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3-Chloro-4-hydroxyfuran-2(5*H*)-one

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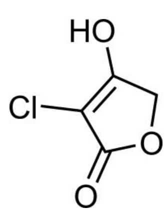
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.026; wR factor = 0.067; data-to-parameter ratio = 10.2.

In the title compound, $C_4H_3ClO_3$, molecules are linked *via* O—H···O hydrogen bonds into an infinite chain with graph-set motif $C(6)$ along the c axis.

Related literature

4-Hydroxy-5*H*-furan-2-one (tetronic acid) forms a subclass of β-hydroxybutenolides with a generic structure, see: Haynes & Plimmer (1960). A great number of these compounds and their metabolites are found in many natural products and exhibit a wide array of biological properties, see: Sodeoka *et al.* (2001). For related structures, see: Ma *et al.* (2004). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_4H_3ClO_3$
M_r = 134.51
Orthorhombic, $Pnma$
a = 12.0437 (6) Å
b = 6.5453 (4) Å
c = 6.3886 (4) Å
V = 503.61 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 298 K
0.50 × 0.50 × 0.30 mm

Data collection

Oxford Gemini S Ultra diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)
T_{min} = 0.736, T_{max} = 0.828
1932 measured reflections
531 independent reflections
500 reflections with $I > 2\sigma(I)$
R_{int} = 0.012

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.067
S = 1.17
531 reflections
52 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.80 (3)	1.85 (3)	2.647 (2)	171 (3)

Symmetry code: (i) $x, y, z-1$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97* and *publCIF* (Westrip, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2226).

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supplementary materials

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3-Chloro-4-hydroxyfuran-2(5*H*)-one

N. Zhang, Z.-Y. Wu, S.-Y. Xie, R.-B. Huang and L.-S. Zheng

Comment

4-hydroxy-5*H*-furan-2-one (Tetronic acid) form a subclass of β-hydroxybutenolides with the generic structure (Haynes & Plimmer, 1960). The best known members of this family are vitamin C (ascorbic acid) and pennicillic acid. A great number of these compounds and their metabolites are found in many natural products, which exhibit a wide array of biological properties (Sodeoka *et al.*, 2001). In the present study, the title comound (I) has been determined as product of double-molecular ring closure of monochloroacetic acid which is halo-substituted tetronic acid.

The molecular structure is depicted in Fig. 1. Bond lengths and angles are in good agreement with previous reported for similar compounds (Ma *et al.*, 2004). The crystal structure is stabilized by O—H···O hydrogen bonding and the molecules are linked in an infinite chain along the c axis, with graph-set motifs C(6) through O— H··· O hydrogen bonds (Bernstein *et al., 1995)* (Fig. 2, Table 1).

Experimental

All reagents and solvents were used as obtained commercially without further purification. To a stirred solution of monochloroacetic acid(2 mmol, 137μ*L*) in 5 mL dry THF is added sodium(1 mmol, 23 mg) under N_2. After the solution has been stirred at room temperature for 24 h, the resulting pale yellow solution was kept in darkness for four days, yellow well formed block-shaped crystals were obtained.

Refinement

The aromatic H atoms were generated geometrically (C—H 0.93 Å) and were allowed to ride on their parent atoms in the riding model approximations, with their temperature factors set to 1.2 times those of the parent atoms. The position and U_{eq} of the hydroxyl H atom were refined with O—H distance restrained to 0.85 Å.

Figures

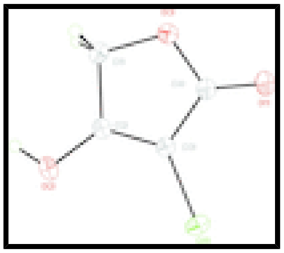

Fig. 1. A view of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability label and H atoms are shown as small spheres of arbitrary radii.

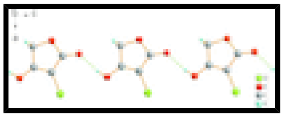

Fig. 2. Partial packing view showing the O—H···O interactions (dashed lines) and the formation of a chain parallel to the c axis.

3-Chloro-4-hydroxyfuran-2(5*H*)-one

Crystal data

$C_4H_3Cl_1O_3$	$F_{000} = 272$
$M_r = 134.51$	$D_x = 1.774$ Mg m^{-3}
Orthorhombic, *Pnma*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 1627 reflections
a = 12.0437 (6) Å	θ = 3.1–28.9º
b = 6.5453 (4) Å	μ = 0.65 mm^{-1}
c = 6.3886 (4) Å	*T* = 298 K
V = 503.61 (5) Å^3	Block, yellow
Z = 4	0.50 × 0.50 × 0.30 mm

