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2-Chloro-4-methoxy-6-methyl-1,3,5-triazine

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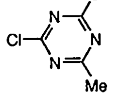
Abstract

Molecules of the title compound, $C_5H_6ClN_3O$, lie on crystallographic mirror planes. Within these planes molecules are linked into zigzag chains by N···Cl intermolecular interactions of 3.139 (3) Å. Molecules in adjacent planes are rotated by 180° but lie almost directly above one another; the interplanar spacing is 3.283 (2) Å. Ring angles at the N atoms are substantially more acute than those at the C atoms.

Comment

The 3-methoxy-5-methyl-1,3,5-triazine system is found in a number of commercially important sulfonylurea herbicides, such as Metsulfuron Methyl and Thifensulfuron (Cremlyn, 1991). Only one crystal structure containing this system has been reported (Lotz, Kiel & Gattow, 1991), and this is of poor quality. The title compound, (I), is also relevant to the recent interest in chlorotriazines, such as cyanuric chloride (Pascal & Ho, 1992; Maginn, Compton, Harding, Brennan & Docherty, 1993), which show N···Cl intermolecular contacts.



(I)

The molecule possesses crystallographic C_s symmetry. The ring C—N bond lengths vary from 1.320 (3) to 1.346 (3) Å and the mean bond length of 1.336 (7) Å is identical to those in aminochloromethoxy-1,3,5-triazines (Glowka & Iwanicka, 1991). As found in other triazines (Glowka & Iwanicka, 1991, and references therein), the ring angles at the N atoms are substantially more acute [112.3 (2)–114.6 (2)°], and those at the C atoms [124.7 (2)–128.4 (2)°] more obtuse, than the ideal value of 120°. A correlation between the C—Cl bond distance and the corresponding endocyclic N—C—N bond angle has been suggested (Glowka & Iwanicka, 1991) and our data fit this relationship.

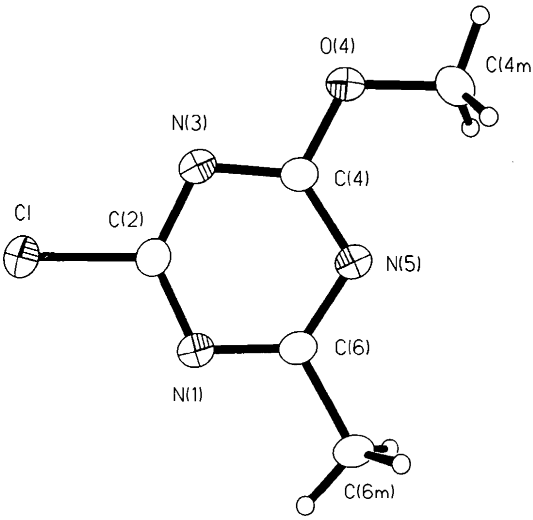


Fig. 1. A view of the molecule with the atom-numbering scheme. Except for two symmetry-related H atoms in each methyl group, all atoms lie on a crystallographic mirror plane. Displacement ellipsoids are plotted at the 30% probability level.

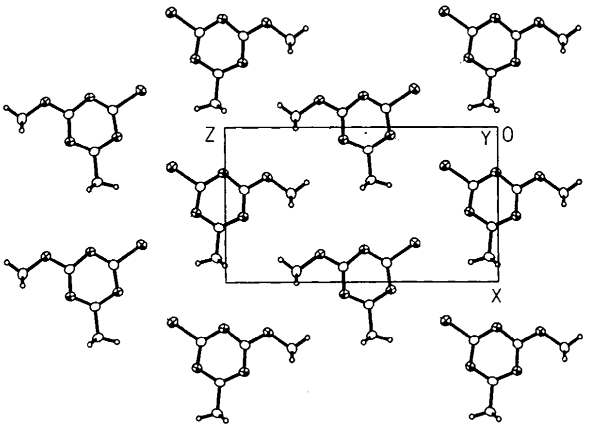


Fig. 2. A view of a plane of molecules at $y = \frac{1}{4}$ linked into zigzag chains by N···Cl intermolecular interactions of 3.139 (3) Å. Molecules in adjacent planes are rotated through 180° by the symmetry operation $(1 - x, \frac{1}{2} + y, -z)$ but lie almost directly above each other with an interplanar spacing of 3.283 (2) Å.

