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Chlorometronidazole

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.128
Data-to-parameter ratio = 16.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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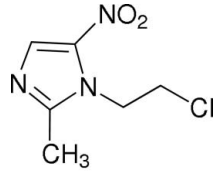
In the title compound, 1-(2-chloroethyl)-2-methyl-5-nitro-1*H*-imidazole, $C_6H_8ClN_3O_2$, the dihedral angle between the imidazole ring and the attached nitro group is 6.5 (1)°.

Comment

Metronidazole is a widely used antibacterial medicine (Credito *et al.*, 2000; Mendz & Mégraud, 2002). As a result of its toxicity, its structural modification has recently been an active research topic (Alcalde *et al.*, 1984). In this paper, we report the structure of chlorometronidazole, (I), in which the hydroxyl group of the metronidazole has been replaced by a chloride group.



(I)

The molecular structure of (I) is shown in Fig. 1. In each molecule, the imidazole ring is a well defined plane with an average deviation of 0.002 (1) Å. The nitro N atom lies 0.060 (1) Å above the plane; the two other groups attached to the ring are located on the opposite side of the plane, with C4

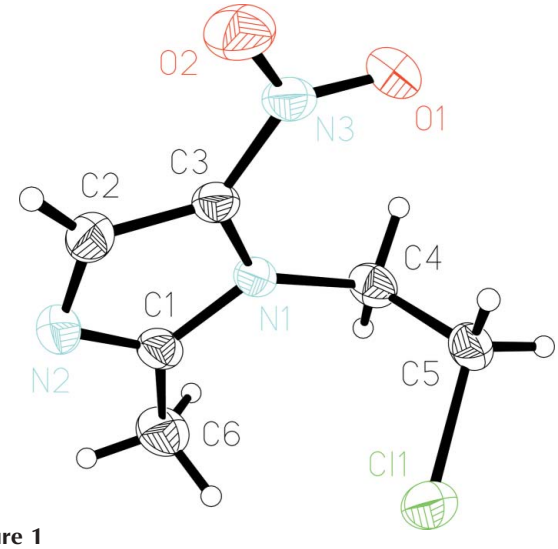


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids for the non-H atoms and the atom-numbering scheme.

0.144 (1) and C6 0.004 (1) Å from the ring. The dihedral angle between the imidazole ring and the nitro plane is 6.5 (1)°.

Experimental

To a pyridine solution (5 ml) of metronidazole (171 mg, 1 mmol) was added SO_2Cl_2 (149 mg, 1.1 mmol) and Na_2CO_3. This mixture was stirred for 3 h at 353 K. The solvents and excess SO_2Cl_2 were then removed. The residue was recrystallized from chloroform, large crystals suitable for X-ray crystal structure determination being formed in 12 h. These were collected by filtration, washed with chloroform and diethyl ether, and dried in a vacuum desiccator using $CaCl_2$ (yield: 98%). Elemental analysis found: C 37.9, H 4.3, N 22.2%. Calculated for $C_6H_8ClN_3O_2$: C 38.0, H 4.2, N 22.2%. ^{1}H NMR (500 MHz, DMSO-d_6): δ 7.95 (1H, *s*, broad, $-CH-$), 4.55 (2H, *t*, J = 7.2 Hz, $-CH_2-$), 3.35 (2H, *t*, J = 7.2 Hz, $-CH_2-$), 2.26 (3H, *s*, $-CH_3-$).

Crystal data

$C_6H_8ClN_3O_2$	D_x = 1.517 Mg m^{-3}
M_r = 189.60	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1562 reflections
a = 12.098 (14) Å	θ = 6.3–27.4°
b = 11.007 (13) Å	μ = 0.42 mm^{-1}
c = 6.295 (7) Å	T = 298 (2) K
β = 97.886 (18)°	Prism, colorless
V = 830.3 (16) Å^3	0.38 × 0.35 × 0.30 mm
Z = 4	

Data collection

Siemens SMART CCD area-detector diffractometer	1778 independent reflections
	1293 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.032
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 27.0°
	$h = -15 \rightarrow 14$
T_{min} = 0.850, T_{max} = 0.890	$k = -14 \rightarrow 14$
4602 measured reflections	$l = -6 \rightarrow 7$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.045	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.128	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
1778 reflections	Extinction correction: *SHELXL97*
111 parameters	Extinction coefficient: 0.039 (6)
H-atom parameters constrained	

All H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.96 Å. They were treated as riding atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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