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2,2-Dimethyl-2,3-dihydro-1-benzofuran-7-yl *N*-ethylcarbamate

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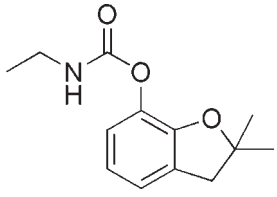
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.056; wR factor = 0.154; data-to-parameter ratio = 10.1.

The title compound, $C_{13}H_{17}NO_3$, crystallizes with two independent molecules in the asymmetric unit. In the crystal, N—H···O hydrogen bonds link the molecules, forming chains propagaiting in [100]. A weak C—H···O interaction also occurs.

Related literature

For background on insecticides related to the title compound, see: Tomlin (1994). For a related structure, see Xu *et al.* (2005).



Experimental

Crystal data

$C_{13}H_{17}NO_3$
M_r = 235.28
Orthorhombic, $P2_12_12_1$
a = 10.362 (2) Å
b = 13.962 (3) Å
c = 18.069 (4) Å
V = 2614.1 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.26 × 0.20 × 0.08 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.978, T_{max} = 0.993
20967 measured reflections
3256 independent reflections
2338 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.154
S = 1.08
3256 reflections
322 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O6	0.82 (4)	2.28 (4)	3.024 (4)	151 (3)
N2—H2···O2^{i}	0.84 (4)	2.19 (4)	2.985 (4)	156 (3)
C19—H19···O5^{ii}	0.93	2.48	3.269 (4)	143

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

Data collection: *CrystalClear* (Rigaku/MSC, 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* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5190).

References

Rigaku/MSC (2005). *CrystalClear* and *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tomlin, C. (1994). *The Pesticide Mannul. A World Compendium*, 10th ed., pp. 152–153. Bath: The British Crop Protection Council, The Bath Press.
Xu, L.-Z., Yu, G.-P. & Yang, S.-H. (2005). *Acta Cryst.* E**61**, o1924–o1926.

supplementary materials

Acta Cryst. (2009). E**65**, o2928 [**doi:10.1107/S1600536809044687**]

2,2-Dimethyl-2,3-dihydro-1-benzofuran-7-yl *N*-ethylcarbamate

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Comment

The title compound, (I), is an anologue to commercial Carbofuran, which is a popular carbamate insecticide (Tomlin, 1994). Herein, we present its single-crystal structure: it crystallizes with two independent molecules in the asymmetric unit (Figs. 1 & 2), and it has the same space group P212121 like Carbofuran reported previouly (Xu *et al.*, 2005). In the molecule shown in Fig 1, the dihedral angle between the carbamate plane O1/C11/O2/N1 and the benzo ring C5—C10 is 78.50 (5)°, and atom C1 deviates from the C4—C10/O3 plane with an angle of 0.167 (2) Å. In the other molecule, shown in Fig 2, the dihedral angle between the plane O5/O6/C24/N2 and the benzo ring C18—C23 is 79.87 (5)°, and atom C14 lies 0.175 (2)Å out of the plane C17—C23/O4. All these are similar to those reported in the literature (Xu *et al.*, 2005).

In the crystal structure, the two independent molecules in the asymmetric unit are linked by a strong N—H···O hydrogen bond, and each links another adjacent molecule by the N—H···O hydrogen bond. Besides, weak C—H···O H-bonding consolidates the packing (Table 1).

Experimental

The title compound was prepared by reaction of 2,3-dihydro-7-hydroxy-2,2-dimethylbenzofuran with ethylcarbamoyl chloride in 283 K, with a 85% yeild. Colourless prisms of (I) were obtained by evaporation from its ethanoic solution at room temperature.

Refinement

All C-bound H atoms were positioned geometrically and constrained to ride on their parent atoms [C—H distances are 0.93 and 0.97Å with U_{iso}(H) = 1.2 U_{eq}(C) for aromatic and CH_2 H atoms, 0.96Å with U_{iso} = 1.5U_{eq} (C) for CH_3 H atoms].

The position and isotropic displacement parameters of the NH H atoms were refined freely. In the absence of significant anomalous dispersion effects, Friedel pairs were merged.

