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Oxonium 2-carboxy-3-(2-furyl)acrylate

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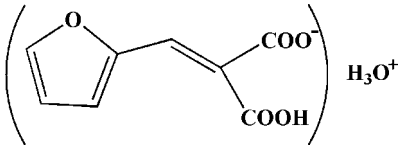
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.071; wR factor = 0.218; data-to-parameter ratio = 13.5.

In the title compound, $H_3O^+\cdot C_8H_5O_5^-$, neighbouring cations and anions are linked by O—H···O hydrogen bonds, forming a one-dimensional chain framework along [001]. The crystal structure is further stabilized by π–π interactions, with centroid–centroid distances of 3.734 (3) Å.

Related literature

For the synthesis of β-aminoacids as precursors of novel biologically active compounds, see: O'Callaghan, *et al.* (1998); Cohen *et al.* (2002); Zeller *et al.* (1965).



Experimental

Crystal data

$H_3O^+\cdot C_8H_5O_5^-$
M_r = 200.14
Monoclinic, $P2_1/c$
a = 13.664 (3) Å
b = 8.7518 (18) Å
c = 7.4664 (15) Å
β = 99.13 (3)°
V = 881.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.50 × 0.45 × 0.15 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.935, T_{max} = 0.980
7954 measured reflections
1727 independent reflections
1406 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.218
S = 1.06
1727 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WB···O4^{i}	0.85	2.58	3.044 (4)	115
O1W—H1WA···O3^{i}	0.85	2.42	3.188 (4)	150
O1W—H1WC···O4^{ii}	0.85	2.48	3.201 (4)	143
O2—H2A···O4^{iii}	0.82	1.74	2.552 (3)	169

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *PRPKAPPA* (Ferguson, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2321).

References

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supporting information

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Oxonium 2-carboxy-3-(2-furyl)acrylate

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S1. Comment

2-[(Furan-2-yl)methylene]malonic acid is a important bicarboxylic acid widely used in coordination chemistry and as an intermediate product in the synthesis of *β*-aminoacids. Recently, there has been an increased interest in the enantiomeric preparation of *β*-aminoacids as precursors for the synthesis of novel biologically active compounds (O′Callaghan *et al.*, 1998; Cohen *et al.*, 2002; Zeller *et al.*, 1965). We report here the crystal structure of the title compound, which was prepared by the reaction of furan-2-carbaldehyde and malonic acid.

The asymmetric unit of the title compound (Fig.1) consists of a 2-[(furan-2-yl)methylene]malonate anion and a oxonium cation. The values of the C–O bond lengths in the carboxylic groups are consistent with a single bond character of the C8–O2 bond (1.308 (4)Å) and with a delocalized double bond character for the C7–O4 and C7–O5 bonds (1.262 (4) and 1.240 (4) Å, respectively). In the crystal packing (Fig. 2), classical intermolecular O—H···O hydrogen bonds connect neighbouring cations and anions, resulting in a one-dimensional chain framework along the *c* axis (Table 1). The crystal structure is further stabilized by *π*–*π* stacking interactions (Table 2) involving adjacent furane rings.

S2. Experimental

A mixture of furan-2-carbaldehyde (0.5 mol, 0.48 g) and malonic acid (0.5 mol, 0.52 g) in ethanol (20 ml) was added in a flask and refluxed for 24 h. The resulting precipitate was separated and dissolved in an ethanol/water (5:1 v/v). Colourless single crystals of the title compound suitable for X-ray analysis were obtained on slow evaporation of the solvents over a period of 48 h.

S3. Refinement

The H atoms bound to O atoms were located in a difference Fourier map and refined with O—H = 0.82-0.85 Å and U_{iso}(H) = 1.5U_{iso}(O). All other H atoms were placed geometrically and allowed, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{iso}(C).

