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Ethyl *N*-(4-methyl-2-oxo-1*H*-benzopyran-7-yl)carbamate

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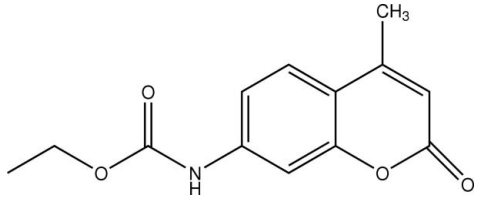
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.144; data-to-parameter ratio = 12.7.

The title compound, $C_{13}H_{13}NO_4$, crystallizes with two molecules in the asymmetric unit. These are linked by one N—H···O hydrogen bond and two C—H···O hydrogen bonds, forming $R_4^4(36)[R_2^1(6)R_4^4(32)]$ and $R_8^8(60)[R_2^1(6)R_8^8(56)]$ rings. The molecules are also linked by C—H···π interactions, resulting in a three-dimensional structure.

Related literature

For related literature, see: Aazam *et al.* (2006); Allen *et al.* (1987); Bernstein *et al.* (1995); Hamaker & McCully (2006); Lemieux *et al.* (2003); Yang *et al.* (2006, 2007); Zhao *et al.* (2004).



Experimental

Crystal data

$C_{13}H_{13}NO_4$
M_r = 247.24
Monoclinic, $P2_1/c$
a = 11.366 (1) Å
b = 24.875 (2) Å
c = 9.075 (1) Å
β = 112.425 (2)°
V = 2371.7 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.58 × 0.50 × 0.31 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.942, T_{max} = 0.969
11821 measured reflections
4185 independent reflections
2261 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.144
S = 1.02
4185 reflections
329 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

*Cg*1*A* is the centroid of the ring C4*A*–C9*A*.

D—H···A	D—H	H···A	D···A	D—H···A
C8*A*—H8*A*···O4*A*	0.93	2.28	2.881 (4)	122
C8*B*—H8*B*···O4*B*	0.93	2.19	2.799 (3)	122
N1*B*—H1*B*···O4*A*	0.86	2.25	3.104 (3)	170
N1*A*—H1*A*···O2*B*^{i}	0.86	2.04	2.878 (3)	164
C6*A*—H6*A*···O2*B*^{i}	0.93	2.48	3.243 (4)	140
C2*A*—H2*A*···O4*B*^{ii}	0.93	2.55	3.392 (4)	151
C13*A*—H13*C*···*Cg*1A^{iii}	0.96	2.79	3.637 (4)	147

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2400).

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supplementary materials

Acta Cryst. (2007). E**63**, o4099 [**doi:10.1107/S1600536807045515**]

Ethyl *N*-(4-methyl-2-oxo-1*H*-benzopyran-7-yl)carbamate

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Comment

Some aminocoumarin derivatives are known to be a category of important fluorogenic dyes (Lemieux *et al.*, 2003; Zhao *et al.*, 2004). The hydrogen bonding interaction in the self-association of some coumarin derivatives in the solid state (Aazam *et al.*, 2006; Hamaker & McCully, 2006; Yang *et al.*, 2006; Yang *et al.*, 2007) have been decribed. Here, we report the crystal structure of (I).

Compound (I) crystallizes in monoclinic space group $P2_1/c$ with the two independent molecules in the asymmetric unit. The molecules *A* and *B* have a similar conformation (Fig. 1). Two coumarin moieties in (I) are nearly planar, and the dihedral angles between the pyrone and the benzene rings are 1.4 (1)° and 0.9 (1)° in molecules *A* and *B*, respectively. Geometric parameters are normal (Allen *et al.*, 1987) in molecules *A* and *B* . In the selected asymmetric unit, each of the independent molecules there is an intramolecular C—H···O hydrogen bond, defining an *S*(6) motif (Bernstein *et al.*, 1995), and the molecules *A* and *B* are linked by one N—H···O hydrogen bond (Table 1 and Fig.1).

In the crystal structure of (I), atom N1A and C6A in the molecule *A* at (*x*, *y*, *z*) act as hydrogen-bond donors to atom O2B in the molecule *B* at (1 + *x*, 1/2 − *y*, 3/2 + *z*), forming a $C_2^2(12)[R_2^1(6)]$ chain of rings (Bernstein *et al.*, 1995). Similarly, the atom C2A in the molecule *A* at (*x*, *y*, *z*) acts as hydrogen-bond donor to the atom O4B in the molecule *B* at (1 − *x*, 1/2 + *y*, 1/2 − *z*), thus generating a $C_2^2(14)$ chain. The combination of these chains generates $R_4^4(36)[R_2^1(6)R_4^4(32)]$ and $R_8^8(60)[R_2^1(6)R_8^8(56)]$ rings (Table 1 and Fig. 2).

