in organic chemistry (Deng *et al.*, 2006).

The triazine ring is essentially planar with an r.m.s. deviation from the mean plane of 0.0038 Å. The molecules are linked by intermolecular N—H···Cl hydrogen bonds (Table 1) forming zigzag chains.

Experimental

The title compound was obtained according to the method of Kaiser & Thurston (1951). 2,4,6-Trichloro-1,3,5-triazine (40.0 g, 0.217 mol) was dissolved in toluene (120 ml) and then cooled to 278 K. With stirring, a solution of 2,2,6,6-tetramethylpiperidin-4-amine(33.23 g, 0.213 mol) in toluene (50 ml) was then added dropwise to the mixture over a period of 0.5 h. Then a solution of Na_2CO_3 (23.02 g, 0.217 mol) in water (50 ml) was then added dropwise for 0.5 h. The mixture was stirred at 273–278 K for further 3 h. The organic layer was washed with water and then concentrated *in vacuo*. 4,6-dichloro-*N*-(2,2,6,6-tetramethylpiperidin-4-yl)-1,3,5-triazin-2-amine (60.88 g) was obtained in powder form in a yield of 85.0%. Crystals were obtained by slow evaporation of a solution of methanol.

Refinement

H atoms bonded to C were positioned geometrically (C—H=0.96–0.97 Å), and refined as riding with U_{iso}(H)=1.2U_{eq}(C) or 1.5$_{eq}$(C_{methyl}). The methyl groups were allowed to rotate but not to tip. H atoms bonded to N were freely refined.

Figures

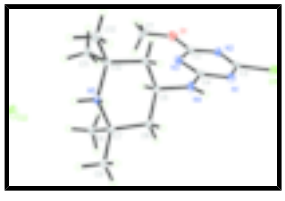

Fig. 1. A view of the molecular structure of the title compound. Displacement ellopsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

4-Chloro-6-methoxy-*N*-(2,2,6,6-tetramethylpiperidin-4-yl)- 1,3,5-triazin-2-aminium chloride

Crystal data

$C_{13}H_{23}ClN_5O^+\cdot Cl^-$	F_{000} = 712
M_r = 336.26	D_x = 1.309 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation

	λ = 0.71073 Å
a = 7.7987 (16) Å	Cell parameters from 2231 reflections
b = 8.9425 (18) Å	θ = 2.3–25.0º
c = 24.472 (5) Å	μ = 0.39 mm^{-1}
β = 90.36 (3)º	*T* = 113 (2) K
V = 1706.7 (6) $Å^3$	Block, colorless
Z = 4	0.30 × 0.26 × 0.22 mm

Data collection

Rigaku Saturn diffractometer	3338 independent reflections
Radiation source: rotating anode	3184 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.026
T = 113(2) K	θ_{max} = 26.0º
ω and φ scans	θ_{min} = 1.7º
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2005)	*h* = −9→9
T_{min} = 0.893, T_{max} = 0.920	*k* = −11→10
13533 measured reflections	*l* = −30→30