Figures

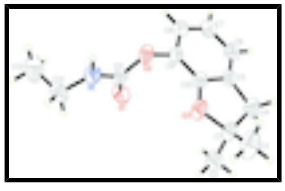

Fig. 1. One molecule in the asymmetric unit of (I), showing d isplacement ellipsoids drawn at the 30% probability level.

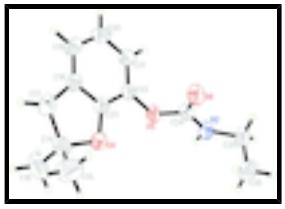

Fig. 2. The other molecule in the asymmetric unit of (I), showing displacement ellipsoids drawn at the 30% level.

2,2-Dimethyl-2,3-dihydro-1-benzofuran-7-yl *N*-ethylcarbamate

Crystal data

$C_{13}H_{17}NO_3$	F_{000} = 1008
M_r = 235.28	D_x = 1.196 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 5268 reflections
a = 10.362 (2) Å	θ = 1.8–27.1°
b = 13.962 (3) Å	μ = 0.09 mm^{-1}
c = 18.069 (4) Å	T = 293 K
V = 2614.1 (10) Å^3	Prism, colourless
Z = 8	0.26 × 0.20 × 0.08 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer	3256 independent reflections
Radiation source: rotating anode	2338 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.049
Detector resolution: 7.31 pixels mm^{-1}	θ_{max} = 27.2°
T = 293 K	θ_{min} = 1.8°
ω and φ scans	h = −10→13
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2005)	k = −17→17
T_{min} = 0.978, T_{max} = 0.993	l = −21→23
20967 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.056	$w = 1/[\sigma^2(F_o^2) + (0.083P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.154	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.08	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
3256 reflections	$\Delta\rho_{min}$ = −0.14 e Å^{-3}

322 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.029 (3)

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}
O1	0.9284 (3)	0.3257 (2)	0.01889 (15)	0.0801 (8)
O2	0.9054 (2)	0.50324 (18)	0.14502 (19)	0.0855 (9)
O3	0.7620 (2)	0.38179 (17)	0.13896 (15)	0.0725 (7)
O4	0.3318 (3)	0.79075 (16)	0.11613 (14)	0.0758 (7)
O5	0.2451 (2)	0.60024 (16)	0.08020 (14)	0.0688 (7)
O6	0.4027 (2)	0.51258 (19)	0.13447 (16)	0.0812 (8)
N1	0.6932 (3)	0.5302 (2)	0.12561 (19)	0.0709 (8)
N2	0.1916 (3)	0.4821 (2)	0.15411 (17)	0.0660 (8)
C1	1.0315 (4)	0.2815 (3)	−0.0269 (2)	0.0762 (10)
C2	1.1341 (5)	0.3563 (3)	−0.0360 (3)	0.1080 (16)
H2A	1.0989	0.4107	−0.0614	0.162*
H2B	1.2044	0.3303	−0.0641	0.162*
H2C	1.1647	0.3757	0.0119	0.162*
C3	0.9693 (6)	0.2508 (4)	−0.0979 (3)	0.127 (2)
H3A	0.9036	0.2043	−0.0875	0.190*
H3B	1.0332	0.2232	−0.1298	0.190*
H3C	0.9312	0.3054	−0.1217	0.190*
C4	1.0807 (5)	0.1960 (3)	0.0181 (2)	0.0931 (13)
H4A	1.0517	0.1360	−0.0033	0.112*
H4B	1.1743	0.1958	0.0205	0.112*
C5	1.0226 (4)	0.2114 (2)	0.0936 (2)	0.0729 (10)
C6	1.0378 (5)	0.1657 (3)	0.1605 (3)	0.0881 (12)
H6	1.0972	0.1161	0.1652	0.106*
C7	0.9655 (5)	0.1933 (3)	0.2198 (3)	0.0947 (13)
H7	0.9766	0.1626	0.2650	0.114*
C8	0.8759 (4)	0.2665 (3)	0.2138 (2)	0.0840 (12)
H8	0.8262	0.2840	0.2545	0.101*
C9	0.8606 (3)	0.3134 (2)	0.1472 (2)	0.0679 (9)