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Molecules lying on crystallographic mirror planes related by the *a*-glide operation ($\frac{1}{2} + x$, y, $\frac{1}{2} - z$) are linked into zigzag chains by N···Cl intermolecular contacts of 3.139 (3) Å, which are shorter than the sum of the van der Waals radii for N and Cl (3.30 Å). This is similar to the situation in cyanuric chloride where the N···Cl contact distance is 3.10 Å (Maginn, Compton, Harding, Brennan & Docherty, 1993). Molecules in adjacent planes are rotated through 180° by the symmetry operation ($1 - x$, $\frac{1}{2} + y$, $-z$) but lie almost directly above each other with interplanar spacings of 3.283 (2) Å. The corresponding spacing in cyanuric chloride is 3.26 Å (Pascal & Ho, 1992).

Experimental

The title compound was synthesized in two steps by the literature method (Kobe, Stanovnik & Tisler, 1970) which involved the sequential displacement of chlorides from cyanuric chloride using, initially, methyl magnesium chloride in diethyl ether–benzene solution, followed by displacement by the methoxide ion in methanol–water solution.

Crystal data

$C_5H_6ClN_3O$
$M_r = 159.58$
Orthorhombic
Pnma
$a = 7.6358\ (12)$ Å
$b = 6.5671\ (15)$ Å
$c = 13.746\ (2)$ Å
$V = 689.3\ (2)$ Å^3
$Z = 4$
$D_x = 1.538$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 55 reflections
$\theta = 14–16°$
$\mu = 0.482$ mm^{-1}
$T = 293\ (2)$ K
Tablet
0.54 × 0.31 × 0.16 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer
ω–2θ scans
Absorption correction: none
2007 measured reflections
1089 independent reflections
842 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.037$
$\theta_{\max} = 30.02°$
$h = -10 \rightarrow 10$
$k = -5 \rightarrow 9$
$l = -19 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity variation: <0.5%

Refinement

Refinement on F^2
$R(F) = 0.0380$
$wR(F^2) = 0.1183$
$S = 1.040$
1079 reflections
62 parameters
H atom U's refined with a common $U_{\rm iso}$
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.3731P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.008$
$\Delta\rho_{\max} = 0.552$ e Å^{-3}
$\Delta\rho_{\min} = -0.497$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL* (Sheldrick, 1994). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1992).

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$
N1	0.5517 (2)	1/4	0.10594 (12)	0.0237 (4)
C2	0.3799 (3)	1/4	0.09075 (14)	0.0221 (4)
N3	0.2952 (2)	1/4	0.00709 (13)	0.0228 (4)
C4	0.4043 (3)	1/4	−0.06966 (14)	0.0212 (4)
N5	0.5792 (2)	1/4	−0.06643 (13)	0.0227 (4)
C6	0.6468 (3)	1/4	0.02351 (14)	0.0218 (4)
Cl	0.25161 (7)	1/4	0.19452 (4)	0.0299 (2)
O4	0.3227 (2)	1/4	−0.15445 (10)	0.0278 (4)
C4*m*	0.4307 (3)	1/4	−0.2411 (2)	0.0319 (5)
C6*m*	0.8447 (3)	1/4	0.0334 (2)	0.0279 (5)

Table 2. *Selected geometric parameters* (Å, °)

N1—C2	1.329 (3)	C4—O4	1.322 (2)
N1—C6	1.346 (3)	C4—N5	1.336 (2)
C2—N3	1.320 (3)	N5—C6	1.340 (3)
C2—Cl	1.730 (2)	C6—C6*m*	1.517 (3)
N3—C4	1.345 (3)	O4—C4*m*	1.448 (3)
C2—N1—C6	113.6 (2)	N5—C4—N3	126.4 (2)
N3—C2—N1	128.4 (2)	C4—N5—C6	114.6 (2)
N3—C2—Cl	116.2 (2)	N5—C6—N1	124.7 (2)
N1—C2—Cl	115.4 (2)	N5—C6—C6*m*	117.8 (2)
C2—N3—C4	112.3 (2)	N1—C6—C6*m*	117.5 (2)
O4—C4—N5	120.1 (2)	C4—O4—C4*m*	117.1 (2)
O4—C4—N3	113.5 (2)		

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1106). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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