The molecules are also linked by C—H···π interactions [C13A···*Cg*1 A^{iii} = 3.637 (4) Å, C13—H13C···*Cg*1 A^{iii} = 147°, where *Cg*1 A is the centroid of the ring C4A–C9A, symmetry code: (iii) *x*, 1/2 − *y*, 1/2 + *z*], resulting in a three-dimensional structure.

Experimental

The reaction mixture containing 3-ethoxycarbonylaminophenol (1.81 g, 10 mmol), acetoacetic ester (1.3 ml, 10 mmol) and phosphoric acid (5.3 ml) was stirred at 343–353 K for 12 h, and then poured into the water. The solid obtained was filtered off, washed with water and dried at room temperature. Colourless crystals of (I) suitable for X-ray structure analysis were obtained by slow evaporation of a solution of ethanol–petroleum (1:1) with the crude product over three weeks.

Refinement

H atoms were placed in calculated positions with C—H = 0.93 Å (aromatic), 0.97 Å (methylene), 0.96 Å (methyl) and N—H = 0.86Å (amino), and refined in riding mode with U_{iso}(H) = 1.5U_{eq}(C) (methyl) and U_{iso}(H) = 1.2U_{eq}(C,*N*)(aromatic, methylene and amino).

Figures

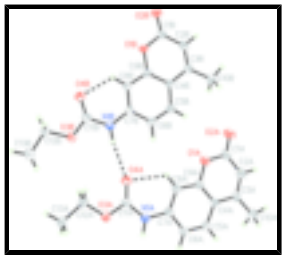

Fig. 1. The two independent molecules in compound (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines indicate hydrogen bonds.

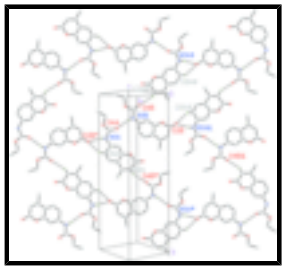

Fig. 2. Part of the crystal structure of (I), showing R_4^4 (36) [$R_2^1(6)R_4^4(32)$] and $R_8^8(60)$ [$R_2^1(6)R_8^8(56)$] rings. For clarity, H atoms bonded to C atoms have been omitted. Dashed lines indicate hydrogen bonds [Symmetry codes: (*) 1 + *x*, 1/2 − *y*, 3/2 + *z*; (#) 1 − *x*, 1/2 + *y*, 1/2 − *z*; (&) −1 + *x*, 1/2 − *y*, −3/2 + *x*; ($) 1 − *x*, −1/2 + *y*, 1/2 − *z*].

Ethyl *N*-(4-methyl-2-oxo-1*H*-benzopyran-7-yl)carbamate

Crystal data

$C_{13}H_{13}NO_4$	F_{000} = 1040
M_r = 247.24	D_x = 1.385 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2636 reflections
a = 11.3660 (10) Å	θ = 2.5–24.0º
b = 24.875 (2) Å	μ = 0.10 mm^{-1}
c = 9.0750 (11) Å	*T* = 298 (2) K
β = 112.425 (2)º	Block, colourless
V = 2371.7 (4) Å^3	0.58 × 0.50 × 0.31 mm
Z = 8	

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	4185 independent reflections
Radiation source: fine-focus sealed tube	2261 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.076
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −13→12
T_{min} = 0.942, T_{max} = 0.969	*k* = −25→29
11821 measured reflections	*l* = −10→7

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.144$	$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 0.9292P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
4185 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
329 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 $F^2 > 2$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}
N1A	0.9015 (2)	0.28355 (9)	0.8953 (3)	0.0481 (7)
H1A	0.9571	0.2848	0.9914	0.058*
N1B	0.5937 (2)	0.15407 (9)	0.4348 (3)	0.0456 (7)
H1B	0.6510	0.1692	0.5157	0.055*
O1A	0.6168 (2)	0.38887 (8)	0.4701 (2)	0.0600 (6)
O2A	0.4820 (2)	0.43292 (10)	0.2653 (3)	0.0832 (9)
O3A	0.9400 (2)	0.19733 (8)	0.9377 (2)	0.0539 (6)
O4A	0.7880 (2)	0.22272 (8)	0.7035 (2)	0.0594 (6)
O1B	0.25690 (18)	0.18418 (7)	−0.0695 (2)	0.0465 (5)
O2B	0.1001 (2)	0.19225 (9)	−0.3016 (2)	0.0597 (6)
O3B	0.68274 (18)	0.07941 (8)	0.5614 (2)	0.0513 (6)
O4B	0.5105 (2)	0.07233 (8)	0.3349 (3)	0.0700 (7)
C1A	0.5710 (3)	0.43702 (14)	0.3906 (4)	0.0595 (9)
C2A	0.6316 (3)	0.48530 (13)	0.4690 (4)	0.0589 (9)
H2A	0.6024	0.5179	0.4178	0.071*
C3A	0.7279 (3)	0.48639 (12)	0.6116 (4)	0.0498 (8)
C4A	0.7739 (3)	0.43540 (11)	0.6909 (3)	0.0412 (7)
C5A	0.8744 (3)	0.42925 (12)	0.8372 (4)	0.0492 (8)