C10	0.9355 (3)	0.2866 (3)	0.0874 (2)	0.0662 (9)
C11	0.7964 (3)	0.4767 (2)	0.13721 (19)	0.0612 (8)
C12	0.6965 (4)	0.6335 (3)	0.1279 (3)	0.0935 (13)
H12A	0.6404	0.6590	0.0897	0.112*
H12B	0.7836	0.6554	0.1179	0.112*
C13	0.6545 (7)	0.6699 (4)	0.2009 (4)	0.136 (2)
H13A	0.5680	0.6489	0.2107	0.205*
H13B	0.6571	0.7387	0.2008	0.205*
H13C	0.7111	0.6459	0.2386	0.205*
C14	0.3854 (4)	0.8896 (3)	0.1104 (2)	0.0773 (10)
C15	0.4434 (6)	0.9120 (5)	0.1854 (3)	0.1229 (18)
H15A	0.3771	0.9094	0.2225	0.184*
H15B	0.4806	0.9749	0.1845	0.184*
H15C	0.5091	0.8658	0.1967	0.184*
C16	0.2739 (5)	0.9540 (3)	0.0911 (4)	0.127 (2)
H16A	0.2339	0.9318	0.0463	0.190*
H16B	0.3047	1.0182	0.0840	0.190*
H16C	0.2119	0.9532	0.1306	0.190*
C17	0.4848 (4)	0.8855 (3)	0.0467 (2)	0.0856 (11)
H17A	0.4712	0.9373	0.0119	0.103*
H17B	0.5723	0.8891	0.0656	0.103*
C18	0.4599 (3)	0.7908 (2)	0.0114 (2)	0.0647 (9)
C19	0.5074 (3)	0.7502 (3)	−0.0522 (2)	0.0731 (10)
H19	0.5640	0.7840	−0.0824	0.088*
C20	0.4699 (4)	0.6584 (3)	−0.0705 (2)	0.0833 (11)
H20	0.5022	0.6299	−0.1132	0.100*
C21	0.3851 (3)	0.6086 (3)	−0.0262 (2)	0.0729 (10)
H21	0.3603	0.5469	−0.0395	0.088*
C22	0.3368 (3)	0.6491 (2)	0.03737 (19)	0.0605 (8)
C23	0.3733 (3)	0.7411 (2)	0.05565 (19)	0.0584 (8)
C24	0.2899 (3)	0.5289 (2)	0.12496 (18)	0.0564 (8)
C25	0.2109 (4)	0.4110 (3)	0.2117 (2)	0.0874 (12)
H25A	0.2936	0.3798	0.2046	0.105*
H25B	0.1439	0.3627	0.2084	0.105*
C26	0.2072 (7)	0.4562 (6)	0.2855 (3)	0.166 (3)
H26A	0.2746	0.5031	0.2890	0.249*
H26B	0.2196	0.4081	0.3228	0.249*
H26C	0.1250	0.4866	0.2926	0.249*
H1	0.622 (4)	0.504 (2)	0.1267 (18)	0.056 (10)*
H2	0.117 (4)	0.503 (3)	0.144 (2)	0.072 (11)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0670 (16)	0.0924 (17)	0.0808 (17)	0.0327 (14)	0.0019 (13)	−0.0074 (14)
O2	0.0409 (12)	0.0715 (15)	0.144 (2)	−0.0031 (11)	−0.0087 (15)	0.0076 (15)
O3	0.0450 (12)	0.0685 (14)	0.1038 (18)	0.0029 (11)	0.0026 (12)	−0.0232 (13)
O4	0.0812 (16)	0.0653 (13)	0.0811 (16)	−0.0084 (13)	0.0173 (13)	0.0042 (12)

