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4-Chloro-6-morpholino-N^2-(2,4,4-trimethylpentan-2-yl)-1,3,5-triazin-2-amine

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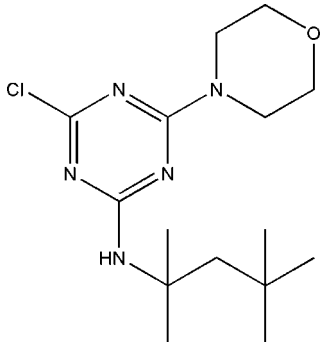
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.114; data-to-parameter ratio = 17.4.

The title compound, $C_{15}H_{26}ClN_5O$, was synthesized by the reaction of 2,4,6-trichloro-1,3,5-triazine with 2,4,4-trimethylpentan-2-amine and morpholine. The triazine ring is planar and the morpholine ring displays a typical chair conformation. Intermolecular N—H···O hydrogen bonding is present in the crystal structure.

Related literature

For general background, see Borzatta & Carrozza (1991). For a related structure, see Deng *et al.* (2006). For synthesis, see Kaiser & Thurston (1951).



Experimental

Crystal data

$C_{15}H_{26}ClN_5O$
M_r = 327.86
Monoclinic, $P2_1/n$
a = 14.119 (3) Å
b = 7.4964 (17) Å
c = 17.083 (4) Å
β = 101.939 (4)°
V = 1769.0 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 294 (2) K
0.24 × 0.18 × 0.12 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: none
9840 measured reflections
3618 independent reflections
2385 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.114
S = 1.00
3618 reflections
208 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O1^{i}	0.891 (9)	2.197 (10)	3.076 (2)	168.9 (17)

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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: XU2270).

References

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Kaiser, D. W. & Thurston, J. T. (1951). *J. Am. Chem. Soc.* **73**, 2984–2986.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

Acta Cryst. (2007). E**63**, o3173 [**doi:10.1107/S1600536807027924**]

4-Chloro-6-morpholino-N^2-(2,4,4-trimethylpentan-2-yl)-1,3,5-triazin-2-amine

Y.-P. Jiang, T. Qiu, L.-E. Wang and J.-N. Wang

Comment

The title compound is an important intermediate of hindered light stabilizers (Borzatta & Carrozza, 1991). These compounds containing triazine ring are widely used (Deng *et al.*, 2006). We report here the crystal structure of the title compound (Fig. 1). The triazine ring in the title compound is essentially planar with an r.m.s. deviation from the mean plane of 0.009 Å.

Experimental

The title compound was prepared 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 2,4,4-trimethylpentan-2-amine (27.5 g, 0.213 mol) in toluene (50 ml) was 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 added dropwise for 0.5 h. The mixture was stirred at 273–278 K for a further 3 h. Morpholine (18.9 g, 0.217 mol) and solid Na_2CO_3 (23.02 g, 0.217 mol) were added to the mixture, keeping temperature at 338 k for 5 h. The title compound (54.91 g) was obtained in powder form in a yield of 78.6%. Single crystals of the title compound were obtained by slow evaporation of a methanol solution.

Refinement

Imino H4 atom was located in a difference Fourier map and isotropically refined with a restraint of O—H = 0.90±0.01 Å. Other H atoms were positioned geometrically with C—H = 0.96–0.97 Å, and refined in riding mode with U_{iso}(H) = 1.5U_{eq}(C) for methyl groups or 1.2U_{eq}(C) for others.

Figures

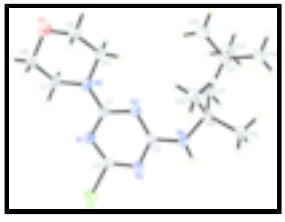

Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

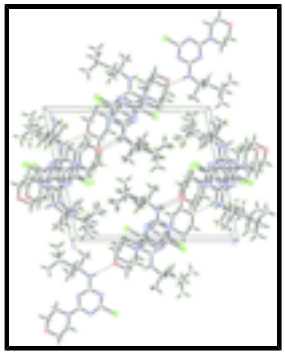

Fig. 2. The unit cell packing diagram of the title compound.