H5A	0.9158	0.4597	0.8920	0.059*
C6A	0.9145 (3)	0.37971 (12)	0.9033 (3)	0.0480 (8)
H6A	0.9810	0.3771	1.0023	0.058*
C7A	0.8560 (3)	0.33308 (11)	0.8226 (3)	0.0425 (8)
C8A	0.7551 (3)	0.33765 (12)	0.6777 (3)	0.0491 (8)
H8A	0.7134	0.3072	0.6233	0.059*
C9A	0.7171 (3)	0.38830 (12)	0.6150 (3)	0.0460 (8)
C10A	0.7875 (3)	0.53776 (13)	0.6904 (4)	0.0713 (11)
H10A	0.7475	0.5675	0.6221	0.107*
H10B	0.8766	0.5374	0.7101	0.107*
H10C	0.7764	0.5414	0.7897	0.107*
C11A	0.8682 (3)	0.23365 (12)	0.8320 (4)	0.0446 (8)
C12A	0.9173 (3)	0.14170 (11)	0.8869 (4)	0.0572 (9)
H12A	0.9315	0.1363	0.7891	0.069*
H12B	0.8305	0.1314	0.8688	0.069*
C13A	1.0104 (3)	0.10912 (12)	1.0206 (4)	0.0620 (10)
H13A	1.0956	0.1202	1.0383	0.093*
H13B	1.0005	0.0717	0.9927	0.093*
H13C	0.9945	0.1147	1.1161	0.093*
C1B	0.1758 (3)	0.21595 (13)	−0.1872 (3)	0.0455 (8)
C2B	0.1863 (3)	0.27277 (12)	−0.1633 (3)	0.0468 (8)
H2B	0.1308	0.2949	−0.2417	0.056*
C3B	0.2722 (3)	0.29573 (11)	−0.0336 (3)	0.0417 (7)
C4B	0.3577 (3)	0.26164 (11)	0.0885 (3)	0.0376 (7)
C5B	0.4538 (3)	0.27906 (12)	0.2297 (3)	0.0469 (8)
H5B	0.4667	0.3157	0.2490	0.056*
C6B	0.5295 (3)	0.24323 (12)	0.3404 (4)	0.0496 (8)
H6B	0.5920	0.2560	0.4338	0.060*
C7B	0.5137 (3)	0.18778 (11)	0.3146 (3)	0.0400 (7)
C8B	0.4216 (3)	0.16943 (11)	0.1753 (3)	0.0424 (8)
H8B	0.4100	0.1327	0.1551	0.051*
C9B	0.3470 (3)	0.20605 (11)	0.0665 (3)	0.0381 (7)
C10B	0.2786 (3)	0.35549 (12)	−0.0124 (4)	0.0608 (10)
H10D	0.2132	0.3721	−0.1019	0.091*
H10E	0.3603	0.3682	−0.0052	0.091*
H10F	0.2663	0.3646	0.0836	0.091*
C11B	0.5883 (3)	0.09907 (12)	0.4339 (4)	0.0470 (8)
C12B	0.6840 (3)	0.02102 (12)	0.5736 (4)	0.0608 (10)
H12C	0.6739	0.0049	0.4722	0.073*
H12D	0.6148	0.0089	0.6030	0.073*
C13B	0.8085 (3)	0.00531 (14)	0.6981 (4)	0.0806 (12)
H13D	0.8760	0.0161	0.6655	0.121*
H13F	0.8109	−0.0330	0.7124	0.121*
H13E	0.8189	0.0227	0.7968	0.121*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

