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3*H*-Furo[3,4-*c*]isochromene-1,5-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.096
Data-to-parameter ratio = 10.2

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

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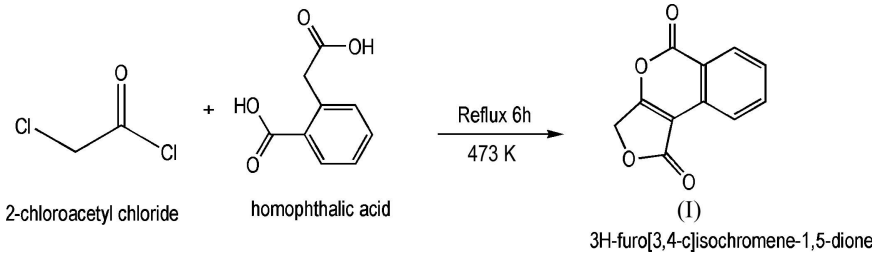
The title compound, $C_{11}H_6O_4$, is a synthetic isocoumarin and is essentially planar.

Comment

Isocoumarin is an abundant structural motif in natural products (Barry, 1964). Many constituents of the steadily growing class of known isocoumarins exhibit valuable biological properties, such as antifungal (Meepagala *et al.*, 2002), antitumor or cytotoxic, anti-inflammatory, anti-allergic (Rossi *et al.*, 2003), and enzyme inhibitory activities (Powers *et al.*, 2002). Naturally occurring haloisocoumarins and their halo-3,4-dihydroisocoumarin derivatives are very rare; however, a few examples of naturally occurring chlorine-containing isocoumarins are known (Larsen & Breinholt, 1999). In view of the importance of this class of compounds, the title compound, (I), has been synthesized, and its crystal structure is reported here.



2-chloroacetyl chloride homophthalic acid (I)
3H-furo[3,4-c]isochromene-1,5-dione

The molecular structure of the title compound is shown in Fig. 1. In (I), all bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The five-membered ring is coplanar with the isocoumarin ring system. The bond lengths within the benzene ring lie between 1.375 (3) Å and 1.405 (2) Å. There is no hydrogen bonding below 3.0 Å found in the crystal structure.

Experimental

A mixture of homophthalic acid (2.0 g, 11 mmol) and chloroacetyl chloride (7.96 g, 5 ml, 46 mmol) was refluxed for 6 h at 473 K with stirring. The reaction mixture was dissolved in ethyl acetate (3 × 100 ml) and an aqueous solution of sodium carbonate (5%, 2 × 100 ml) was added in order to remove the unreacted homophthalic acid. The organic layer was separated, concentrated and chromatographed on silica gel using petroleum ether (313–353 K fraction) as eluant to afford the title compound (yield 66%, m.p. 404 K). Colorless single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate solution.

Crystal data

$C_{11}H_6O_4$
M_r = 202.16
Triclinic, $P\overline{1}$
a = 7.9892 (13) Å
b = 8.0899 (13) Å
c = 8.2075 (13) Å
α = 67.089 (3)°
β = 83.871 (3)°
γ = 62.738 (3)°
V = 432.68 (12) Å^3
Z = 2
D_x = 1.552 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
Block, colorless
0.32 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.850, T_{max} = 1.000
1966 measured reflections
1396 independent reflections
1105 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.096
S = 1.03
1396 reflections
137 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2 + 0.0519P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.010 (5)

All H atoms were placed in idealized positions with C—H = 0.93–0.97 Å and constrained to ride on their parent atoms; U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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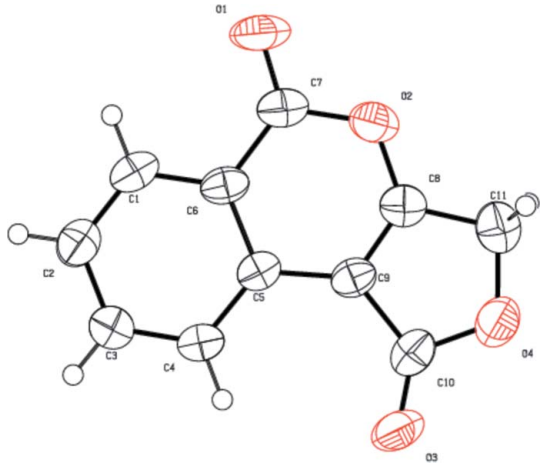

Figure 1
View of the title compound with displacement ellipsoids drawn at the 50% probability level.

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