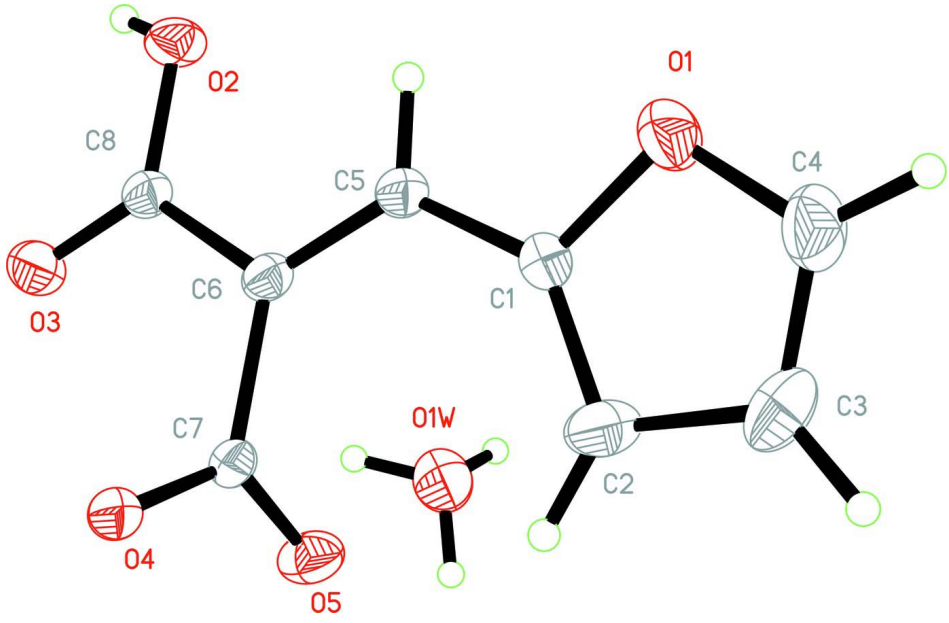

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

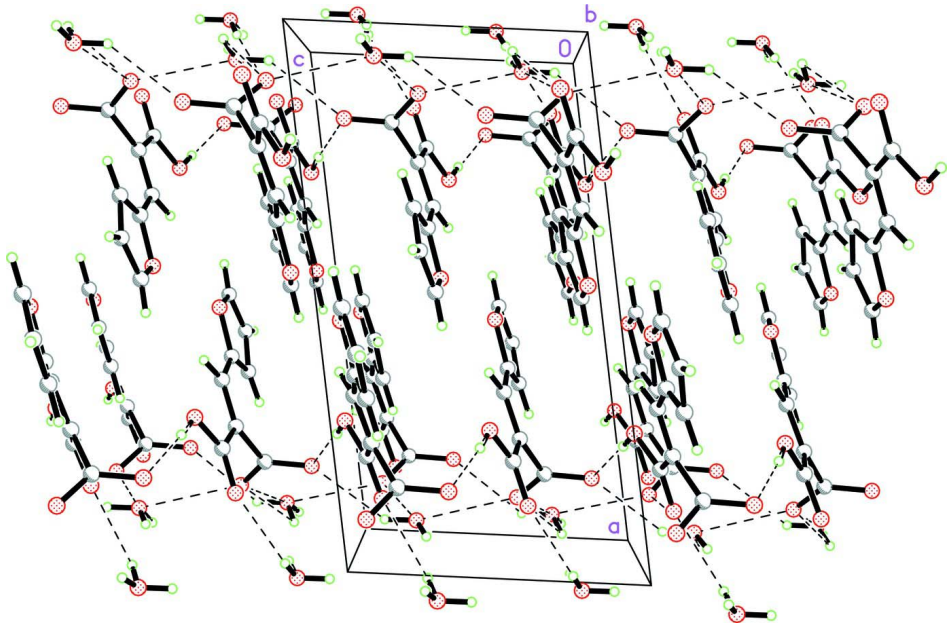

Figure 2

Packing diagram of the title compound viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

Oxonium 2-carboxy-3-(2-furyl)acrylate

Crystal data

$H_3O^+ \cdot C_8H_5O_5^-$
$M_r = 200.14$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

$a = 13.664\ (3)$ Å
$b = 8.7518\ (18)$ Å
$c = 7.4664\ (15)$ Å
$\beta = 99.13\ (3)°$

V = 881.5 (3) Å^3
Z = 4
F(000) = 416
D_x = 1.508 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1406 reflections

θ = 3.1–27.4°
μ = 0.13 mm^{-1}
T = 293 K
Prism, colourless
0.50 × 0.45 × 0.15 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD profile fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.935, T_{max} = 0.980

7954 measured reflections
1727 independent reflections
1406 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 26.0°, θ_{min} = 3.0°
h = −16→16
k = −10→10
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.218
S = 1.06
1727 reflections
128 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
$w = 1/[\sigma^2(F_o^2) + (0.1228P)^2 + 1.0867P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.0014 (2)

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}
C1	0.6364 (2)	0.3648 (4)	0.3879 (4)	0.0326 (7)
C2	0.6705 (3)	0.2258 (4)	0.3518 (5)	0.0471 (9)
H2	0.7334	0.2021	0.3275	0.057*
C3	0.5905 (3)	0.1220 (4)	0.3582 (6)	0.0552 (10)
H3	0.5918	0.0168	0.3408	0.066*
C4	0.5149 (3)	0.2026 (5)	0.3932 (6)	0.0548 (10)
H4	0.4530	0.1626	0.4032	0.066*
C5	0.6780 (2)	0.5157 (3)	0.4112 (4)	0.0316 (7)

