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4,6-Dichloro-*N*-(4-methoxyphenyl)-1,3,5-triazin-2-amine

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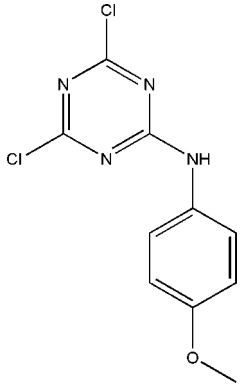
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.086; data-to-parameter ratio = 15.1.

In the title compound, $C_{10}H_8Cl_2N_4O$, the dihedral angle between the benzene and triazine rings is 29.57 (8)°. The crystal structure is stabilized by weak N—H···N interactions.

Related literature

For related literature, see: Manasek & Hrdlovik (1990); Mathias & Simanek (1994).



Experimental

Crystal data

$C_{10}H_8Cl_2N_4O$
M_r = 271.10
Monoclinic, $P2_1/c$
a = 7.6090 (16) Å
b = 20.635 (4) Å
c = 8.3081 (17) Å
β = 115.908 (3)°
V = 1173.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.881, T_{max} = 0.909
6560 measured reflections
2400 independent reflections
2016 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.087
S = 1.05
2400 reflections
159 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N3^{i}	0.886 (9)	2.646 (15)	3.377 (2)	140.6 (17)

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2630).

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supplementary materials

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Comment

2,4,6-Trichloro-1,3,5-triazine and its derivatives have been widely investigated, in fields such as drugs and light stabilizers (Mathias & Simanek, 1994; Manasek & Hrdlovik, 1990).

In the present paper, we describe the title compound, (I), (Fig. 1) which has been synthesied from 4-methoxylaniline and 2,4,6-trichloro-1,3,5-triazine. The bond lengths and angles in (I) are within normal ranges. The dihedral angle between the triazine ring and the benzene ring is 29.57 (8)°. The crystal packing is stabilized by weak N—H···N interactions (Table 1, Fig. 2).

Experimental

2,4,6-Trichloro-1,3,5-triazine (9.22 g, 0.05 mol), and 4-methoxylaniline (6.15 g, 0.05 mol) were added to 60 ml diethyl ether with stirring at 268 K for 2 h. A solution of Na_2CO_3 (2.76 g, 0.026 mol) in water (20 ml) was then added dropwise over 1 h. The reaction mixture was stirred at 273–278 K for a further 3 h. The precipitate was filtered off, and the acetone was evaporated under reduced pressure. The title compound (12.13 g) was obtained in a yield of 89.5%. Colourless blocks of (I) (m.p. 442–443 K) were obtained by slow evaporation of a mixture of ethyl acetate and ethanol.

Refinement

The N-bound H atom was located in a difference map and freely refined. All other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å or 0.96 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

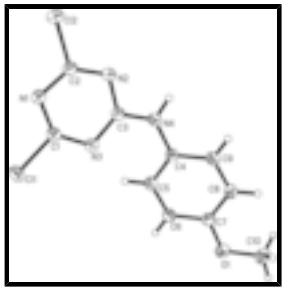

Fig. 1. A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

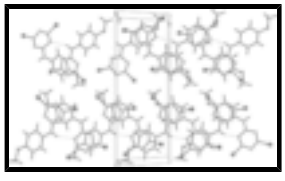

Fig. 2. The packing diagram of (I). Dashed lines indicate the N—H···N bonds.

4,6-dichloro-*N*-(4-methoxyphenyl)-1,3,5-triazin-2-amine

Crystal data

$C_{10}H_8Cl_2N_4O$	F_{000} = 552
M_r = 271.10	D_x = 1.535 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3751 reflections
a = 7.6090 (16) Å	θ = 3.0–26.4º
b = 20.635 (4) Å	μ = 0.54 mm^{-1}
c = 8.3081 (17) Å	T = 294 (2) K
β = 115.908 (3)º	Block, colourless
V = 1173.3 (4) Å^3	0.24 × 0.22 × 0.18 mm
Z = 4	