N1A	0.0580 (17)	0.0410 (15)	0.0322 (14)	−0.0013 (13)	0.0027 (12)	−0.0006 (11)
N1B	0.0447 (15)	0.0375 (14)	0.0396 (15)	−0.0048 (12)	−0.0006 (12)	0.0000 (11)
O1A	0.0583 (14)	0.0525 (14)	0.0496 (14)	0.0015 (12)	−0.0013 (11)	0.0056 (11)
O2A	0.0740 (17)	0.0756 (19)	0.0654 (17)	0.0060 (14)	−0.0123 (14)	0.0118 (13)
O3A	0.0693 (15)	0.0388 (12)	0.0426 (12)	0.0008 (11)	0.0088 (11)	0.0012 (10)
O4A	0.0624 (14)	0.0488 (13)	0.0476 (14)	−0.0058 (11)	−0.0007 (12)	−0.0027 (10)
O1B	0.0519 (12)	0.0405 (12)	0.0348 (11)	−0.0050 (10)	0.0027 (10)	−0.0013 (9)
O2B	0.0616 (14)	0.0591 (14)	0.0399 (13)	−0.0086 (12)	−0.0013 (11)	−0.0006 (11)
O3B	0.0499 (13)	0.0380 (12)	0.0526 (13)	0.0032 (10)	0.0045 (11)	0.0043 (10)
O4B	0.0766 (16)	0.0415 (13)	0.0597 (15)	−0.0072 (12)	−0.0101 (13)	−0.0020 (11)
C1A	0.052 (2)	0.063 (2)	0.053 (2)	0.0091 (19)	0.0067 (18)	0.0127 (18)
C2A	0.061 (2)	0.047 (2)	0.063 (2)	0.0050 (18)	0.0177 (19)	0.0125 (17)
C3A	0.0500 (19)	0.0433 (19)	0.057 (2)	0.0040 (16)	0.0212 (17)	0.0067 (15)
C4A	0.0407 (18)	0.0414 (18)	0.0422 (18)	0.0037 (15)	0.0164 (15)	0.0005 (14)
C5A	0.051 (2)	0.0458 (19)	0.046 (2)	−0.0012 (16)	0.0137 (16)	−0.0025 (15)
C6A	0.0511 (19)	0.0476 (19)	0.0374 (17)	0.0012 (16)	0.0079 (15)	−0.0015 (15)
C7A	0.0446 (18)	0.0423 (18)	0.0380 (17)	0.0011 (15)	0.0128 (15)	0.0032 (14)
C8A	0.0499 (19)	0.0423 (19)	0.0457 (19)	−0.0047 (16)	0.0078 (16)	−0.0016 (14)
C9A	0.0425 (18)	0.0485 (19)	0.0399 (18)	0.0024 (16)	0.0079 (15)	0.0045 (15)
C10A	0.079 (3)	0.045 (2)	0.084 (3)	−0.003 (2)	0.024 (2)	0.0048 (18)
C11A	0.0451 (18)	0.0440 (19)	0.0391 (19)	0.0004 (16)	0.0097 (16)	0.0007 (15)
C12A	0.080 (2)	0.0373 (18)	0.052 (2)	−0.0050 (18)	0.0219 (19)	−0.0014 (15)
C13A	0.078 (2)	0.0411 (19)	0.064 (2)	0.0036 (18)	0.0241 (19)	0.0051 (16)
C1B	0.0435 (19)	0.054 (2)	0.0331 (17)	−0.0028 (16)	0.0077 (15)	0.0007 (15)
C2B	0.0481 (19)	0.0461 (19)	0.0407 (18)	0.0031 (16)	0.0107 (15)	0.0085 (14)
C3B	0.0447 (18)	0.0388 (17)	0.0427 (18)	−0.0023 (15)	0.0180 (15)	0.0007 (14)
C4B	0.0383 (17)	0.0345 (17)	0.0405 (17)	−0.0022 (14)	0.0157 (14)	−0.0018 (13)
C5B	0.0529 (19)	0.0344 (17)	0.0488 (19)	−0.0072 (15)	0.0142 (16)	−0.0049 (14)
C6B	0.0473 (19)	0.0439 (19)	0.0439 (19)	−0.0073 (16)	0.0020 (15)	−0.0069 (15)
C7B	0.0380 (16)	0.0389 (17)	0.0392 (17)	0.0002 (14)	0.0102 (14)	−0.0023 (13)
C8B	0.0461 (18)	0.0336 (16)	0.0423 (18)	−0.0025 (14)	0.0112 (15)	−0.0044 (13)
C9B	0.0378 (17)	0.0403 (17)	0.0320 (16)	−0.0044 (14)	0.0086 (13)	−0.0054 (13)
C10B	0.069 (2)	0.0387 (19)	0.066 (2)	0.0035 (18)	0.0171 (19)	0.0040 (16)
C11B	0.0415 (18)	0.047 (2)	0.045 (2)	−0.0008 (17)	0.0082 (16)	0.0032 (15)
C12B	0.070 (2)	0.0381 (19)	0.062 (2)	0.0033 (18)	0.0114 (19)	0.0026 (16)
C13B	0.078 (3)	0.057 (2)	0.084 (3)	0.014 (2)	0.005 (2)	0.011 (2)