4-Chloro-6-morpholino-*N*^{2}-(2,4,4-trimethylpentan-2-yl)-1,3,5-triazin-2-amine

Crystal data

$C_{15}H_{26}ClN_5O$	F_{000} = 704
M_r = 327.86	D_x = 1.231 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 369-371 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 14.119 (3) Å	Cell parameters from 2545 reflections
b = 7.4964 (17) Å	θ = 2.9–24.5º
c = 17.083 (4) Å	μ = 0.23 mm^{-1}
β = 101.939 (4)º	T = 294 (2) K
V = 1769.0 (7) Å^3	Prism, colourless
Z = 4	0.24 × 0.18 × 0.12 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	2385 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.035
Monochromator: graphite	θ_{max} = 26.7º
T = 294(2) K	θ_{min} = 1.7º
φ and ω scans	h = −16→17
Absorption correction: none	k = −7→9
9840 measured reflections	l = −21→17
3618 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.114	$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.2056P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
3618 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	0.43258 (4)	0.68022 (8)	1.16149 (3)	0.05564 (19)
O1	0.31616 (10)	0.8309 (2)	0.72604 (8)	0.0534 (4)
N1	0.43414 (11)	0.7080 (2)	1.01064 (9)	0.0402 (4)
N2	0.58416 (11)	0.7425 (2)	1.10318 (8)	0.0385 (4)
N3	0.57800 (10)	0.7775 (2)	0.96303 (8)	0.0385 (4)
N4	0.72149 (11)	0.8004 (2)	1.05739 (9)	0.0419 (4)
N5	0.43047 (11)	0.7482 (2)	0.87630 (9)	0.0466 (4)
C1	0.48256 (13)	0.7441 (2)	0.95198 (10)	0.0368 (4)
C2	0.49106 (13)	0.7137 (2)	1.08183 (11)	0.0373 (4)
C3	0.62534 (12)	0.7724 (2)	1.03887 (10)	0.0344 (4)
C4	0.47478 (15)	0.7818 (3)	0.80787 (11)	0.0532 (6)
H4A	0.4837	0.6699	0.7818	0.064*
H4B	0.5378	0.8366	0.8257	0.064*
C5	0.41114 (15)	0.9024 (3)	0.75028 (12)	0.0528 (5)
H5A	0.4075	1.0178	0.7752	0.063*
H5B	0.4392	0.9197	0.7036	0.063*
C6	0.27284 (14)	0.8130 (3)	0.79388 (12)	0.0518 (5)
H6A	0.2075	0.7675	0.7768	0.062*
H6B	0.2688	0.9294	0.8178	0.062*
C7	0.32985 (14)	0.6891 (3)	0.85542 (12)	0.0506 (5)
H7A	0.3022	0.6883	0.9029	0.061*
H7B	0.3269	0.5688	0.8342	0.061*
C8	0.78825 (13)	0.8290 (3)	1.00235 (10)	0.0390 (4)
C9	0.88419 (15)	0.8807 (3)	1.05725 (13)	0.0624 (6)
H9A	0.8739	0.9798	1.0901	0.094*

