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(*Z*)-5-Chlorofuran-2-carbaldehyde oxime

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The molecule of the title compound, $C_5H_4ClNO_2$, is essentially planar, with normal values for the bond lengths and angles. In the crystal structure, intermolecular O—H···N hydrogen bonds link the molecules into zigzag chains extending along the *b* axis.

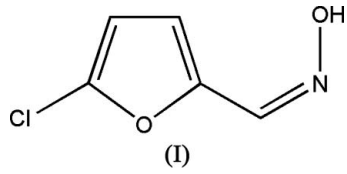
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.136
Data-to-parameter ratio = 12.9

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

Comment

The title compound, (I) (Fig. 1), is an important intermediate in organic synthesis, in particular, in producing isoxazole (Sharghi & Sarvari, 2001). Here, we report the crystal structure of (I).



(I)

In compound (I), the bond lengths and angles of the furan ring are normal and comparable with those observed in analogous published structures (Matsuoka *et al.*, 1991; Olszak *et al.*, 1995). The molecular skeleton of (I) is essentially planar, the largest deviation from the mean plane being 0.031 (2) Å for atom Cl1.

In the crystal structure (Fig. 2), intermolecular O—H···N hydrogen bonds (Table 1) link the molecules into zigzag chains extending along the *b* axis.

Experimental

To a solution of (*Z*)-furan-2-carbaldehyde oxime (20 mmol) in dimethylformamide (10 ml) was added *N*-chlorosuccinimide (NCS; 5 mmol) in one portion. Further NCS (15 mmol) was added in small

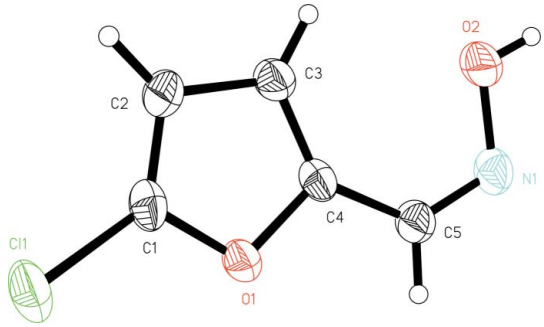


Figure 1
A view of the molecular structure of (I), with displacement ellipsoids drawn at the 40% probability level.

portions while keeping the temperature below 308 K. The mixture was stirred at room temperature for 1 h, poured into water and extracted with diethyl ether. The organic phase was washed with brine and dried over magnesium sulfate, and the solvent was removed. The residue was chromatographed on silica gel (ethyl acetate–petroleum 1:3) to give the title compound, (I), in 51% yield (Himo *et al.*, 2005). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution of (I) in a hexane–dichloromethane mixture (1:1 *v*/*v*) at room temperature over a period of one week.

Crystal data

$C_5H_4ClNO_2$
M_r = 145.54
Monoclinic, $P2_1/n$
a = 9.394 (3) Å
b = 5.0879 (15) Å
c = 12.870 (4) Å
β = 102.301 (4)°
V = 601.0 (3) Å^3
Z = 4
D_x = 1.608 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 298 (2) K
Block, colourless
0.41 × 0.34 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.807, T_{max} = 0.875
2747 measured reflections
1055 independent reflections
910 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.136
S = 1.09
1055 reflections
82 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0777P)^2 + 0.2968P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N1^{i}	0.82	2.05	2.823 (3)	156

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

All H atoms were placed in calculated positions, with C—H = 0.93 Å and O—H = 0.82 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O).

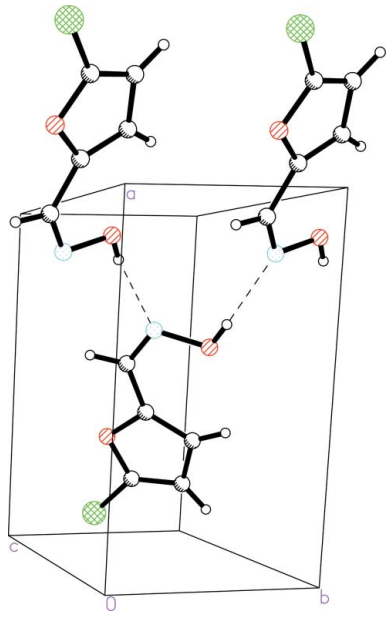


Figure 2
Part of the crystal packing for (I), showing the hydrogen-bonded (dashed lines) chain.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

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