

2,4-Dichloro-6-morpholino-1,3,5-triazine

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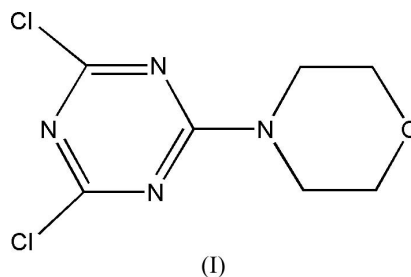
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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.029
 wR factor = 0.087
Data-to-parameter ratio = 18.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.This paper reports the synthesis of the title compound, $\text{C}_7\text{H}_8\text{Cl}_2\text{N}_4\text{O}$, and its crystal structure. The molecule possesses a mirror plane and the morpholine ring adopts a chair conformation.

Comment

2,4,6-Trichloro-1,3,5-triazine and its derivatives have been widely investigated, as a result of their importance as starting materials for many products, including active dyes, drugs and hindered amine light stabilizers (Borzatta & Carrozza, 1991; Manasek & Hrdlovik, 1990).

In the present paper, the title compound, $\text{C}_7\text{H}_8\text{Cl}_2\text{N}_4\text{O}$, (I), has been synthesized from 2,4,6-trichloro-1,3,5-triazine and morpholine in water. A crystallographic mirror plane at $y = \frac{1}{4}$ passes through atoms O1, N3, C2 and N1 (Fig. 1) The morpholine ring adopts a chair conformation. The bond lengths and angles (Table 1) are normal and compare well with those of a similar compound, *viz.* 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)morpholine (Fridman *et al.*, 2003), although the C–N bond connecting the two rings is somewhat longer [1.357 (4) Å] in the latter compound. The crystal structure is stabilized by intermolecular C–H...Cl interactions (Table 2 and Fig. 2).

Experimental

 Na_2CO_3 (23.02 g, 0.217 mol) and 2,4,6-trichloro-1,3,5-triazine (40.00 g, 0.217 mol) were added, with stirring, to water (200 ml) at 278 K. A solution of morpholine (18.52 g, 0.213 mol) in water (50 ml) was then added dropwise for 0.5 h. The reaction mixture was stirred at 273–278 K for a further 3 h. The precipitate was filtered off, washed with water and dried at 313 K. The title compound (39.03 g) in powder form was obtained in a yield of 76.5%. Suitable crystals were obtained by slow evaporation of a solution in a mixture of dichloromethane and cyclohexane (m.p. 425–428 K). ^1H NMR (CDCl_3 , p.p.m.): δ 3.76 (*t*, $J = 4.8$ Hz, 4H), 3.90 (*t*, $J = 4.8$ Hz, 4H); ^{13}C NMR (CDCl_3 , p.p.m.): δ 44.65 (4C), 66.56 (4C), 164.24 (2C), 170.59 (1C).Received 7 March 2005
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Crystal data

$C_7H_8Cl_2N_4O$
 $M_r = 235.07$
 Orthorhombic, $Pnma$
 $a = 9.6003$ (11) Å
 $b = 13.0545$ (15) Å
 $c = 7.6874$ (9) Å
 $V = 963.44$ (19) Å³
 $Z = 4$
 $D_x = 1.621$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 2735 reflections
 $\theta = 2.6$ – 28.1°
 $\mu = 0.65$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.26 \times 0.24 \times 0.20$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.833$, $T_{\max} = 0.879$
 6195 measured reflections

1278 independent reflections
 1056 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 28.6^\circ$
 $h = -11 \rightarrow 12$
 $k = -17 \rightarrow 14$
 $l = -10 \rightarrow 9$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.087$
 $S = 1.08$
 1278 reflections
 71 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.0972P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.032 (3)

Table 1

Selected geometric parameters (Å, °).

C1—C1	1.7318 (12)	N2—C2	1.3573 (12)
O1—C4	1.4168 (15)	N3—C2	1.331 (2)
N1—C1	1.3254 (14)	N3—C3	1.4604 (14)
N2—C1	1.3028 (15)	C3—C4	1.5083 (19)
C4 ⁱ —O1—C4	110.91 (14)	N2—C1—C1	115.79 (9)
C2—N3—C3	123.35 (7)	N1—C1—C1	114.62 (9)
C3—N3—C3 ⁱ	113.22 (14)	N3—C2—N2	118.09 (7)

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C3—H3B \cdots Cl1 ⁱⁱ	0.97	2.91	3.736 (2)	144

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

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve

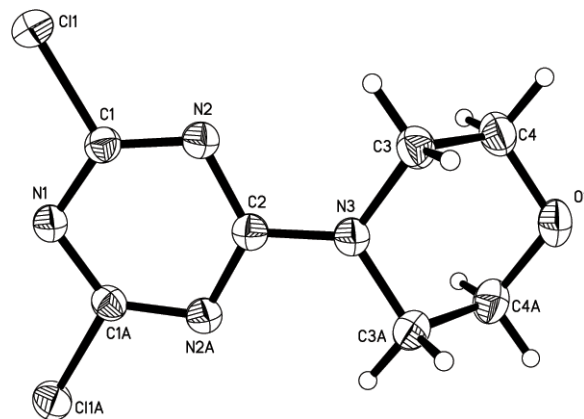


Figure 1

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. The suffix *A* indicates the symmetry position $(x, \frac{1}{2} - y, z)$.

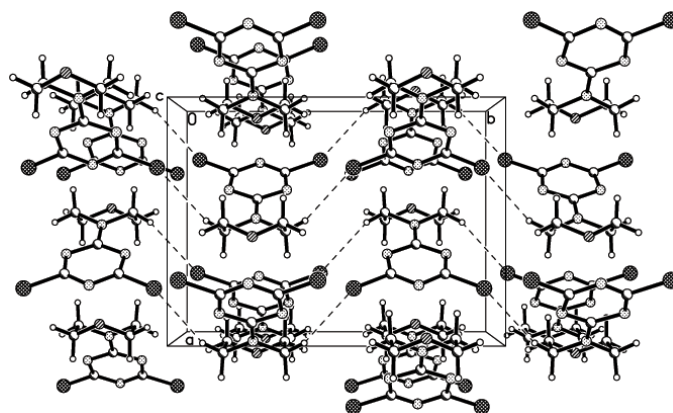


Figure 2

The crystal structure of (I), viewed along the *c* axis. Dashed lines indicate hydrogen-bond interactions.

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.

References

- Borzatta, V. & Carrozza, P. (1991). European Patent EP 0462 069.
 Bruker (1997). SMART, SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.
 Fridman, N., Kapon, M. & Kaftory, M. (2003). Acta Cryst. C59, o687–o689.
 Manasek, Z. & Hrdlovik, P. (1990). European Patent EP 0377 324.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.