O5	0.0460 (11)	0.0603 (12)	0.1001 (18)	0.0058 (10)	0.0084 (12)	0.0259 (12)
O6	0.0417 (12)	0.0868 (16)	0.115 (2)	0.0025 (11)	−0.0052 (13)	0.0285 (16)
N1	0.0387 (15)	0.0758 (19)	0.098 (2)	0.0029 (14)	−0.0018 (15)	0.0000 (17)
N2	0.0449 (15)	0.0672 (16)	0.086 (2)	−0.0045 (14)	−0.0054 (14)	0.0208 (15)
C1	0.064 (2)	0.084 (2)	0.081 (2)	0.023 (2)	0.0069 (18)	−0.012 (2)
C2	0.091 (3)	0.090 (3)	0.143 (4)	0.013 (3)	0.032 (3)	−0.007 (3)
C3	0.122 (4)	0.151 (5)	0.107 (4)	0.045 (4)	−0.030 (3)	−0.049 (3)
C4	0.084 (3)	0.092 (3)	0.104 (3)	0.034 (2)	0.010 (2)	−0.003 (2)
C5	0.064 (2)	0.0597 (18)	0.094 (3)	0.0074 (17)	0.0040 (19)	−0.0047 (19)
C6	0.095 (3)	0.062 (2)	0.107 (3)	0.016 (2)	0.003 (3)	0.004 (2)
C7	0.110 (4)	0.072 (2)	0.102 (3)	0.010 (3)	0.013 (3)	0.008 (2)
C8	0.095 (3)	0.070 (2)	0.086 (3)	−0.006 (2)	0.019 (2)	−0.006 (2)
C9	0.0520 (18)	0.0592 (18)	0.093 (3)	−0.0028 (15)	0.0054 (18)	−0.0162 (18)
C10	0.0541 (19)	0.0673 (19)	0.077 (2)	0.0050 (16)	−0.0014 (17)	−0.0106 (18)
C11	0.0451 (17)	0.0670 (19)	0.072 (2)	−0.0005 (15)	0.0029 (15)	−0.0018 (17)
C12	0.063 (2)	0.082 (2)	0.135 (4)	0.007 (2)	0.000 (3)	0.029 (3)
C13	0.147 (5)	0.094 (3)	0.168 (5)	0.004 (4)	−0.023 (5)	−0.044 (3)
C14	0.073 (2)	0.0630 (19)	0.096 (3)	−0.0086 (18)	0.004 (2)	−0.0076 (19)
C15	0.124 (4)	0.136 (4)	0.108 (4)	−0.031 (4)	0.006 (3)	−0.024 (3)
C16	0.091 (3)	0.076 (3)	0.213 (6)	0.008 (3)	0.011 (4)	0.015 (3)
C17	0.084 (3)	0.077 (2)	0.096 (3)	−0.021 (2)	0.006 (2)	0.011 (2)
C18	0.0516 (18)	0.0676 (19)	0.075 (2)	−0.0058 (16)	−0.0013 (16)	0.0140 (18)
C19	0.0522 (19)	0.092 (3)	0.075 (2)	−0.0105 (19)	0.0040 (18)	0.012 (2)
C20	0.063 (2)	0.102 (3)	0.085 (3)	−0.004 (2)	0.015 (2)	−0.012 (2)
C21	0.058 (2)	0.069 (2)	0.092 (3)	−0.0068 (17)	0.0035 (19)	−0.002 (2)
C22	0.0447 (16)	0.0575 (17)	0.079 (2)	0.0017 (14)	0.0040 (16)	0.0155 (16)
C23	0.0476 (17)	0.0603 (17)	0.0673 (19)	−0.0004 (14)	0.0007 (15)	0.0105 (15)
C24	0.0436 (16)	0.0517 (15)	0.074 (2)	−0.0015 (13)	−0.0072 (15)	0.0029 (16)
C25	0.070 (2)	0.087 (3)	0.106 (3)	−0.003 (2)	0.000 (2)	0.037 (2)
C26	0.187 (7)	0.215 (7)	0.096 (4)	0.112 (6)	0.043 (4)	0.060 (4)

Geometric parameters (Å, °)