Geometric parameters (Å, °)

N1A—C11A	1.359 (3)	N1B—C11B	1.369 (4)
N1A—C7A	1.400 (3)	N1B—C7B	1.401 (3)
N1A—H1A	0.8600	N1B—H1B	0.8600
O1A—C9A	1.373 (3)	O1B—C1B	1.365 (3)
O1A—C1A	1.393 (4)	O1B—C9B	1.380 (3)
O2A—C1A	1.204 (4)	O2B—C1B	1.218 (3)
O3A—C11A	1.344 (3)	O3B—C11B	1.335 (3)
O3A—C12A	1.450 (3)	O3B—C12B	1.456 (3)
O4A—C11A	1.206 (3)	O4B—C11B	1.194 (3)
C1A—C2A	1.431 (4)	C1B—C2B	1.428 (4)

C2A—C3A	1.339 (4)	C2B—C3B	1.337 (4)
C2A—H2A	0.9300	C2B—H2B	0.9300
C3A—C4A	1.453 (4)	C3B—C4B	1.439 (4)
C3A—C10A	1.494 (4)	C3B—C10B	1.497 (4)
C4A—C9A	1.387 (4)	C4B—C9B	1.396 (4)
C4A—C5A	1.391 (4)	C4B—C5B	1.398 (4)
C5A—C6A	1.370 (4)	C5B—C6B	1.373 (4)
C5A—H5A	0.9300	C5B—H5B	0.9300
C6A—C7A	1.397 (4)	C6B—C7B	1.399 (4)
C6A—H6A	0.9300	C6B—H6B	0.9300
C7A—C8A	1.381 (4)	C7B—C8B	1.376 (3)
C8A—C9A	1.382 (4)	C8B—C9B	1.373 (4)
C8A—H8A	0.9300	C8B—H8B	0.9300
C10A—H10A	0.9600	C10B—H10D	0.9600
C10A—H10B	0.9600	C10B—H10E	0.9600
C10A—H10C	0.9600	C10B—H10F	0.9600
C12A—C13A	1.507 (4)	C12B—C13B	1.487 (4)
C12A—H12A	0.9700	C12B—H12C	0.9700
C12A—H12B	0.9700	C12B—H12D	0.9700
C13A—H13A	0.9600	C13B—H13D	0.9600
C13A—H13B	0.9600	C13B—H13F	0.9600
C13A—H13C	0.9600	C13B—H13E	0.9600
C11A—N1A—C7A	127.7 (2)	C11B—N1B—C7B	125.4 (2)
C11A—N1A—H1A	116.2	C11B—N1B—H1B	117.3
C7A—N1A—H1A	116.2	C7B—N1B—H1B	117.3
C9A—O1A—C1A	121.0 (2)	C1B—O1B—C9B	121.4 (2)
C11A—O3A—C12A	115.4 (2)	C11B—O3B—C12B	114.4 (2)
O2A—C1A—O1A	115.5 (3)	O2B—C1B—O1B	115.7 (3)
O2A—C1A—C2A	127.7 (3)	O2B—C1B—C2B	126.8 (3)
O1A—C1A—C2A	116.8 (3)	O1B—C1B—C2B	117.5 (2)
C3A—C2A—C1A	123.9 (3)	C3B—C2B—C1B	123.2 (3)
C3A—C2A—H2A	118.0	C3B—C2B—H2B	118.4
C1A—C2A—H2A	118.0	C1B—C2B—H2B	118.4
C2A—C3A—C4A	117.9 (3)	C2B—C3B—C4B	118.5 (3)
C2A—C3A—C10A	122.3 (3)	C2B—C3B—C10B	121.4 (3)
C4A—C3A—C10A	119.9 (3)	C4B—C3B—C10B	120.1 (3)
C9A—C4A—C5A	116.0 (3)	C9B—C4B—C5B	115.7 (2)
C9A—C4A—C3A	118.7 (3)	C9B—C4B—C3B	118.5 (2)
C5A—C4A—C3A	125.3 (3)	C5B—C4B—C3B	125.8 (3)
C6A—C5A—C4A	122.1 (3)	C6B—C5B—C4B	121.5 (3)
C6A—C5A—H5A	119.0	C6B—C5B—H5B	119.3
C4A—C5A—H5A	119.0	C4B—C5B—H5B	119.3
C5A—C6A—C7A	120.4 (3)	C5B—C6B—C7B	120.9 (3)
C5A—C6A—H6A	119.8	C5B—C6B—H6B	119.6
C7A—C6A—H6A	119.8	C7B—C6B—H6B	119.6
C8A—C7A—C6A	119.1 (3)	C8B—C7B—C6B	119.0 (3)
C8A—C7A—N1A	123.0 (3)	C8B—C7B—N1B	123.8 (3)
C6A—C7A—N1A	117.9 (2)	C6B—C7B—N1B	117.2 (2)
C7A—C8A—C9A	118.9 (3)	C9B—C8B—C7B	119.0 (3)