Data collection

Oxford Gemini S Ultra diffractometer	531 independent reflections
Radiation source: fine-focus sealed tube	500 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.012$
Detector resolution: 16.1903 pixels mm^{-1}	$\theta_{max} = 26.0$º
T = 298 K	$\theta_{min} = 3.4$º
ω scans	$h = -14\rightarrow14$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008)	$k = -7\rightarrow8$
$T_{min} = 0.736$, $T_{max} = 0.828$	$l = -7\rightarrow7$
1932 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.026$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.067$	$w = 1/[\sigma^2(F_o^2) + (0.0374P)^2 + 0.1215P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.17	$(\Delta/\sigma)_{max} < 0.001$
531 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
52 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > \sigma(F^2)$ 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}	Occ. (<1)
Cl1	0.34253 (4)	0.7500	0.60829 (9)	0.0400 (2)	
O1	0.10530 (15)	0.7500	0.8246 (3)	0.0531 (5)	
O2	0.24683 (15)	0.7500	0.1417 (2)	0.0409 (4)	
O3	0.02339 (12)	0.7500	0.5138 (2)	0.0410 (4)	
C1	0.05689 (17)	0.7500	0.2974 (3)	0.0348 (5)	
H1A	0.0295	0.8706	0.2259	0.042*	0.50
H1B	0.0295	0.6294	0.2259	0.042*	0.50
C2	0.18096 (17)	0.7500	0.3064 (3)	0.0288 (4)	
C3	0.21185 (17)	0.7500	0.5069 (3)	0.0289 (4)	
C4	0.11471 (18)	0.7500	0.6361 (3)	0.0335 (5)	
H2	0.210 (3)	0.7500	0.037 (5)	0.059 (9)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0274 (3)	0.0523 (4)	0.0403 (3)	0.000	−0.0084 (2)	0.000
O1	0.0421 (9)	0.0940 (14)	0.0231 (8)	0.000	0.0033 (6)	0.000
O2	0.0376 (8)	0.0614 (11)	0.0238 (8)	0.000	0.0062 (7)	0.000
O3	0.0260 (8)	0.0679 (10)	0.0292 (9)	0.000	0.0011 (6)	0.000
C1	0.0308 (10)	0.0487 (12)	0.0249 (11)	0.000	−0.0042 (8)	0.000
C2	0.0284 (10)	0.0337 (10)	0.0241 (10)	0.000	0.0019 (8)	0.000
C3	0.0251 (10)	0.0355 (10)	0.0261 (11)	0.000	−0.0006 (7)	0.000
C4	0.0299 (10)	0.0460 (12)	0.0247 (11)	0.000	0.0006 (8)	0.000

Geometric parameters (Å, °)

Cl1—C3	1.702 (2)	C1—C2	1.495 (3)
O1—C4	1.210 (3)	C1—H1A	0.9700
O2—C2	1.318 (2)	C1—H1B	0.9700
O2—H2	0.80 (3)	C2—C3	1.334 (3)
O3—C4	1.349 (3)	C3—C4	1.431 (3)
O3—C1	1.440 (3)		

C2—O2—H2	109 (2)	O2—C2—C1	124.82 (18)
C4—O3—C1	109.11 (16)	C3—C2—C1	108.38 (17)
O3—C1—C2	104.08 (16)	C2—C3—C4	109.00 (19)
O3—C1—H1A	110.9	C2—C3—Cl1	128.55 (17)
C2—C1—H1A	110.9	C4—C3—Cl1	122.45 (15)
O3—C1—H1B	110.9	O1—C4—O3	120.0 (2)
C2—C1—H1B	110.9	O1—C4—C3	130.6 (2)
H1A—C1—H1B	109.0	O3—C4—C3	109.43 (17)
O2—C2—C3	126.8 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.80 (3)	1.85 (3)	2.647 (2)	171 (3)

Symmetry codes: (i) *x*, *y*, *z*−1.

Fig. 1

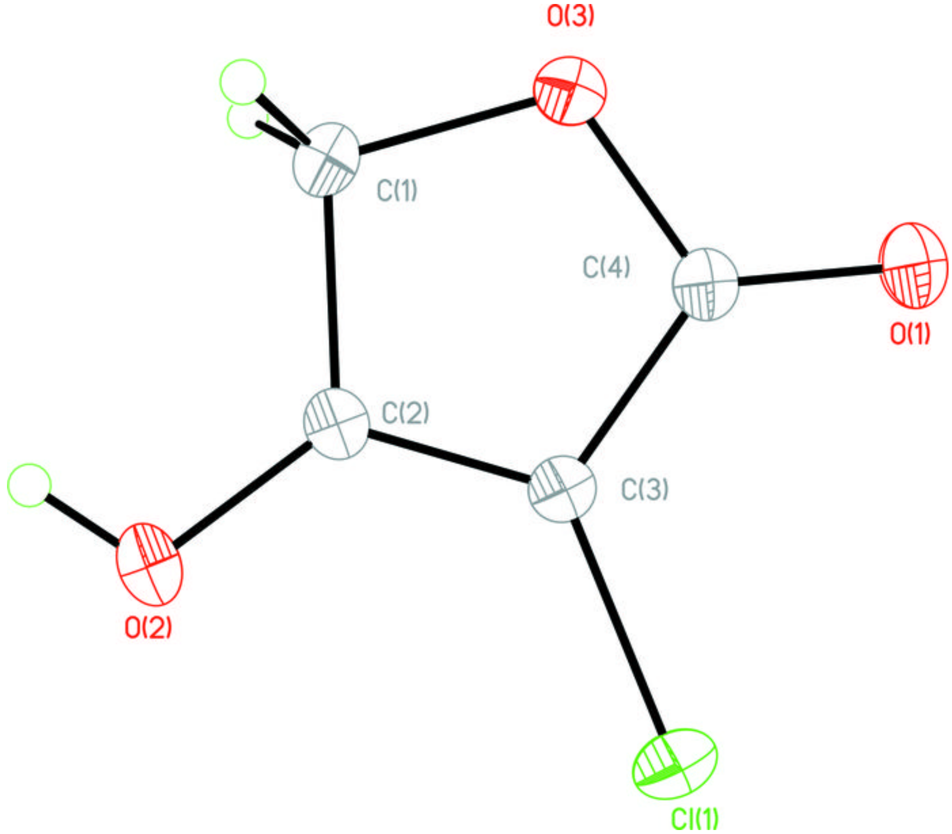

Fig. 2