H9B	0.9300	0.9137	1.0254	0.094*
H9C	0.9088	0.7812	1.0907	0.094*
C10	0.75325 (16)	0.9854 (3)	0.94675 (13)	0.0574 (6)
H10A	0.6952	0.9521	0.9096	0.086*
H10B	0.8024	1.0169	0.9179	0.086*
H10C	0.7401	1.0858	0.9778	0.086*
C11	0.79641 (13)	0.6485 (2)	0.96018 (11)	0.0400 (4)
H11A	0.7311	0.6017	0.9451	0.048*
H11B	0.8315	0.5688	1.0008	0.048*
C12	0.84258 (13)	0.6268 (3)	0.88572 (11)	0.0458 (5)
C13	0.85638 (17)	0.4252 (3)	0.87652 (14)	0.0687 (7)
H13A	0.7955	0.3656	0.8732	0.103*
H13B	0.9022	0.3813	0.9220	0.103*
H13C	0.8801	0.4028	0.8287	0.103*
C14	0.94073 (16)	0.7175 (3)	0.89346 (15)	0.0703 (7)
H14A	0.9672	0.6897	0.8475	0.105*
H14B	0.9838	0.6757	0.9409	0.105*
H14C	0.9329	0.8443	0.8968	0.105*
C15	0.77405 (17)	0.6911 (3)	0.80975 (12)	0.0640 (6)
H15A	0.7701	0.8189	0.8104	0.096*
H15B	0.7109	0.6412	0.8073	0.096*
H15C	0.7981	0.6536	0.7638	0.096*
H4	0.7492 (13)	0.778 (2)	1.1082 (6)	0.046 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0497 (3)	0.0763 (4)	0.0457 (3)	−0.0021 (3)	0.0209 (2)	0.0055 (3)
O1	0.0470 (8)	0.0700 (10)	0.0372 (7)	−0.0010 (7)	−0.0050 (6)	−0.0001 (7)
N1	0.0337 (8)	0.0479 (10)	0.0386 (9)	−0.0018 (7)	0.0062 (7)	0.0017 (7)
N2	0.0345 (9)	0.0487 (10)	0.0317 (8)	0.0000 (7)	0.0058 (7)	−0.0015 (7)
N3	0.0315 (8)	0.0501 (10)	0.0325 (8)	−0.0008 (7)	0.0029 (6)	−0.0006 (7)
N4	0.0319 (8)	0.0616 (11)	0.0310 (8)	−0.0034 (7)	0.0036 (7)	−0.0017 (8)
N5	0.0317 (9)	0.0696 (12)	0.0356 (9)	−0.0029 (8)	−0.0001 (7)	0.0061 (8)
C1	0.0362 (10)	0.0365 (10)	0.0362 (10)	0.0020 (8)	0.0044 (8)	0.0018 (8)
C2	0.0388 (10)	0.0379 (11)	0.0369 (10)	0.0012 (8)	0.0114 (8)	−0.0008 (8)
C3	0.0320 (9)	0.0380 (11)	0.0325 (9)	0.0006 (8)	0.0053 (8)	−0.0019 (8)
C4	0.0396 (11)	0.0823 (17)	0.0363 (11)	0.0022 (10)	0.0048 (9)	0.0043 (11)
C5	0.0512 (13)	0.0620 (14)	0.0416 (11)	−0.0059 (10)	0.0012 (9)	0.0019 (10)
C6	0.0386 (11)	0.0620 (14)	0.0497 (12)	0.0023 (10)	−0.0029 (9)	−0.0047 (11)
C7	0.0397 (11)	0.0608 (14)	0.0461 (11)	−0.0087 (10)	−0.0032 (9)	0.0011 (10)
C8	0.0326 (9)	0.0482 (12)	0.0367 (10)	−0.0047 (8)	0.0082 (8)	0.0016 (9)
C9	0.0423 (12)	0.0892 (18)	0.0562 (13)	−0.0200 (12)	0.0114 (10)	−0.0147 (13)
C10	0.0591 (14)	0.0479 (14)	0.0699 (14)	0.0007 (10)	0.0244 (11)	0.0082 (11)
C11	0.0342 (10)	0.0456 (12)	0.0396 (10)	−0.0004 (8)	0.0061 (8)	0.0061 (9)
C12	0.0399 (11)	0.0559 (14)	0.0432 (11)	−0.0027 (9)	0.0119 (9)	−0.0034 (10)
C13	0.0656 (15)	0.0692 (17)	0.0730 (16)	0.0102 (13)	0.0182 (12)	−0.0128 (13)
C14	0.0497 (14)	0.099 (2)	0.0697 (16)	−0.0147 (13)	0.0286 (12)	−0.0141 (14)

C15	0.0684 (15)	0.0829 (18)	0.0412 (12)	−0.0026 (13)	0.0122 (11)	0.0033 (12)

Geometric parameters (Å, °)

