Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 2-[(4-chloro-2-methoxy-5-oxo-2,5-dihydrofuran-3-yl)amino]acetate

Yang-Qing Mo, Zhao-Yang Wang,* Jian-Hua Fu and Hua-Cai Fang

School of Chemistry and Environment, South China Normal University, Guangzhou 510006, People's Republic of China
Correspondence e-mail: wangwangzhaoyang@tom.com

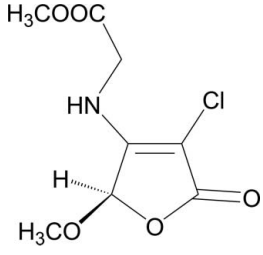
Received 17 June 2010; accepted 23 June 2010

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.088; data-to-parameter ratio = 12.3.

The title compound, $C_8H_{10}ClNO_5$, was obtained *via* a tandem Michael addition–elimination reaction of 3,4-dichloro-5-methoxyfuran-2(5*H*)-one and glycine methyl ester in the presence of triethylamine. The molecular structure contains an approximately planar [maximum atomic deviation = 0.010 (2) Å] five-membered furanone ring. The crystal packing is stabilized by intermolecular N—H···O and weak C—H···O hydrogen bonding.

Related literature

For biologicallly active 4-amino-2(5*H*)-furanones, see: Kimura *et al.* (2000); Tanoury *et al.* (2008). For related furanone structures, see: Song *et al.* (2009*b*); Li *et al.* (2009). For the synthesis, see: Song *et al.* (2009*a*).



Experimental

Crystal data

$C_8H_{10}ClNO_5$ M_r = 235.62
Monoclinic, $P2_1/c$
a = 5.1366 (10) Å
b = 9.8316 (19) Å
c = 20.685 (4) Å
β = 102.532 (4)°
V = 1019.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 296 K
0.21 × 0.21 × 0.21 mm

Data collection

Bruker APEXII CCD diffractometer
3333 measured reflections
1714 independent reflections
1221 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.088
S = 1.05
1714 reflections
139 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.06	2.911 (3)	171
C6—H6B···O5^{ii}	0.97	2.42	3.366 (3)	166

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The work was supported by the National Natural Science Foundation of China (grant No. 20772035) and the Natural Science Foundation of Guangdong Province, China (grant No. 5300082).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2785).

References

Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Kimura, Y., Mizuno, T., Kawano, T., Okada, K. & Shimad, A. (2000). *Phytochemistry*, **53**, 829–831.
Li, Z., Song, X., Wang, Z. & Yang, K. (2009). *Acta Cryst.* E**65**, o1030.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Song, X.-M., Li, Z.-Y., Wang, Z.-Y. & Fu, J.-H. (2009*b*). *Acta Cryst.* E**65**, o1838.
Song, X.-M., Wang, Z.-Y., Fu, J.-H., Li, J. & -, X. (2009*a*). *J. S. China Norm. Univ. (Nat. Sci. Ed.)*, **4**, 75–80.
Tanoury, G. J., Chen, M. Z., Dong, Y., Forslund, R. E. & Magdziak, D. (2008). *Org. Lett.* **10**, 185–188.

supporting information

Acta Cryst. (2010). E**66**, o1857 [doi:10.1107/S160053681002461X]

Methyl 2-[(4-chloro-2-methoxy-5-oxo-2,5-dihydrofuran-3-yl)amino]acetate

Yang-Qing Mo, Zhao-Yang Wang, Jian-Hua Fu and Hua-Cai Fang

S1. Comment

2(5*H*)-Furanone is a simplest sub-unit of a large class of five membered heterocyclic carbonyl compounds. At the same time, 4-amino-2(5*H*)-furanone is an attractive moiety in chemical, pharmaceutical and agrochemical research. Many 4-amino-2(5*H*)-furanones have been patented as prodrugs or insecticides and herbicides (Kimura *et al.*, 2000; Tanoury *et al.*, 2008). Attracted by versatile 4-amino-2(5*H*)-furanones, we synthesized the title compound with 3,4-dichloro-5-methoxyfuran-2(5*H*)-one and glycine methylester in the presence of triethylamine *via* the tandem Michael addition-elimination reaction. With 2(5*H*)-furanone moiety and polyfunctional groups (carboxyl, amino, halo), the title compound is expected to be a biologically active product.

The structure of the title compound (I) is illustrated in Fig. 1. The title compound contains a five-membered furanone ring and a methoxy connected each other *via* C4—O3—C5 ether bond. The furanone ring is approximately planar, smilar to that found in a related compound (Song *et al.*, 2009*b*). Additionally, the molecules are linked by intermolecular hydrogen bonds of N—H···O and C—H···O (Table 1).