C7A—C8A—H8A	120.6	C9B—C8B—H8B	120.5
C9A—C8A—H8A	120.6	C7B—C8B—H8B	120.5
O1A—C9A—C8A	114.7 (3)	C8B—C9B—O1B	115.2 (2)
O1A—C9A—C4A	121.7 (3)	C8B—C9B—C4B	124.0 (2)
C8A—C9A—C4A	123.5 (3)	O1B—C9B—C4B	120.9 (2)
C3A—C10A—H10A	109.5	C3B—C10B—H10D	109.5
C3A—C10A—H10B	109.5	C3B—C10B—H10E	109.5
H10A—C10A—H10B	109.5	H10D—C10B—H10E	109.5
C3A—C10A—H10C	109.5	C3B—C10B—H10F	109.5
H10A—C10A—H10C	109.5	H10D—C10B—H10F	109.5
H10B—C10A—H10C	109.5	H10E—C10B—H10F	109.5
O4A—C11A—O3A	124.6 (3)	O4B—C11B—O3B	124.6 (3)
O4A—C11A—N1A	126.9 (3)	O4B—C11B—N1B	125.4 (3)
O3A—C11A—N1A	108.5 (2)	O3B—C11B—N1B	109.9 (3)
O3A—C12A—C13A	105.8 (2)	O3B—C12B—C13B	107.4 (3)
O3A—C12A—H12A	110.6	O3B—C12B—H12C	110.2
C13A—C12A—H12A	110.6	C13B—C12B—H12C	110.2
O3A—C12A—H12B	110.6	O3B—C12B—H12D	110.2
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C12A—C13A—H13B	109.5	C12B—C13B—H13F	109.5
H13A—C13A—H13B	109.5	H13D—C13B—H13F	109.5
C12A—C13A—H13C	109.5	C12B—C13B—H13E	109.5
H13A—C13A—H13C	109.5	H13D—C13B—H13E	109.5
H13B—C13A—H13C	109.5	H13F—C13B—H13E	109.5
C9A—O1A—C1A—O2A	−179.3 (3)	C9B—O1B—C1B—O2B	−179.9 (2)
C9A—O1A—C1A—C2A	−1.3 (5)	C9B—O1B—C1B—C2B	−0.9 (4)
O2A—C1A—C2A—C3A	177.7 (4)	O2B—C1B—C2B—C3B	179.9 (3)
O1A—C1A—C2A—C3A	0.0 (5)	O1B—C1B—C2B—C3B	1.0 (5)
C1A—C2A—C3A—C4A	0.4 (5)	C1B—C2B—C3B—C4B	−0.4 (5)
C1A—C2A—C3A—C10A	−179.1 (3)	C1B—C2B—C3B—C10B	−179.0 (3)
C2A—C3A—C4A—C9A	0.6 (5)	C2B—C3B—C4B—C9B	−0.2 (4)
C10A—C3A—C4A—C9A	−180.0 (3)	C10B—C3B—C4B—C9B	178.4 (3)
C2A—C3A—C4A—C5A	178.6 (3)	C2B—C3B—C4B—C5B	179.0 (3)
C10A—C3A—C4A—C5A	−1.9 (5)	C10B—C3B—C4B—C5B	−2.4 (5)
C9A—C4A—C5A—C6A	−0.4 (5)	C9B—C4B—C5B—C6B	−1.8 (4)
C3A—C4A—C5A—C6A	−178.5 (3)	C3B—C4B—C5B—C6B	179.0 (3)
C4A—C5A—C6A—C7A	1.2 (5)	C4B—C5B—C6B—C7B	0.7 (5)
C5A—C6A—C7A—C8A	−1.8 (5)	C5B—C6B—C7B—C8B	0.6 (5)
C5A—C6A—C7A—N1A	179.5 (3)	C5B—C6B—C7B—N1B	−179.1 (3)
C11A—N1A—C7A—C8A	8.8 (5)	C11B—N1B—C7B—C8B	−2.9 (5)
C11A—N1A—C7A—C6A	−172.6 (3)	C11B—N1B—C7B—C6B	176.8 (3)
C6A—C7A—C8A—C9A	1.6 (5)	C6B—C7B—C8B—C9B	−0.7 (4)
N1A—C7A—C8A—C9A	−179.8 (3)	N1B—C7B—C8B—C9B	179.0 (3)
C1A—O1A—C9A—C8A	−178.1 (3)	C7B—C8B—C9B—O1B	−179.8 (2)
C1A—O1A—C9A—C4A	2.3 (4)	C7B—C8B—C9B—C4B	−0.6 (5)
C7A—C8A—C9A—O1A	179.5 (3)	C1B—O1B—C9B—C8B	179.6 (3)
C7A—C8A—C9A—C4A	−0.9 (5)	C1B—O1B—C9B—C4B	0.3 (4)