Data collection

Bruker SMART CCD diffractometer	2400 independent reflections
Radiation source: fine-focus sealed tube	2016 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.019
T = 294(2) K	θ_{max} = 26.4º
ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −9→9
T_{min} = 0.881, T_{max} = 0.909	k = −25→25
6560 measured reflections	l = −10→8

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.031	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.087	$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.3097P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
2400 reflections	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
159 parameters	$\Delta\rho_{min}$ = −0.36 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	0.48399 (7)	0.55535 (2)	0.67070 (6)	0.05566 (16)
Cl2	0.92722 (8)	0.62654 (2)	1.33265 (6)	0.06189 (18)
O1	−0.0465 (2)	0.89691 (6)	0.24801 (16)	0.0534 (3)
N1	0.6998 (2)	0.59535 (6)	0.99908 (18)	0.0410 (3)
N2	0.69411 (19)	0.70612 (6)	1.09037 (17)	0.0385 (3)
N3	0.49723 (19)	0.67436 (6)	0.78247 (17)	0.0382 (3)
N4	0.4870 (2)	0.77990 (7)	0.88813 (18)	0.0419 (3)
C1	0.5709 (2)	0.61570 (8)	0.8365 (2)	0.0377 (4)
C2	0.7551 (2)	0.64553 (8)	1.1163 (2)	0.0390 (4)
C3	0.5590 (2)	0.71897 (7)	0.9180 (2)	0.0349 (3)
C4	0.3479 (2)	0.80867 (8)	0.7242 (2)	0.0364 (3)
C5	0.1925 (2)	0.77394 (8)	0.5928 (2)	0.0404 (4)
H5	0.1749	0.7304	0.6103	0.048*
C6	0.0648 (2)	0.80499 (8)	0.4360 (2)	0.0416 (4)
H6	−0.0367	0.7817	0.3485	0.050*
C7	0.0880 (2)	0.87127 (8)	0.4087 (2)	0.0396 (4)
C8	0.2396 (3)	0.90674 (8)	0.5418 (2)	0.0420 (4)
H8	0.2544	0.9507	0.5263	0.050*
C9	0.3686 (2)	0.87490 (8)	0.6984 (2)	0.0407 (4)
H9	0.4696	0.8981	0.7866	0.049*
C10	−0.0269 (3)	0.96487 (9)	0.2171 (3)	0.0571 (5)
H10A	−0.0403	0.9902	0.3081	0.086*
H10B	−0.1267	0.9770	0.1018	0.086*
H10C	0.0993	0.9725	0.2209	0.086*
H4	0.534 (3)	0.8050 (9)	0.9843 (19)	0.057 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0621 (3)	0.0410 (2)	0.0471 (3)	−0.00029 (19)	0.0083 (2)	−0.01340 (18)
Cl2	0.0728 (3)	0.0490 (3)	0.0365 (2)	0.0036 (2)	−0.0015 (2)	0.00528 (18)
O1	0.0634 (8)	0.0446 (7)	0.0401 (7)	0.0091 (6)	0.0115 (6)	0.0059 (5)

N1	0.0439 (8)	0.0353 (7)	0.0363 (7)	0.0004 (6)	0.0106 (6)	0.0015 (5)
N2	0.0415 (7)	0.0371 (7)	0.0310 (6)	−0.0014 (6)	0.0103 (6)	−0.0010 (5)
N3	0.0434 (7)	0.0338 (7)	0.0320 (7)	0.0009 (6)	0.0116 (6)	−0.0009 (5)
N4	0.0507 (8)	0.0343 (7)	0.0323 (7)	0.0039 (6)	0.0103 (6)	−0.0047 (6)
C1	0.0384 (8)	0.0353 (8)	0.0360 (8)	−0.0034 (6)	0.0130 (7)	−0.0039 (6)
C2	0.0395 (8)	0.0395 (8)	0.0325 (8)	−0.0011 (7)	0.0108 (7)	0.0038 (6)
C3	0.0358 (8)	0.0351 (8)	0.0319 (7)	−0.0013 (6)	0.0132 (6)	0.0005 (6)
C4	0.0397 (8)	0.0341 (8)	0.0337 (8)	0.0052 (6)	0.0145 (7)	−0.0019 (6)
C5	0.0436 (9)	0.0315 (8)	0.0424 (9)	0.0003 (7)	0.0155 (7)	−0.0009 (6)
C6	0.0404 (9)	0.0375 (8)	0.0397 (8)	0.0019 (7)	0.0110 (7)	−0.0048 (7)
C7	0.0442 (9)	0.0389 (9)	0.0354 (8)	0.0089 (7)	0.0173 (7)	0.0012 (6)
C8	0.0518 (10)	0.0307 (8)	0.0437 (9)	0.0029 (7)	0.0211 (8)	−0.0004 (7)
C9	0.0443 (9)	0.0335 (8)	0.0406 (9)	−0.0008 (7)	0.0152 (7)	−0.0073 (7)
C10	0.0694 (13)	0.0470 (11)	0.0521 (11)	0.0145 (9)	0.0238 (10)	0.0148 (9)