S2. Experimental

The precursor 3,4-dichloro-5-methoxyfuran-2(5*H*)-furanone was prepared according to the literature procedure (Song *et al.*, 2009a). After the mixture of glycine methylester (3.0 mmol) and triethylamine (2.8 ml) was dissolved in absolute tetrahydrofuran under nitrogen atmosphere, dichloromethane solution of 3,4-chloro-5-methoxyfuran-2(5*H*)-furanone (2.0 mmol) was added. The reaction was carried out under the stirring at room temperature for 55 h. Once the reaction was complete, the solvents were removed under reduced pressure. The residual solid was dissolved in dichloromethane. Then the combined organic layers from extraction were concentrated under reduced pressure, and the crude product was purified by silica gel column chromatography with the gradient mixture of petroleum ether and ethyl acetate to give the product yielding (I) 0.2372 g (50.6%).

S3. Refinement

H atoms were positioned in calculated positions with C—H = 0.96-0.98 Å and N—H = 0.86 Å, and were refined using a riding model with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C,N)$ for the other H atoms.

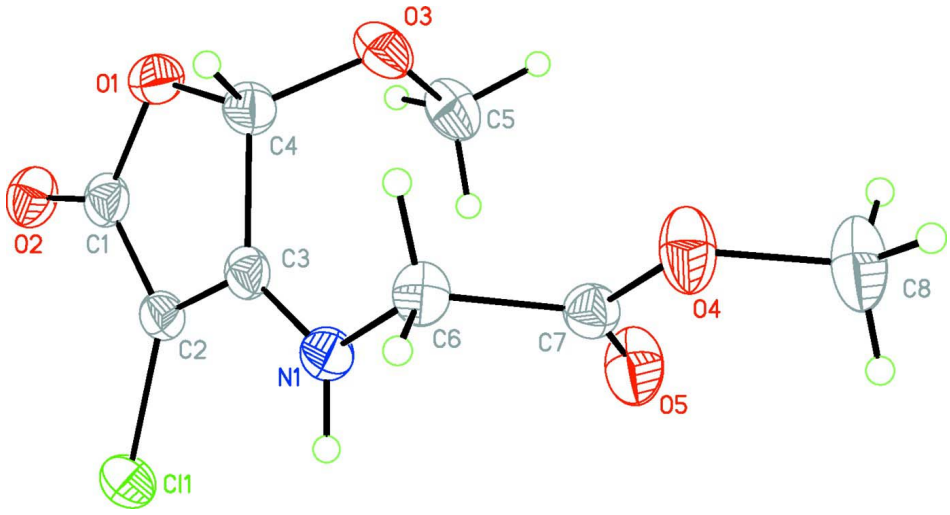


Figure 1

View of the title compound showing the atom-labelling scheme. Ellipsoids are drawn at the 30% probability level.

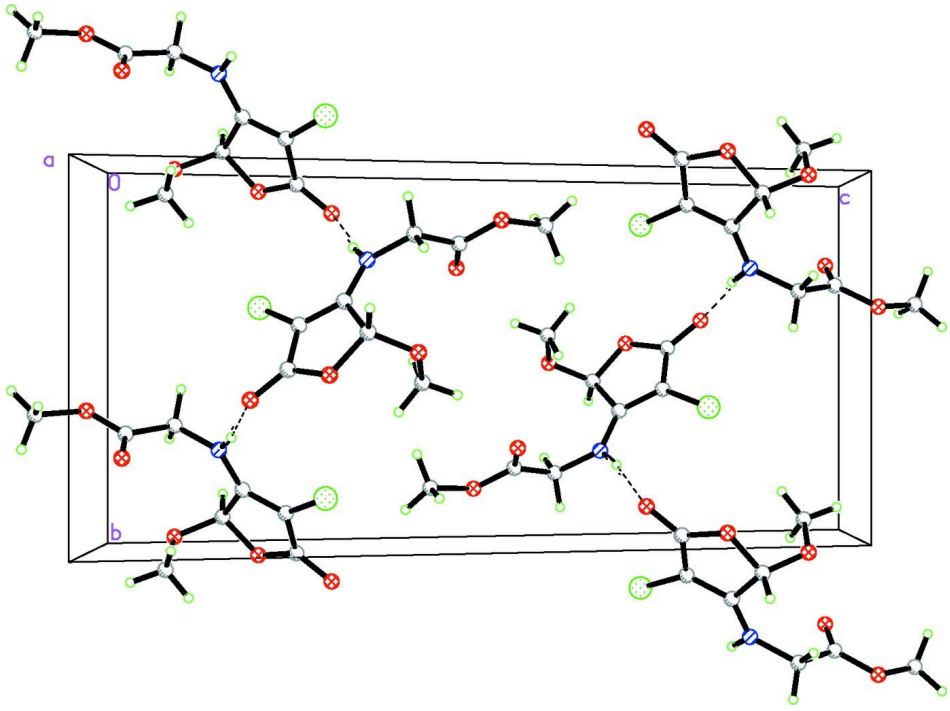


Figure 2

Perspective view of the packing of (I). Dashed lines stand for hydrogen bonds.

