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1,2-Di-2-furylethane-1,2-dione

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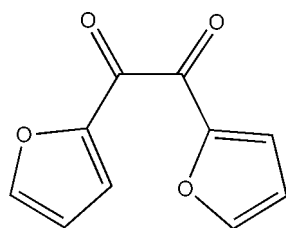
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.041; wR factor = 0.112; data-to-parameter ratio = 8.3.

The title compound, $\text{C}_{10}\text{H}_6\text{O}_4$, lies across a twofold rotation axis through the midpoint of the C—C bond between the two carbonyl groups. The furan ring plane and the plane through all atoms are inclined at 23.88 (1°). In the crystal structure, weak C—H $\cdots$ O hydrogen bonds form sheets in the bc plane and columns down the c axis.

Related literature

For background to the chemistry of vicinal polycarbonyl compounds, see: Rubin & White (1982); Beddoes *et al.* (1975). For related structures and bond-length data, see: Brown & Sadanaga (1965).



Experimental

Crystal data

$\text{C}_{10}\text{H}_6\text{O}_4$
 $M_r = 190.15$
Orthorhombic, $Fdd2$
 $a = 14.903$ (4) Å
 $b = 30.511$ (6) Å
 $c = 3.7770$ (8) Å

$V = 1717.4$ (7) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹
 $T = 293$ (2) K
 $0.25 \times 0.22 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer
Absorption correction: none
2069 measured reflections
537 independent reflections

473 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.098$
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.113$
 $S = 1.14$
537 reflections
65 parameters

1 restraint
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C1}-\text{H1A}\cdots\text{O1}^{\text{i}}$	0.93	2.61	3.435 (3)	149
$\text{C1}-\text{H1A}\cdots\text{O2}^{\text{ii}}$	0.93	2.82	3.322 (4)	115
$\text{C2}-\text{H2A}\cdots\text{O1}^{\text{iii}}$	0.93	2.64	3.439 (3)	145
$\text{C3}-\text{H3A}\cdots\text{O1}^{\text{iv}}$	0.93	2.70	3.074 (3)	105

Symmetry codes: (i) $x - \frac{1}{4}, -y + \frac{1}{4}, z + \frac{3}{4}$; (ii) $x - \frac{1}{4}, -y + \frac{1}{4}, z - \frac{1}{4}$; (iii) $x - \frac{1}{2}, y, z + \frac{1}{2}$; (iv) $-x + \frac{1}{2}, -y + \frac{1}{2}, z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2428).

References

- Beddoes, R. L., Cannon, J. R., Heller, M., Mills, O. S., Patrick, V. A. & Rubin, M. B. (1975). *Chem. Rev.* **75**, 177–202.
Brown, C. J. & Sadanaga, R. (1965). *Acta Cryst.* **18**, 158–164.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Rubin, M. B. & White, A. H. (1982). *Aust. J. Chem.* **35**, 543–556.
Sheldrick, G. M. (1990). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

supporting information

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1,2-Di-2-furylethane-1,2-dione

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S1. Comment

The structures of vicinal polycarbonyl compounds have been of interest for many years (Rubin & White, 1982). Bond lengths, bond angles, and torsion angles in such molecules can deviate from 'normal' values in order to minimize the repulsive interactions resulting from juxtaposition of dipolar carbonyl groups (Brown & Sadanaga, 1965) and the steric interactions of the chain of carbonyl groups with the end groups present (Beddoes *et al.*, 1975). We report here the crystal structure of the title vicinal dione compound (I), Fig 1.

The molecule lies about a twofold rotation axis at the mid-point of the C5—C5A bond ($A = -x + 1/2, -y + 1/2, z$). Bond lengths and angles are similar to those observed for benzil (Brown & Sadanaga, 1965). The molecule is approximately planar with the maximum deviation from the plane through all atoms 0.954 (1) Å for the O1. The furan ring plane (O2, C2...C4) and the plane through all atoms are inclined at 23.88 (1)°. In the crystal structure, weak C—H...O hydrogen bonds, Table 1, form sheets in the *bc* plane and columns down the *c* axis.

S2. Experimental

Furfural (1.92 g, 20.0 mmol) was added to water (20 ml) together with the *N,N*-dialkylbenzimidazolium salt (1.14 g, 4.0 mmol) and triethylamine (0.5 ml, 3.6 mmol) and the solution stirred vigorously for 2 h under reflux to afford the title compound (1.71 g, yield 70%). Single crystals suitable for X-ray measurements were obtained by recrystallization from THF at room temperature.