Cl1—C2	1.7497 (18)	C8—C10	1.526 (3)
O1—C5	1.424 (2)	C8—C9	1.530 (3)
O1—C6	1.424 (2)	C8—C11	1.548 (3)
N1—C2	1.312 (2)	C9—H9A	0.9600
N1—C1	1.352 (2)	C9—H9B	0.9600
N2—C2	1.307 (2)	C9—H9C	0.9600
N2—C3	1.364 (2)	C10—H10A	0.9600
N3—C3	1.330 (2)	C10—H10B	0.9600
N3—C1	1.345 (2)	C10—H10C	0.9600
N4—C3	1.345 (2)	C11—C12	1.553 (2)
N4—C8	1.478 (2)	C11—H11A	0.9700
N4—H4	0.891 (9)	C11—H11B	0.9700
N5—C1	1.349 (2)	C12—C14	1.525 (3)
N5—C4	1.457 (2)	C12—C15	1.528 (3)
N5—C7	1.460 (2)	C12—C13	1.536 (3)
C4—C5	1.493 (3)	C13—H13A	0.9600
C4—H4A	0.9700	C13—H13B	0.9600
C4—H4B	0.9700	C13—H13C	0.9600
C5—H5A	0.9700	C14—H14A	0.9600
C5—H5B	0.9700	C14—H14B	0.9600
C6—C7	1.505 (3)	C14—H14C	0.9600
C6—H6A	0.9700	C15—H15A	0.9600
C6—H6B	0.9700	C15—H15B	0.9600
C7—H7A	0.9700	C15—H15C	0.9600
C7—H7B	0.9700		
C5—O1—C6	109.63 (14)	N4—C8—C11	106.76 (14)
C2—N1—C1	111.93 (15)	C10—C8—C11	115.32 (15)
C2—N2—C3	111.97 (15)	C9—C8—C11	111.43 (16)
C3—N3—C1	114.73 (15)	C8—C9—H9A	109.5
C3—N4—C8	128.20 (14)	C8—C9—H9B	109.5
C3—N4—H4	114.7 (13)	H9A—C9—H9B	109.5
C8—N4—H4	116.0 (13)	C8—C9—H9C	109.5
C1—N5—C4	122.23 (16)	H9A—C9—H9C	109.5
C1—N5—C7	122.48 (16)	H9B—C9—H9C	109.5
C4—N5—C7	114.44 (15)	C8—C10—H10A	109.5
N3—C1—N5	117.51 (16)	C8—C10—H10B	109.5
N3—C1—N1	125.37 (16)	H10A—C10—H10B	109.5
N5—C1—N1	117.12 (16)	C8—C10—H10C	109.5
N2—C2—N1	130.66 (17)	H10A—C10—H10C	109.5
N2—C2—Cl1	114.57 (14)	H10B—C10—H10C	109.5
N1—C2—Cl1	114.77 (14)	C8—C11—C12	123.87 (15)
N3—C3—N4	120.26 (16)	C8—C11—H11A	106.4
N3—C3—N2	125.28 (16)	C12—C11—H11A	106.4
N4—C3—N2	114.46 (15)	C8—C11—H11B	106.4
N5—C4—C5	109.48 (16)	C12—C11—H11B	106.4

N5—C4—H4A	109.8	H11A—C11—H11B	106.4
C5—C4—H4A	109.8	C14—C12—C15	109.72 (19)
N5—C4—H4B	109.8	C14—C12—C13	108.39 (18)
C5—C4—H4B	109.8	C15—C12—C13	107.16 (19)
H4A—C4—H4B	108.2	C14—C12—C11	114.21 (17)
O1—C5—C4	111.46 (18)	C15—C12—C11	111.30 (16)
O1—C5—H5A	109.3	C13—C12—C11	105.71 (16)
C4—C5—H5A	109.3	C12—C13—H13A	109.5
O1—C5—H5B	109.3	C12—C13—H13B	109.5
C4—C5—H5B	109.3	H13A—C13—H13B	109.5
H5A—C5—H5B	108.0	C12—C13—H13C	109.5
O1—C6—C7	111.63 (16)	H13A—C13—H13C	109.5
O1—C6—H6A	109.3	H13B—C13—H13C	109.5
C7—C6—H6A	109.3	C12—C14—H14A	109.5
O1—C6—H6B	109.3	C12—C14—H14B	109.5
C7—C6—H6B	109.3	H14A—C14—H14B	109.5
H6A—C6—H6B	108.0	C12—C14—H14C	109.5
N5—C7—C6	109.64 (17)	H14A—C14—H14C	109.5
N5—C7—H7A	109.7	H14B—C14—H14C	109.5
C6—C7—H7A	109.7	C12—C15—H15A	109.5
N5—C7—H7B	109.7	C12—C15—H15B	109.5
C6—C7—H7B	109.7	H15A—C15—H15B	109.5
H7A—C7—H7B	108.2	C12—C15—H15C	109.5
N4—C8—C10	109.89 (15)	H15A—C15—H15C	109.5
N4—C8—C9	104.31 (14)	H15B—C15—H15C	109.5
C10—C8—C9	108.56 (17)		
C3—N3—C1—N5	179.69 (17)	C1—N5—C4—C5	−139.1 (2)
C3—N3—C1—N1	0.3 (3)	C7—N5—C4—C5	51.2 (2)
C4—N5—C1—N3	1.9 (3)	C6—O1—C5—C4	61.8 (2)
C7—N5—C1—N3	170.86 (17)	N5—C4—C5—O1	−56.2 (2)
C4—N5—C1—N1	−178.61 (17)	C5—O1—C6—C7	−60.6 (2)
C7—N5—C1—N1	−9.7 (3)	C1—N5—C7—C6	140.15 (19)
C2—N1—C1—N3	1.7 (3)	C4—N5—C7—C6	−50.1 (2)
C2—N1—C1—N5	−177.68 (17)	O1—C6—C7—N5	54.1 (2)
C3—N2—C2—N1	0.7 (3)	C3—N4—C8—C10	55.6 (2)
C3—N2—C2—Cl1	−178.35 (12)	C3—N4—C8—C9	171.8 (2)
C1—N1—C2—N2	−2.3 (3)	C3—N4—C8—C11	−70.1 (2)
C1—N1—C2—Cl1	176.73 (13)	N4—C8—C11—C12	167.39 (15)
C1—N3—C3—N4	178.78 (17)	C10—C8—C11—C12	45.0 (2)
C1—N3—C3—N2	−2.2 (3)	C9—C8—C11—C12	−79.3 (2)
C8—N4—C3—N3	−3.7 (3)	C8—C11—C12—C14	48.2 (3)
C8—N4—C3—N2	177.19 (17)	C8—C11—C12—C15	−76.7 (2)
C2—N2—C3—N3	1.8 (3)	C8—C11—C12—C13	167.27 (18)
C2—N2—C3—N4	−179.15 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O1^{i}	0.891 (9)	2.197 (10)	3.076 (2)	168.9 (17)