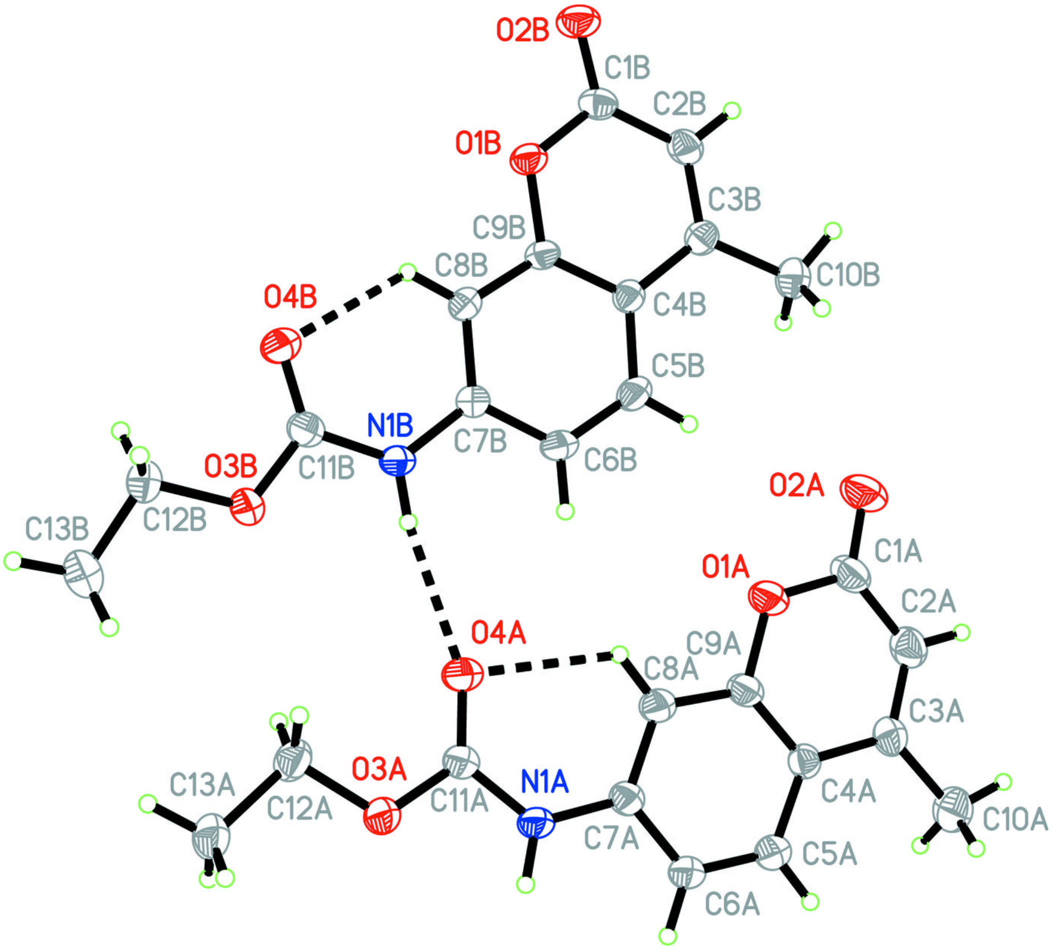
C5A—C4A—C9A—O1A	179.9 (3)	C5B—C4B—C9B—C8B	1.8 (4)
C3A—C4A—C9A—O1A	−1.9 (4)	C3B—C4B—C9B—C8B	−178.9 (3)
C5A—C4A—C9A—C8A	0.3 (5)	C5B—C4B—C9B—O1B	−179.0 (2)
C3A—C4A—C9A—C8A	178.5 (3)	C3B—C4B—C9B—O1B	0.3 (4)
C12A—O3A—C11A—O4A	1.1 (4)	C12B—O3B—C11B—O4B	−0.7 (5)
C12A—O3A—C11A—N1A	−179.1 (3)	C12B—O3B—C11B—N1B	178.8 (3)
C7A—N1A—C11A—O4A	−4.1 (5)	C7B—N1B—C11B—O4B	−4.2 (5)
C7A—N1A—C11A—O3A	176.1 (3)	C7B—N1B—C11B—O3B	176.3 (2)
C11A—O3A—C12A—C13A	178.2 (3)	C11B—O3B—C12B—C13B	167.5 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8A—H8A···O4A	0.93	2.28	2.881 (4)	122
C8B—H8B···O4B	0.93	2.19	2.799 (3)	122
N1B—H1B···O4A	0.86	2.25	3.104 (3)	170
N1A—H1A···O2B^{i}	0.86	2.04	2.878 (3)	164
C6A—H6A···O2B^{i}	0.93	2.48	3.243 (4)	140
C2A—H2A···O4B^{ii}	0.93	2.55	3.392 (4)	151
C13A—H13C···Cg1A^{iii}	0.96	2.79	3.637 (4)	147