S3. Refinement

In the absence of significant anomalous scattering effects, Friedel pairs were merged. Hydrogen atoms were fixed geometrically and allowed to ride on their parent atoms, with $d(\text{C—H}) = 0.93$ Å, and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

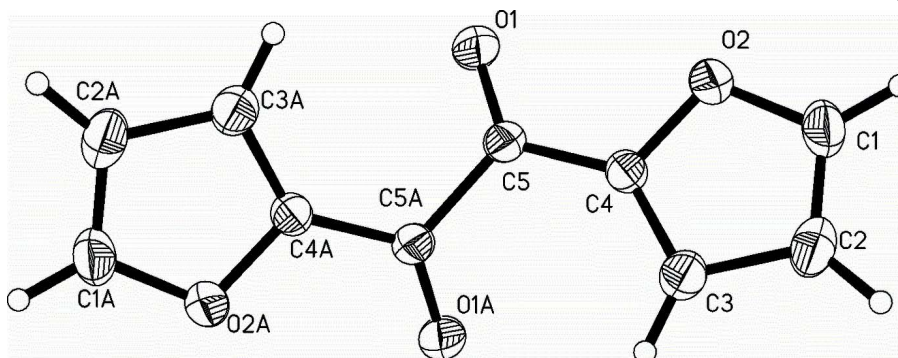


Figure 1

The molecular structure and atom-labeling scheme for (I), with displacement ellipsoids drawn at the 30% probability level. Atoms labelled A are related to other atoms by the symmetry operation $-x + 1/2, -y + 1/2, z$.

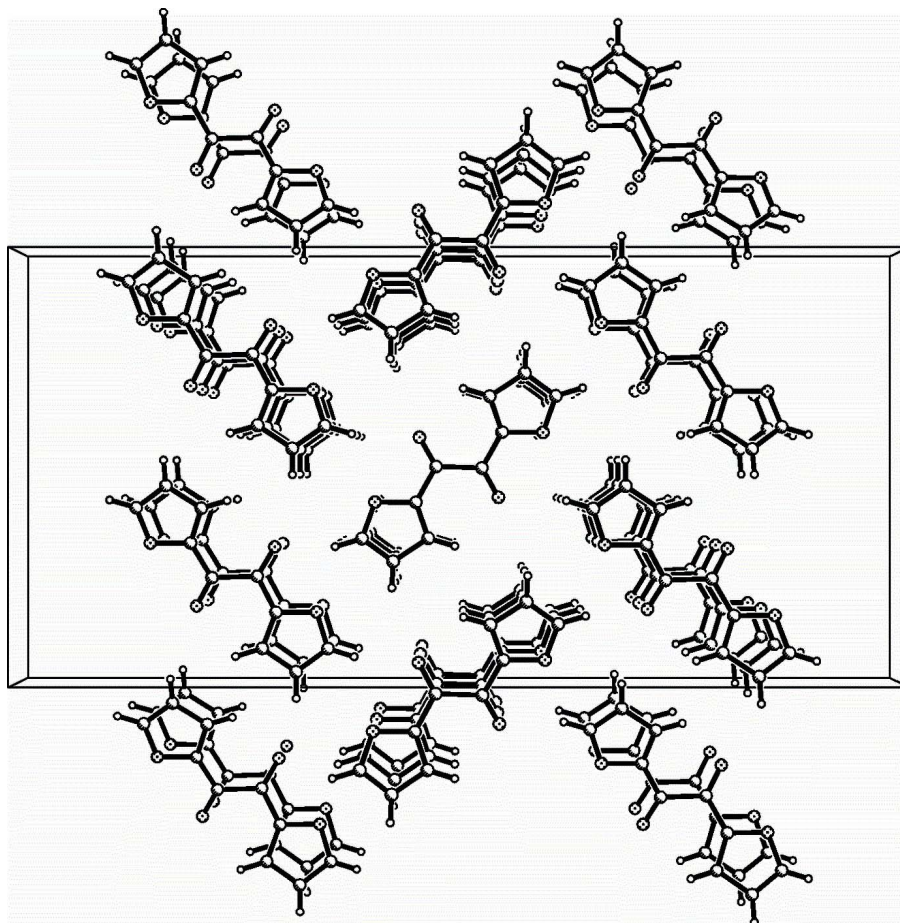


Figure 2

The Crystal packing of (I), viewed down the *c* axis.