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*+1/2.

Fig. 1

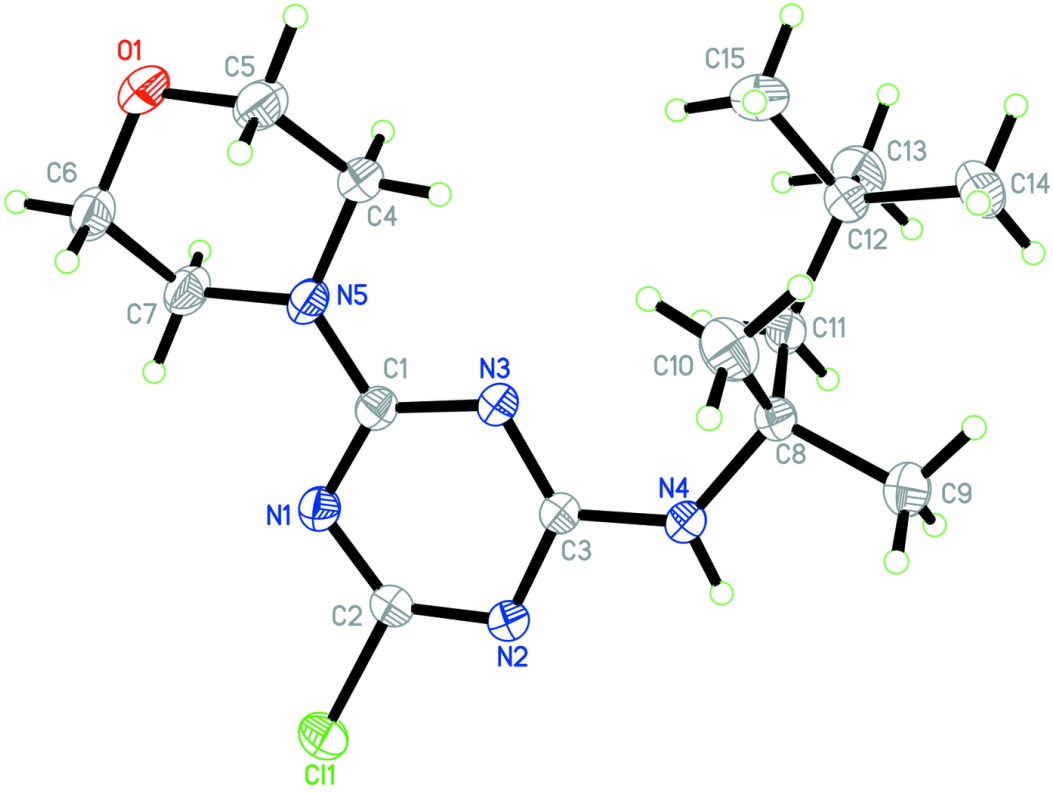

Fig. 2