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.041	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.100	$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 1.4811P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.003
3338 reflections	$\Delta\rho_{max}$ = 0.73 e $Å^{-3}$
207 parameters	$\Delta\rho_{min}$ = −0.38 e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* 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 $F^2 > 2\text{sigma}(F^2)$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.81987 (7)	1.44507 (5)	0.04206 (2)	0.02810 (15)
O1	0.8043 (2)	0.94806 (17)	−0.03288 (6)	0.0332 (4)
C1	0.7679 (3)	0.7961 (3)	−0.03155 (10)	0.0363 (5)
H1A	0.8330	0.7497	−0.0028	0.054*
H1B	0.7979	0.7517	−0.0659	0.054*
H1C	0.6477	0.7818	−0.0251	0.054*
Cl2	0.39494 (7)	0.25965 (5)	0.22079 (2)	0.02840 (15)
N1	0.6796 (2)	1.21886 (18)	0.09038 (7)	0.0225 (4)
N2	0.8057 (2)	1.17694 (19)	0.00360 (7)	0.0248 (4)
N3	0.6822 (2)	0.97133 (19)	0.05197 (7)	0.0232 (4)
N4	0.5635 (2)	1.02010 (19)	0.13604 (7)	0.0225 (4)
H4	0.533 (3)	1.085 (3)	0.1578 (10)	0.031 (6)*
N5	0.3789 (2)	0.61101 (18)	0.20083 (6)	0.0158 (3)
C2	0.7615 (3)	1.0330 (2)	0.00984 (8)	0.0239 (4)
C3	0.7600 (2)	1.2577 (2)	0.04591 (8)	0.0219 (4)
C4	0.6431 (2)	1.0695 (2)	0.09169 (8)	0.0215 (4)
C5	0.5324 (2)	0.8625 (2)	0.14724 (7)	0.0189 (4)
H5	0.6095	0.8027	0.1245	0.023*
C6	0.5720 (2)	0.8292 (2)	0.20701 (8)	0.0203 (4)
H6A	0.4947	0.8863	0.2298	0.024*
H6B	0.6881	0.8612	0.2153	0.024*
C7	0.5540 (2)	0.6633 (2)	0.22059 (7)	0.0178 (4)
C8	0.6969 (2)	0.5703 (2)	0.19527 (8)	0.0236 (4)
H8A	0.6676	0.4661	0.1972	0.035*
H8B	0.8021	0.5875	0.2149	0.035*
H8C	0.7111	0.5986	0.1577	0.035*
C9	0.5543 (3)	0.6403 (2)	0.28253 (8)	0.0254 (4)
H9A	0.4610	0.6952	0.2983	0.038*
H9B	0.6608	0.6756	0.2976	0.038*
H9C	0.5413	0.5359	0.2905	0.038*
C10	0.3192 (2)	0.6498 (2)	0.14307 (7)	0.0187 (4)
C11	0.4114 (3)	0.5538 (2)	0.10096 (8)	0.0259 (4)
H11A	0.3499	0.5574	0.0669	0.039*
H11B	0.4167	0.4523	0.1136	0.039*
H11C	0.5255	0.5912	0.0959	0.039*
C12	0.1282 (3)	0.6144 (2)	0.14119 (9)	0.0301 (5)
H12A	0.0703	0.6708	0.1689	0.045*
H12B	0.1113	0.5095	0.1475	0.045*
H12C	0.0826	0.6405	0.1059	0.045*
C13	0.3487 (2)	0.8168 (2)	0.13443 (8)	0.0199 (4)
H13A	0.3221	0.8421	0.0968	0.024*
H13B	0.2715	0.8727	0.1577	0.024*
H5A	0.375 (3)	0.516 (3)	0.2050 (8)	0.020 (5)*
H5B	0.307 (3)	0.649 (3)	0.2227 (10)	0.030 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0336 (3)	0.0207 (3)	0.0301 (3)	−0.00790 (19)	0.0058 (2)	0.00253 (18)
O1	0.0408 (9)	0.0347 (9)	0.0240 (7)	0.0047 (7)	0.0050 (6)	0.0038 (6)
C1	0.0308 (12)	0.0306 (12)	0.0474 (14)	0.0022 (9)	−0.0033 (10)	−0.0169 (10)
Cl2	0.0318 (3)	0.0169 (2)	0.0367 (3)	−0.00237 (18)	0.0135 (2)	0.00176 (19)
N1	0.0243 (8)	0.0185 (8)	0.0247 (8)	−0.0038 (6)	0.0008 (7)	0.0033 (6)
N2	0.0283 (9)	0.0219 (8)	0.0243 (8)	−0.0009 (7)	0.0002 (7)	0.0019 (7)
N3	0.0235 (8)	0.0236 (8)	0.0225 (8)	−0.0002 (7)	−0.0005 (7)	0.0036 (7)
N4	0.0292 (9)	0.0151 (8)	0.0233 (8)	−0.0030 (7)	0.0054 (7)	0.0011 (7)
N5	0.0155 (7)	0.0135 (8)	0.0183 (8)	−0.0011 (6)	0.0021 (6)	0.0013 (6)
C2	0.0261 (10)	0.0256 (10)	0.0199 (9)	0.0036 (8)	−0.0035 (8)	−0.0008 (8)
C3	0.0210 (9)	0.0217 (10)	0.0230 (10)	−0.0031 (7)	−0.0023 (8)	0.0040 (7)
C4	0.0192 (9)	0.0223 (10)	0.0230 (9)	−0.0017 (7)	−0.0015 (7)	0.0042 (8)
C5	0.0212 (9)	0.0146 (9)	0.0209 (9)	−0.0018 (7)	0.0020 (7)	0.0026 (7)
C6	0.0208 (9)	0.0180 (9)	0.0222 (9)	−0.0048 (7)	−0.0042 (7)	−0.0002 (7)
C7	0.0148 (8)	0.0186 (9)	0.0200 (9)	−0.0029 (7)	−0.0024 (7)	0.0008 (7)
C8	0.0177 (9)	0.0234 (10)	0.0298 (10)	0.0020 (7)	0.0013 (8)	0.0048 (8)
C9	0.0292 (10)	0.0259 (10)	0.0210 (10)	0.0000 (8)	−0.0049 (8)	0.0031 (8)
C10	0.0181 (9)	0.0201 (9)	0.0180 (9)	−0.0031 (7)	−0.0030 (7)	0.0005 (7)
C11	0.0342 (11)	0.0228 (10)	0.0208 (9)	−0.0035 (8)	−0.0005 (8)	−0.0055 (8)
C12	0.0205 (10)	0.0339 (11)	0.0357 (12)	−0.0081 (9)	−0.0062 (9)	0.0021 (9)
C13	0.0208 (9)	0.0191 (9)	0.0197 (9)	0.0005 (7)	−0.0032 (7)	0.0034 (7)