Geometric parameters (Å, °)

Cl1—C1	1.7576 (16)	C4—C9	1.403 (2)
Cl2—C2	1.7443 (16)	C4—C5	1.406 (2)
O1—C7	1.384 (2)	C5—C6	1.395 (2)
O1—C10	1.445 (2)	C5—H5	0.9300
N1—C1	1.343 (2)	C6—C7	1.410 (2)
N1—C2	1.356 (2)	C6—H6	0.9300
N2—C2	1.318 (2)	C7—C8	1.405 (2)
N2—C3	1.3755 (19)	C8—C9	1.404 (2)
N3—C1	1.327 (2)	C8—H8	0.9300
N3—C3	1.369 (2)	C9—H9	0.9300
N4—C3	1.350 (2)	C10—H10A	0.9600
N4—C4	1.438 (2)	C10—H10B	0.9600
N4—H4	0.886 (9)	C10—H10C	0.9600
C7—O1—C10	116.87 (14)	C6—C5—H5	120.1
C1—N1—C2	110.15 (13)	C4—C5—H5	120.1
C2—N2—C3	113.75 (13)	C5—C6—C7	120.69 (15)
C1—N3—C3	113.13 (13)	C5—C6—H6	119.7
C3—N4—C4	129.06 (13)	C7—C6—H6	119.7
C3—N4—H4	114.0 (13)	O1—C7—C8	124.52 (15)
C4—N4—H4	117.0 (13)	O1—C7—C6	115.71 (14)
N3—C1—N1	129.79 (14)	C8—C7—C6	119.77 (15)
N3—C1—Cl1	114.99 (12)	C9—C8—C7	119.12 (15)
N1—C1—Cl1	115.21 (12)	C9—C8—H8	120.4
N2—C2—N1	129.06 (14)	C7—C8—H8	120.4
N2—C2—Cl2	115.70 (12)	C4—C9—C8	121.16 (15)
N1—C2—Cl2	115.23 (12)	C4—C9—H9	119.4
N4—C3—N3	120.61 (14)	C8—C9—H9	119.4
N4—C3—N2	115.44 (14)	O1—C10—H10A	109.5
N3—C3—N2	123.95 (14)	O1—C10—H10B	109.5
C9—C4—C5	119.36 (14)	H10A—C10—H10B	109.5
C9—C4—N4	117.49 (14)	O1—C10—H10C	109.5
C5—C4—N4	123.13 (14)	H10A—C10—H10C	109.5