1,2-Di-2-furylethane-1,2-dione

Crystal data

$C_{10}H_6O_4$

$M_r = 190.15$

Orthorhombic, *Fdd2*

Hall symbol: *F* 2 -2d

$a = 14.903$ (4) Å

$b = 30.511$ (6) Å

$c = 3.7770$ (8) Å

$V = 1717.4$ (7) Å³

$Z = 8$

$F(000) = 784$

$D_x = 1.471$ Mg m⁻³

Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 4\text{--}14^\circ$

$\mu = 0.12$ mm⁻¹

$T = 293$ K

Block, colourless

$0.25 \times 0.22 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

2069 measured reflections

537 independent reflections

473 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.098$

$\theta_{\text{max}} = 27.0^\circ$, $\theta_{\text{min}} = 2.7^\circ$

$h = -18 \rightarrow 18$

$k = -36 \rightarrow 37$

$l = -4 \rightarrow 0$

3 standard reflections every 100 reflections

intensity decay: none

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.041$ $wR(F^2) = 0.113$ $S = 1.14$

537 reflections

65 parameters

1 restraint

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.6456P]$
where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL*, $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.005 (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 ($\text{\AA}^2$)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.31781 (10)	0.20624 (6)	0.2936 (8)	0.0645 (7)
O2	0.17183 (10)	0.15778 (5)	0.5125 (7)	0.0586 (6)
C1	0.09123 (18)	0.14450 (9)	0.6479 (12)	0.0668 (9)
H1A	0.0735	0.1154	0.6673	0.080*
C2	0.04119 (18)	0.17857 (9)	0.7488 (9)	0.0598 (8)
H2A	-0.0160	0.1775	0.8472	0.072*
C3	0.09224 (16)	0.21647 (8)	0.6764 (7)	0.0488 (6)
H3A	0.0751	0.2453	0.7178	0.059*
C4	0.17150 (13)	0.20269 (6)	0.5341 (7)	0.0409 (6)
C5	0.25168 (15)	0.22475 (6)	0.4110 (8)	0.0416 (6)

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0430 (8)	0.0496 (9)	0.1008 (17)	0.0044 (7)	0.0119 (12)	-0.0137 (11)
O2	0.0446 (8)	0.0400 (8)	0.0913 (15)	-0.0025 (7)	-0.0098 (10)	0.0031 (11)
C1	0.0514 (13)	0.0527 (13)	0.096 (2)	-0.0165 (11)	-0.0162 (17)	0.0175 (17)
C2	0.0439 (11)	0.0709 (16)	0.0646 (18)	-0.0134 (11)	-0.0013 (13)	0.0095 (15)
C3	0.0435 (11)	0.0518 (12)	0.0513 (13)	-0.0025 (10)	0.0012 (12)	-0.0029 (12)
C4	0.0393 (11)	0.0366 (10)	0.0468 (11)	-0.0013 (8)	-0.0081 (10)	0.0021 (11)
C5	0.0361 (10)	0.0406 (11)	0.0481 (12)	0.0017 (8)	-0.0032 (10)	-0.0034 (10)

Geometric parameters (Å, °)

O1—C5	1.219 (3)	C2—H2A	0.9300
O2—C1	1.367 (4)	C3—C4	1.364 (3)
O2—C4	1.373 (2)	C3—H3A	0.9300
C1—C2	1.335 (4)	C4—C5	1.448 (3)
C1—H1A	0.9300	C5—C5 ⁱ	1.541 (4)
C2—C3	1.411 (4)		
C1—O2—C4	105.7 (2)	C4—C3—H3A	126.6
C2—C1—O2	111.5 (2)	C2—C3—H3A	126.6
C2—C1—H1A	124.2	C3—C4—O2	109.52 (19)
O2—C1—H1A	124.2	C3—C4—C5	134.25 (19)
C1—C2—C3	106.4 (2)	O2—C4—C5	116.23 (19)
C1—C2—H2A	126.8	O1—C5—C4	124.63 (19)
C3—C2—H2A	126.8	O1—C5—C5 ⁱ	119.3 (2)
C4—C3—C2	106.9 (2)	C4—C5—C5 ⁱ	116.0 (2)
C4—O2—C1—C2	0.5 (4)	C1—O2—C4—C5	178.9 (3)
O2—C1—C2—C3	−0.2 (4)	C3—C4—C5—O1	178.9 (3)
C1—C2—C3—C4	−0.1 (3)	O2—C4—C5—O1	−0.3 (4)
C2—C3—C4—O2	0.4 (3)	C3—C4—C5—C5 ⁱ	−5.0 (4)
C2—C3—C4—C5	−178.9 (3)	O2—C4—C5—C5 ⁱ	175.79 (18)
C1—O2—C4—C3	−0.5 (3)		

Symmetry code: (i) $-x+1/2, -y+1/2, z$.*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C1—H1A $\cdots$ O1 ⁱⁱ	0.93	2.61	3.435 (3)	149
C1—H1A $\cdots$ O2 ⁱⁱⁱ	0.93	2.82	3.322 (4)	115
C2—H2A $\cdots$ O1 ^{iv}	0.93	2.64	3.439 (3)	145
C3—H3A $\cdots$ O1 ⁱ	0.93	2.70	3.074 (3)	105

Symmetry codes: (i) $-x+1/2, -y+1/2, z$; (ii) $x-1/4, -y+1/4, z+3/4$; (iii) $x-1/4, -y+1/4, z-1/4$; (iv) $x-1/2, y, z+1/2$.