O1—C10	1.356 (5)	C8—H8	0.9300
O1—C1	1.486 (4)	C9—C10	1.381 (5)
O2—C11	1.198 (4)	C12—C13	1.478 (7)
O3—C11	1.373 (4)	C12—H12A	0.9700
O3—C9	1.406 (4)	C12—H12B	0.9700
O4—C23	1.364 (4)	C13—H13A	0.9600
O4—C14	1.491 (4)	C13—H13B	0.9600
O5—C24	1.365 (4)	C13—H13C	0.9600
O5—C22	1.402 (4)	C14—C16	1.506 (6)
O6—C24	1.203 (4)	C14—C15	1.515 (6)
N1—C11	1.321 (4)	C14—C17	1.545 (6)
N1—C12	1.443 (5)	C15—H15A	0.9600
N1—H1	0.82 (4)	C15—H15B	0.9600
N2—C24	1.320 (4)	C15—H15C	0.9600
N2—C25	1.451 (5)	C16—H16A	0.9600
N2—H2	0.84 (4)	C16—H16B	0.9600

C1—C3	1.497 (6)	C16—H16C	0.9600
C1—C2	1.498 (6)	C17—C18	1.491 (5)
C1—C4	1.532 (6)	C17—H17A	0.9700
C2—H2A	0.9600	C17—H17B	0.9700
C2—H2B	0.9600	C18—C19	1.372 (5)
C2—H2C	0.9600	C18—C23	1.389 (4)
C3—H3A	0.9600	C19—C20	1.379 (6)
C3—H3B	0.9600	C19—H19	0.9300
C3—H3C	0.9600	C20—C21	1.377 (5)
C4—C5	1.506 (6)	C20—H20	0.9300
C4—H4A	0.9700	C21—C22	1.375 (5)
C4—H4B	0.9700	C21—H21	0.9300
C5—C6	1.376 (6)	C22—C23	1.379 (5)
C5—C10	1.389 (5)	C25—C26	1.475 (7)
C6—C7	1.364 (6)	C25—H25A	0.9700
C6—H6	0.9300	C25—H25B	0.9700
C7—C8	1.385 (6)	C26—H26A	0.9600
C7—H7	0.9300	C26—H26B	0.9600
C8—C9	1.379 (6)	C26—H26C	0.9600
C10—O1—C1	107.6 (3)	C12—C13—H13B	109.5
C11—O3—C9	118.0 (2)	H13A—C13—H13B	109.5
C23—O4—C14	107.3 (3)	C12—C13—H13C	109.5
C24—O5—C22	116.8 (2)	H13A—C13—H13C	109.5
C11—N1—C12	122.8 (3)	H13B—C13—H13C	109.5
C11—N1—H1	118 (2)	O4—C14—C16	106.5 (3)
C12—N1—H1	117 (2)	O4—C14—C15	106.0 (4)
C24—N2—C25	121.2 (3)	C16—C14—C15	112.8 (4)
C24—N2—H2	117 (3)	O4—C14—C17	105.4 (3)
C25—N2—H2	121 (3)	C16—C14—C17	111.2 (4)
O1—C1—C3	106.6 (3)	C15—C14—C17	114.2 (4)
O1—C1—C2	106.3 (3)	C14—C15—H15A	109.5
C3—C1—C2	114.3 (4)	C14—C15—H15B	109.5
O1—C1—C4	105.5 (3)	H15A—C15—H15B	109.5
C3—C1—C4	112.0 (4)	C14—C15—H15C	109.5
C2—C1—C4	111.4 (4)	H15A—C15—H15C	109.5
C1—C2—H2A	109.5	H15B—C15—H15C	109.5
C1—C2—H2B	109.5	C14—C16—H16A	109.5
H2A—C2—H2B	109.5	C14—C16—H16B	109.5
C1—C2—H2C	109.5	H16A—C16—H16B	109.5
H2A—C2—H2C	109.5	C14—C16—H16C	109.5
H2B—C2—H2C	109.5	H16A—C16—H16C	109.5
C1—C3—H3A	109.5	H16B—C16—H16C	109.5
C1—C3—H3B	109.5	C18—C17—C14	103.7 (3)
H3A—C3—H3B	109.5	C18—C17—H17A	111.0
C1—C3—H3C	109.5	C14—C17—H17A	111.0
H3A—C3—H3C	109.5	C18—C17—H17B	111.0
H3B—C3—H3C	109.5	C14—C17—H17B	111.0
C5—C4—C1	103.7 (3)	H17A—C17—H17B	109.0
C5—C4—H4A	111.0	C19—C18—C23	120.5 (3)