Methyl 2-[(4-chloro-2-methoxy-5-oxo-2,5-dihydrofuran-3-yl)amino]acetate

Crystal data

$C_8H_{10}ClNO_5$
$M_r = 235.62$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.1366\ (10)$ Å
$b = 9.8316\ (19)$ Å
$c = 20.685\ (4)$ Å
$\beta = 102.532\ (4)°$
$V = 1019.7\ (3)$ Å^3
$Z = 4$

$F(000) = 488$
$D_x = 1.535$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 826 reflections
$\theta = 2.3–25.2°$
$\mu = 0.38$ mm^{-1}
$T = 296$ K
Cubic, colorless
0.21 × 0.21 × 0.21 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
3333 measured reflections
1714 independent reflections
1221 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 25.2°$, $\theta_{min} = 2.0°$
$h = -5 \rightarrow 6$
$k = -5 \rightarrow 11$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.088$
$S = 1.05$
1714 reflections
139 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0339P)^2 + 0.3573P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Experimental. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): 3.52 (3*H*, *s*, CH, CH_3), 3.82 (3*H*, *s*, CH, CH_3), 4.29 (2*H*, *s*, CH, CH_2), 5.75 (1*H*, *s*, CH).

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}
Cl1	0.60822 (13)	0.63332 (7)	0.79402 (3)	0.0553 (3)
O1	−0.0366 (3)	0.45788 (17)	0.69305 (7)	0.0483 (5)
O2	0.1905 (4)	0.39330 (18)	0.79393 (8)	0.0539 (5)
O3	−0.0115 (3)	0.51417 (18)	0.58504 (7)	0.0514 (5)
O4	0.1774 (4)	0.86029 (19)	0.48157 (8)	0.0584 (5)
O5	0.5179 (4)	0.7421 (2)	0.54029 (8)	0.0643 (6)
N1	0.3070 (4)	0.7560 (2)	0.65232 (9)	0.0422 (5)
H1	0.4557	0.7914	0.6723	0.051*
C1	0.1684 (5)	0.4711 (3)	0.74761 (11)	0.0430 (6)
C2	0.3270 (5)	0.5858 (2)	0.73770 (10)	0.0391 (6)
C3	0.2222 (4)	0.6466 (2)	0.67961 (10)	0.0370 (6)

C4	−0.0206 (5)	0.5655 (3)	0.64653 (11)	0.0431 (6)
H4	−0.1788	0.6234	0.6422	0.052*
C5	0.2212 (6)	0.4363 (3)	0.58209 (12)	0.0557 (7)
H5A	0.2345	0.3603	0.6118	0.084*
H5B	0.3767	0.4925	0.5948	0.084*
H5C	0.2083	0.4039	0.5377	0.084*
C6	0.1683 (5)	0.8198 (3)	0.59157 (11)	0.0437 (6)
H6A	0.1498	0.9162	0.5993	0.052*
H6B	−0.0092	0.7813	0.5785	0.052*
C7	0.3126 (5)	0.8008 (2)	0.53614 (11)	0.0406 (6)
C8	0.2996 (7)	0.8523 (3)	0.42456 (13)	0.0732 (9)
H8A	0.2823	0.7614	0.4072	0.110*
H8B	0.4851	0.8755	0.4378	0.110*
H8C	0.2122	0.9146	0.3911	0.110*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0510 (4)	0.0741 (5)	0.0358 (3)	−0.0075 (3)	−0.0018 (3)	−0.0010 (3)
O1	0.0403 (11)	0.0567 (11)	0.0465 (9)	−0.0055 (8)	0.0062 (7)	0.0025 (9)
O2	0.0549 (12)	0.0595 (12)	0.0498 (10)	0.0069 (9)	0.0166 (8)	0.0126 (9)
O3	0.0497 (12)	0.0609 (12)	0.0378 (9)	0.0078 (9)	−0.0033 (7)	−0.0068 (8)
O4	0.0615 (13)	0.0742 (13)	0.0422 (9)	0.0274 (10)	0.0172 (8)	0.0172 (9)
O5	0.0448 (12)	0.0992 (16)	0.0502 (10)	0.0270 (11)	0.0129 (8)	0.0102 (10)
N1	0.0366 (12)	0.0530 (13)	0.0352 (10)	−0.0009 (10)	0.0041 (8)	0.0014 (10)
C1	0.0370 (15)	0.0543 (16)	0.0393 (13)	0.0080 (12)	0.0119 (10)	−0.0011 (13)
C2	0.0365 (14)	0.0498 (15)	0.0305 (12)	0.0000 (11)	0.0061 (9)	−0.0013 (11)
C3	0.0323 (14)	0.0465 (14)	0.0332 (12)	0.0073 (11)	0.0094 (9)	−0.0029 (11)
C4	0.0369 (15)	0.0519 (16)	0.0389 (13)	0.0040 (11)	0.0045 (10)	0.0006 (12)
C5	0.0641 (19)	0.0567 (17)	0.0443 (14)	0.0112 (14)	0.0074 (12)	−0.0074 (13)
C6	0.0427 (16)	0.0466 (15)	0.0422 (13)	0.0086 (12)	0.0099 (11)	0.0054 (11)
C7	0.0382 (16)	0.0427 (14)	0.0400 (13)	0.0001 (12)	0.0067 (11)	−0.0007 (11)
C8	0.087 (2)	0.093 (2)	0.0451 (15)	0.0227 (19)	0.0262 (15)	0.0180 (16)