C6—C5—C4	119.86 (15)	H10B—C10—H10C	109.5
C3—N3—C1—N1	−3.0 (3)	C3—N4—C4—C9	−147.41 (18)
C3—N3—C1—Cl1	176.03 (11)	C3—N4—C4—C5	33.8 (3)
C2—N1—C1—N3	−0.3 (3)	C9—C4—C5—C6	2.1 (2)
C2—N1—C1—Cl1	−179.33 (12)	N4—C4—C5—C6	−179.15 (15)
C3—N2—C2—N1	−1.7 (3)	C4—C5—C6—C7	−0.9 (3)
C3—N2—C2—Cl2	179.86 (11)	C10—O1—C7—C8	0.2 (2)
C1—N1—C2—N2	2.9 (3)	C10—O1—C7—C6	179.60 (16)
C1—N1—C2—Cl2	−178.59 (12)	C5—C6—C7—O1	179.70 (15)
C4—N4—C3—N3	−1.4 (3)	C5—C6—C7—C8	−0.9 (3)
C4—N4—C3—N2	178.88 (15)	O1—C7—C8—C9	−179.16 (16)
C1—N3—C3—N4	−175.35 (15)	C6—C7—C8—C9	1.5 (2)
C1—N3—C3—N2	4.4 (2)	C5—C4—C9—C8	−1.5 (2)
C2—N2—C3—N4	177.34 (15)	N4—C4—C9—C8	179.69 (15)
C2—N2—C3—N3	−2.4 (2)	C7—C8—C9—C4	−0.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···N3^{i}	0.886 (9)	2.646 (15)	3.377 (2)	140.6 (17)

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

Fig. 1

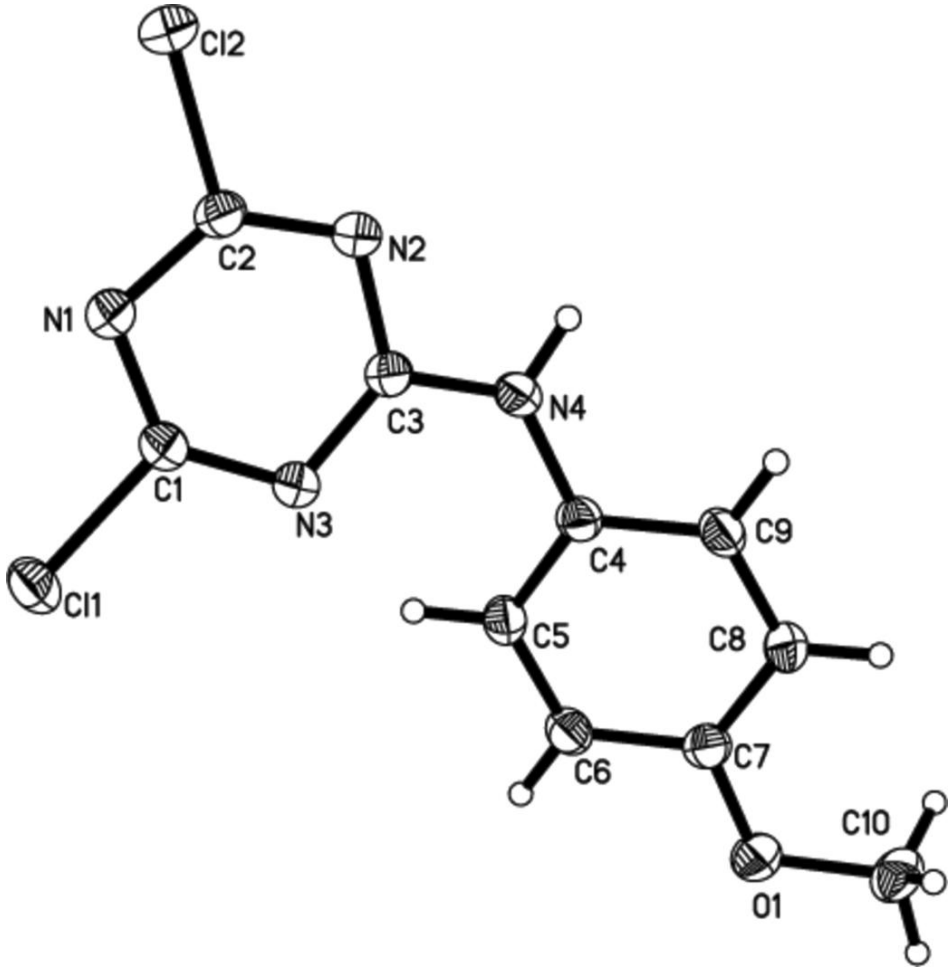

Fig. 2