C1—C4—H4A	111.0	C19—C18—C17	131.5 (3)
C5—C4—H4B	111.0	C23—C18—C17	108.0 (3)
C1—C4—H4B	111.0	C18—C19—C20	118.9 (3)
H4A—C4—H4B	109.0	C18—C19—H19	120.6
C6—C5—C10	119.7 (4)	C20—C19—H19	120.6
C6—C5—C4	133.1 (3)	C21—C20—C19	120.7 (4)
C10—C5—C4	107.2 (3)	C21—C20—H20	119.7
C7—C6—C5	119.8 (4)	C19—C20—H20	119.7
C7—C6—H6	120.1	C22—C21—C20	120.7 (4)
C5—C6—H6	120.1	C22—C21—H21	119.7
C6—C7—C8	121.0 (4)	C20—C21—H21	119.7
C6—C7—H7	119.5	C21—C22—C23	118.9 (3)
C8—C7—H7	119.5	C21—C22—O5	120.6 (3)
C9—C8—C7	119.7 (4)	C23—C22—O5	120.4 (3)
C9—C8—H8	120.2	O4—C23—C22	125.4 (3)
C7—C8—H8	120.2	O4—C23—C18	114.3 (3)
C8—C9—C10	119.3 (3)	C22—C23—C18	120.3 (3)
C8—C9—O3	119.8 (3)	O6—C24—N2	126.8 (3)
C10—C9—O3	120.6 (3)	O6—C24—O5	123.6 (3)
O1—C10—C9	125.1 (3)	N2—C24—O5	109.6 (3)
O1—C10—C5	114.4 (3)	N2—C25—C26	110.6 (4)
C9—C10—C5	120.5 (3)	N2—C25—H25A	109.5
O2—C11—N1	127.4 (3)	C26—C25—H25A	109.5
O2—C11—O3	122.7 (3)	N2—C25—H25B	109.5
N1—C11—O3	109.9 (3)	C26—C25—H25B	109.5
N1—C12—C13	111.3 (4)	H25A—C25—H25B	108.1
N1—C12—H12A	109.4	C25—C26—H26A	109.5
C13—C12—H12A	109.4	C25—C26—H26B	109.5
N1—C12—H12B	109.4	H26A—C26—H26B	109.5
C13—C12—H12B	109.4	C25—C26—H26C	109.5
H12A—C12—H12B	108.0	H26A—C26—H26C	109.5
C12—C13—H13A	109.5	H26B—C26—H26C	109.5
C10—O1—C1—C3	131.3 (4)	C23—O4—C14—C16	107.5 (4)
C10—O1—C1—C2	−106.3 (4)	C23—O4—C14—C15	−132.1 (4)
C10—O1—C1—C4	12.1 (4)	C23—O4—C14—C17	−10.7 (4)
O1—C1—C4—C5	−12.4 (4)	O4—C14—C17—C18	11.4 (4)
C3—C1—C4—C5	−128.0 (4)	C16—C14—C17—C18	−103.6 (4)
C2—C1—C4—C5	102.6 (4)	C15—C14—C17—C18	127.4 (4)
C1—C4—C5—C6	−173.3 (4)	C14—C17—C18—C19	172.3 (4)
C1—C4—C5—C10	8.7 (5)	C14—C17—C18—C23	−8.4 (4)
C10—C5—C6—C7	1.2 (6)	C23—C18—C19—C20	−1.4 (5)
C4—C5—C6—C7	−176.6 (5)	C17—C18—C19—C20	177.9 (4)
C5—C6—C7—C8	0.5 (7)	C18—C19—C20—C21	0.7 (6)
C6—C7—C8—C9	−1.1 (7)	C19—C20—C21—C22	−0.4 (6)
C7—C8—C9—C10	−0.1 (6)	C20—C21—C22—C23	0.8 (5)
C7—C8—C9—O3	174.4 (3)	C20—C21—C22—O5	176.5 (3)
C11—O3—C9—C8	106.3 (4)	C24—O5—C22—C21	78.1 (4)
C11—O3—C9—C10	−79.3 (4)	C24—O5—C22—C23	−106.3 (3)
C1—O1—C10—C9	175.9 (3)	C14—O4—C23—C22	−174.8 (3)