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

Fig. 1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O3B
C11B
C6B
O2A
C13B
C12B
O1A
C1A
C2A
O4A
C9A
C8A
C3A
O3A
C13A
C12A
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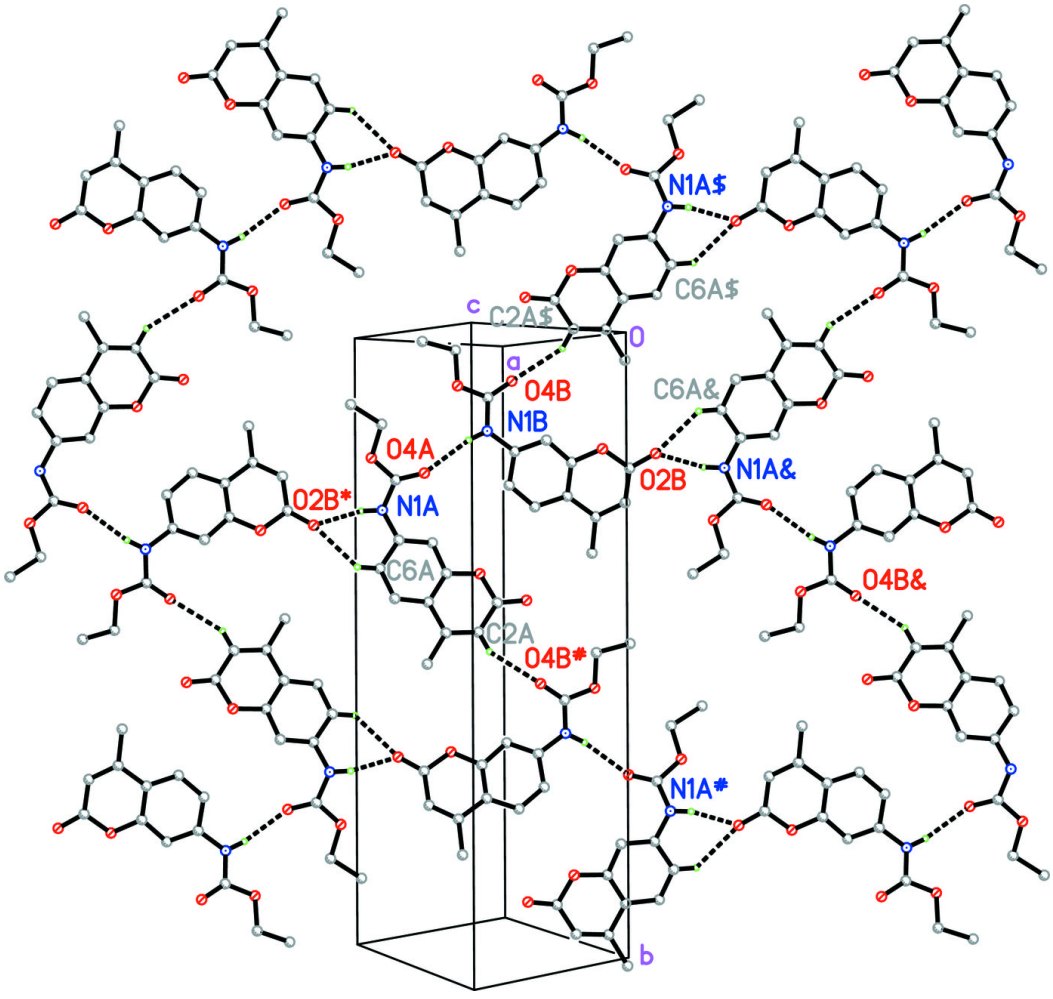
C4A
C10A
C7A
C5A
C6A

Fig. 2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C6A$
c
C2A$
0
a
O4B
C6A&
N1B
O4A
O2B
N1A&
O2B*
N1A
C6A
O4B&
C2A
O4B#
N1A#
b