Geometric parameters (Å, °)

Cl1—C3	1.742 (2)	C6—C7	1.527 (3)
O1—C2	1.336 (2)	C6—H6A	0.9700
O1—C1	1.389 (3)	C6—H6B	0.9700
C1—H1A	0.9600	C7—C8	1.525 (3)
C1—H1B	0.9600	C7—C9	1.530 (3)
C1—H1C	0.9600	C8—H8A	0.9600
N1—C3	1.307 (3)	C8—H8B	0.9600
N1—C4	1.366 (2)	C8—H8C	0.9600
N2—C3	1.313 (3)	C9—H9A	0.9600
N2—C2	1.341 (3)	C9—H9B	0.9600
N3—C2	1.326 (3)	C9—H9C	0.9600
N3—C4	1.346 (3)	C10—C12	1.523 (3)
N4—C4	1.329 (2)	C10—C11	1.525 (3)
N4—C5	1.456 (2)	C10—C13	1.526 (3)
N4—H4	0.82 (3)	C11—H11A	0.9600
N5—C7	1.520 (2)	C11—H11B	0.9600
N5—C10	1.525 (2)	C11—H11C	0.9600
N5—H5A	0.86 (2)	C12—H12A	0.9600
N5—H5B	0.85 (3)	C12—H12B	0.9600
C5—C13	1.521 (3)	C12—H12C	0.9600
C5—C6	1.522 (3)	C13—H13A	0.9700