H5	0.6393	0.5894	0.4562	0.038*
C6	0.7666 (2)	0.5621 (3)	0.3751 (4)	0.0294 (7)
C7	0.8392 (2)	0.4604 (3)	0.3007 (4)	0.0281 (7)
C8	0.8008 (2)	0.7221 (3)	0.4080 (4)	0.0299 (7)
O1	0.53944 (18)	0.3521 (3)	0.4129 (4)	0.0502 (7)
O2	0.73994 (16)	0.8126 (3)	0.4760 (3)	0.0389 (6)
H2A	0.7694	0.8902	0.5153	0.058*
O3	0.88184 (17)	0.7637 (3)	0.3750 (3)	0.0436 (7)
O4	0.84296 (16)	0.4683 (2)	0.1332 (3)	0.0354 (6)
O5	0.89313 (18)	0.3763 (3)	0.4078 (3)	0.0431 (6)
O1W	0.9457 (2)	0.4115 (3)	0.7823 (4)	0.0579 (8)
H1WC	0.9463	0.4130	0.8963	0.087*
H1WA	0.9797	0.3362	0.7549	0.087*
H1WB	0.9703	0.4941	0.7495	0.087*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0310 (16)	0.0324 (16)	0.0356 (16)	−0.0014 (12)	0.0094 (12)	0.0014 (12)
C2	0.0446 (19)	0.0367 (19)	0.059 (2)	0.0089 (15)	0.0065 (16)	−0.0065 (16)
C3	0.068 (3)	0.0307 (18)	0.064 (2)	−0.0096 (18)	0.001 (2)	−0.0026 (17)
C4	0.050 (2)	0.048 (2)	0.067 (3)	−0.0196 (18)	0.0112 (18)	−0.0031 (19)
C5	0.0362 (17)	0.0269 (15)	0.0333 (15)	0.0027 (12)	0.0106 (12)	−0.0005 (12)
C6	0.0336 (16)	0.0230 (14)	0.0319 (15)	0.0023 (12)	0.0062 (12)	−0.0003 (11)
C7	0.0301 (15)	0.0205 (13)	0.0344 (16)	−0.0016 (11)	0.0074 (12)	0.0002 (11)
C8	0.0331 (16)	0.0250 (14)	0.0318 (15)	0.0008 (12)	0.0061 (12)	−0.0013 (12)
O1	0.0416 (14)	0.0451 (15)	0.0675 (17)	−0.0090 (11)	0.0199 (12)	−0.0047 (12)
O2	0.0382 (12)	0.0280 (11)	0.0521 (14)	0.0001 (9)	0.0120 (10)	−0.0121 (10)
O3	0.0409 (13)	0.0333 (12)	0.0602 (16)	−0.0059 (10)	0.0195 (11)	−0.0077 (11)
O4	0.0447 (13)	0.0290 (12)	0.0352 (12)	0.0061 (9)	0.0149 (9)	0.0025 (9)
O5	0.0481 (14)	0.0401 (13)	0.0397 (12)	0.0148 (11)	0.0030 (10)	0.0027 (10)
O1W	0.0586 (17)	0.0583 (17)	0.0589 (17)	−0.0026 (14)	0.0162 (13)	0.0010 (13)

Geometric parameters (Å, º)

C1—C2	1.345 (5)	C6—C8	1.485 (4)
C1—O1	1.372 (4)	C6—C7	1.503 (4)
C1—C5	1.437 (4)	C7—O5	1.240 (4)
C2—C3	1.428 (6)	C7—O4	1.262 (4)
C2—H2	0.9300	C8—O3	1.226 (4)
C3—C4	1.311 (6)	C8—O2	1.308 (4)
C3—H3	0.9300	O2—H2A	0.8200
C4—O1	1.352 (5)	O1W—H1WC	0.8500
C4—H4	0.9300	O1W—H1WA	0.8500
C5—C6	1.344 (4)	O1W—H1WB	0.8500
C5—H5	0.9300		
C2—C1—O1	109.0 (3)	C5—C6—C8	121.5 (3)

C2—C1—C5	135.4 (3)	C5—C6—C7	124.3 (3)
O1—C1—C5	115.5 (3)	C8—C6—C7	114.3 (2)
C1—C2—C3	106.1 (3)	O5—C7—O4	123.9 (3)
C1—C2—H2	126.9	O5—C7—C6	118.2 (3)
C3—C2—H2	126.9	O4—C7—C6	117.8 (3)
C4—C3—C2	107.2 (3)	O3—C8—O2	123.2 (3)
C4—C3—H3	126.4	O3—C8—C6	121.1 (3)
C2—C3—H3	126.4	O2—C8—C6	115.6 (3)
C3—C4—O1	110.7 (3)	C4—O1—C1	107.0 (3)
C3—C4—H4	124.7	C8—O2—H2A	109.5
O1—C4—H4	124.7	H1WC—O1W—H1WA	109.5
C6—C5—C1	127.2 (3)	H1WC—O1W—H1WB	109.5
C6—C5—H5	116.4	H1WA—O1W—H1WB	109.5
C1—C5—H5	116.4		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WB*···O4^{i}	0.85	2.58	3.044 (4)	115
O1*W*—H1*WA*···O3^{i}	0.85	2.42	3.188 (4)	150
O1*W*—H1*WC*···O4^{ii}	0.85	2.48	3.201 (4)	143
O2—H2*A*···O4^{iii}	0.82	1.74	2.552 (3)	169

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