C1—O1—C10—C5	−6.9 (4)	C14—O4—C23—C18	5.7 (4)
C8—C9—C10—O1	178.9 (4)	C21—C22—C23—O4	179.0 (3)
O3—C9—C10—O1	4.4 (5)	O5—C22—C23—O4	3.3 (5)
C8—C9—C10—C5	1.8 (5)	C21—C22—C23—C18	−1.6 (5)
O3—C9—C10—C5	−172.6 (3)	O5—C22—C23—C18	−177.2 (3)
C6—C5—C10—O1	−179.7 (4)	C19—C18—C23—O4	−178.6 (3)
C4—C5—C10—O1	−1.4 (5)	C17—C18—C23—O4	2.0 (4)
C6—C5—C10—C9	−2.4 (6)	C19—C18—C23—C22	1.9 (5)
C4—C5—C10—C9	175.9 (3)	C17—C18—C23—C22	−177.5 (3)
C12—N1—C11—O2	−6.4 (7)	C25—N2—C24—O6	9.2 (6)
C12—N1—C11—O3	173.9 (3)	C25—N2—C24—O5	−171.5 (3)
C9—O3—C11—O2	−2.7 (5)	C22—O5—C24—O6	7.3 (5)
C9—O3—C11—N1	176.9 (3)	C22—O5—C24—N2	−172.0 (3)
C11—N1—C12—C13	−96.9 (5)	C24—N2—C25—C26	89.0 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O6	0.82 (4)	2.28 (4)	3.024 (4)	151 (3)
N2—H2···O2^{i}	0.84 (4)	2.19 (4)	2.985 (4)	156 (3)
C19—H19···O5^{ii}	0.93	2.48	3.269 (4)	143

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

Fig. 1

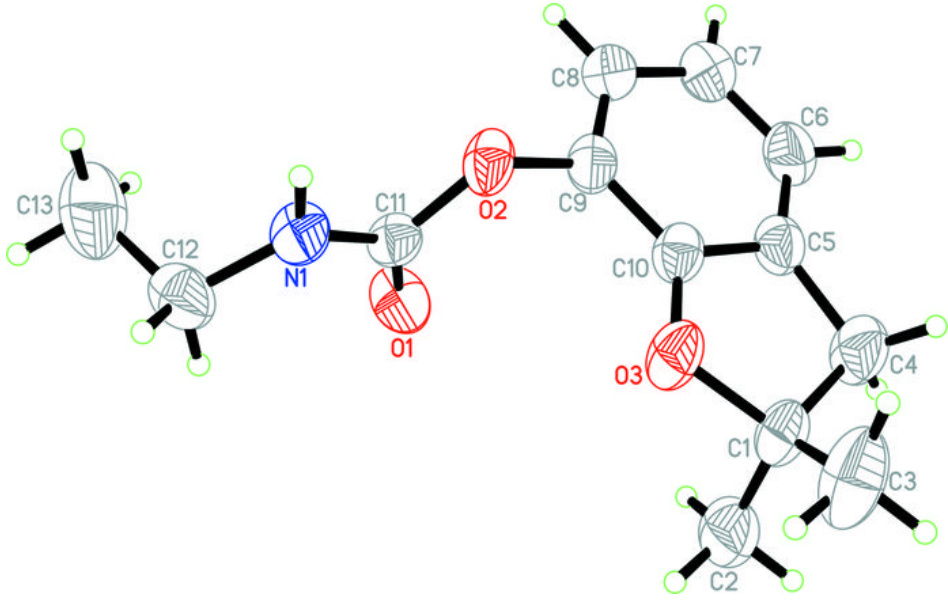

Fig. 2