C5—H5	0.9800	C13—H13B	0.9700
C2—O1—C1	119.09 (17)	N5—C7—C6	108.24 (14)
O1—C1—H1A	109.5	C8—C7—C6	111.90 (15)
O1—C1—H1B	109.5	N5—C7—C9	105.65 (14)
H1A—C1—H1B	109.5	C8—C7—C9	109.46 (16)
O1—C1—H1C	109.5	C6—C7—C9	110.26 (15)
H1A—C1—H1C	109.5	C7—C8—H8A	109.5
H1B—C1—H1C	109.5	C7—C8—H8B	109.5
C3—N1—C4	112.36 (17)	H8A—C8—H8B	109.5
C3—N2—C2	111.51 (17)	C7—C8—H8C	109.5
C2—N3—C4	113.51 (17)	H8A—C8—H8C	109.5
C4—N4—C5	123.67 (17)	H8B—C8—H8C	109.5
C4—N4—H4	115.7 (17)	C7—C9—H9A	109.5
C5—N4—H4	120.6 (17)	C7—C9—H9B	109.5
C7—N5—C10	119.56 (14)	H9A—C9—H9B	109.5
C7—N5—H5A	107.6 (14)	C7—C9—H9C	109.5
C10—N5—H5A	108.8 (14)	H9A—C9—H9C	109.5
C7—N5—H5B	105.8 (16)	H9B—C9—H9C	109.5
C10—N5—H5B	107.0 (16)	C12—C10—C11	109.14 (16)
H5A—N5—H5B	107 (2)	C12—C10—N5	105.87 (15)
N3—C2—O1	119.45 (18)	C11—C10—N5	110.86 (15)
N3—C2—N2	127.54 (18)	C12—C10—C13	110.33 (16)
O1—C2—N2	113.02 (17)	C11—C10—C13	112.65 (15)
N1—C3—N2	130.13 (18)	N5—C10—C13	107.78 (14)
N1—C3—Cl1	115.52 (15)	C10—C11—H11A	109.5
N2—C3—Cl1	114.34 (14)	C10—C11—H11B	109.5
N4—C4—N3	118.80 (17)	H11A—C11—H11B	109.5
N4—C4—N1	116.26 (18)	C10—C11—H11C	109.5
N3—C4—N1	124.94 (17)	H11A—C11—H11C	109.5
N4—C5—C13	112.27 (15)	H11B—C11—H11C	109.5
N4—C5—C6	109.68 (15)	C10—C12—H12A	109.5
C13—C5—C6	109.31 (15)	C10—C12—H12B	109.5
N4—C5—H5	108.5	H12A—C12—H12B	109.5
C13—C5—H5	108.5	C10—C12—H12C	109.5
C6—C5—H5	108.5	H12A—C12—H12C	109.5
C5—C6—C7	112.37 (15)	H12B—C12—H12C	109.5
C5—C6—H6A	109.1	C5—C13—C10	112.14 (15)
C7—C6—H6A	109.1	C5—C13—H13A	109.2
C5—C6—H6B	109.1	C10—C13—H13A	109.2
C7—C6—H6B	109.1	C5—C13—H13B	109.2
H6A—C6—H6B	107.9	C10—C13—H13B	109.2
N5—C7—C8	111.14 (15)	H13A—C13—H13B	107.9
C4—N3—C2—O1	−178.66 (17)	C4—N4—C5—C6	136.65 (19)
C4—N3—C2—N2	0.6 (3)	N4—C5—C6—C7	−176.00 (15)
C1—O1—C2—N3	−1.4 (3)	C13—C5—C6—C7	60.5 (2)
C1—O1—C2—N2	179.17 (18)	C10—N5—C7—C8	−74.9 (2)
C3—N2—C2—N3	−0.1 (3)	C10—N5—C7—C6	48.4 (2)
C3—N2—C2—O1	179.19 (17)	C10—N5—C7—C9	166.45 (15)

C4—N1—C3—N2	1.5 (3)	C5—C6—C7—N5	−52.0 (2)
C4—N1—C3—Cl1	−177.39 (13)	C5—C6—C7—C8	70.9 (2)
C2—N2—C3—N1	−1.1 (3)	C5—C6—C7—C9	−167.06 (15)
C2—N2—C3—Cl1	177.82 (14)	C7—N5—C10—C12	−167.06 (16)
C5—N4—C4—N3	7.1 (3)	C7—N5—C10—C11	74.7 (2)
C5—N4—C4—N1	−172.68 (17)	C7—N5—C10—C13	−49.0 (2)
C2—N3—C4—N4	−179.88 (18)	N4—C5—C13—C10	176.70 (15)
C2—N3—C4—N1	−0.1 (3)	C6—C5—C13—C10	−61.3 (2)
C3—N1—C4—N4	178.98 (17)	C12—C10—C13—C5	168.49 (16)
C3—N1—C4—N3	−0.8 (3)	C11—C10—C13—C5	−69.3 (2)
C4—N4—C5—C13	−101.6 (2)	N5—C10—C13—C5	53.34 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···Cl2^{i}	0.82 (3)	2.45 (3)	3.2639 (19)	171 (2)
N5—H5A···Cl2	0.86 (2)	2.33 (2)	3.1821 (17)	173.2 (19)
N5—H5B···Cl2^{ii}	0.85 (3)	2.32 (3)	3.1714 (18)	177 (2)

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2.

Fig. 1