Geometric parameters (Å, º)

Cl1—C2	1.712 (2)	C2—C3	1.345 (3)
O1—C1	1.372 (3)	C3—C4	1.513 (3)
O1—C4	1.444 (3)	C4—H4	0.9800
O2—C1	1.212 (3)	C5—H5A	0.9600
O3—C4	1.378 (3)	C5—H5B	0.9600
O3—C5	1.432 (3)	C5—H5C	0.9600
O4—C7	1.326 (3)	C6—C7	1.506 (3)
O4—C8	1.453 (3)	C6—H6A	0.9700
O5—C7	1.189 (3)	C6—H6B	0.9700
N1—C3	1.331 (3)	C8—H8A	0.9600
N1—C6	1.446 (3)	C8—H8B	0.9600
N1—H1	0.8600	C8—H8C	0.9600

C1—C2	1.432 (4)		
C1—O1—C4	109.53 (18)	O3—C5—H5A	109.5
C4—O3—C5	115.52 (18)	O3—C5—H5B	109.5
C7—O4—C8	115.4 (2)	H5A—C5—H5B	109.5
C3—N1—C6	125.0 (2)	O3—C5—H5C	109.5
C3—N1—H1	117.5	H5A—C5—H5C	109.5
C6—N1—H1	117.5	H5B—C5—H5C	109.5
O2—C1—O1	121.0 (2)	N1—C6—C7	112.1 (2)
O2—C1—C2	130.7 (2)	N1—C6—H6A	109.2
O1—C1—C2	108.4 (2)	C7—C6—H6A	109.2
C3—C2—C1	110.4 (2)	N1—C6—H6B	109.2
C3—C2—Cl1	127.0 (2)	C7—C6—H6B	109.2
C1—C2—Cl1	122.65 (18)	H6A—C6—H6B	107.9
N1—C3—C2	129.3 (2)	O5—C7—O4	124.6 (2)
N1—C3—C4	123.20 (19)	O5—C7—C6	125.5 (2)
C2—C3—C4	107.5 (2)	O4—C7—C6	109.9 (2)
O3—C4—O1	111.4 (2)	O4—C8—H8A	109.5
O3—C4—C3	114.9 (2)	O4—C8—H8B	109.5
O1—C4—C3	104.20 (17)	H8A—C8—H8B	109.5
O3—C4—H4	108.7	O4—C8—H8C	109.5
O1—C4—H4	108.7	H8A—C8—H8C	109.5
C3—C4—H4	108.7	H8B—C8—H8C	109.5
C4—O1—C1—O2	178.2 (2)	C5—O3—C4—C3	52.9 (3)
C4—O1—C1—C2	−1.2 (2)	C1—O1—C4—O3	124.6 (2)
O2—C1—C2—C3	−177.5 (2)	C1—O1—C4—C3	0.2 (2)
O1—C1—C2—C3	1.9 (3)	N1—C3—C4—O3	57.7 (3)
O2—C1—C2—Cl1	2.5 (4)	C2—C3—C4—O3	−121.2 (2)
O1—C1—C2—Cl1	−178.19 (16)	N1—C3—C4—O1	179.8 (2)
C6—N1—C3—C2	−175.1 (2)	C2—C3—C4—O1	1.0 (2)
C6—N1—C3—C4	6.3 (3)	C3—N1—C6—C7	−110.6 (3)
C1—C2—C3—N1	179.5 (2)	C8—O4—C7—O5	−1.1 (4)
Cl1—C2—C3—N1	−0.4 (4)	C8—O4—C7—C6	178.5 (2)
C1—C2—C3—C4	−1.7 (3)	N1—C6—C7—O5	−0.7 (4)
Cl1—C2—C3—C4	178.33 (18)	N1—C6—C7—O4	179.7 (2)
C5—O3—C4—O1	−65.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.06	2.911 (3)	171
C6—H6*B*···O5^{ii}	0.97	2.42	3.366 (3)	166

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) *x*−1, *y*, *z*.