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2,2-Dimethyl-1,3-benzodioxol-4-yl *N*-methylcarbamate

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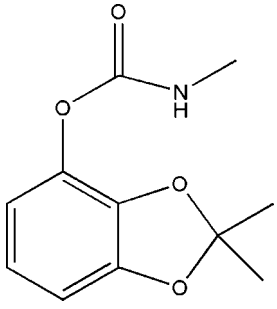
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.098; data-to-parameter ratio = 13.7.

In the title compound, $C_{11}H_{13}NO_4$, the two fused rings are almost coplanar, making a dihedral angle of 3.02 (8)°. In the crystal, chains are formed parallel to [010] through N—H···O hydrogen bonds between the amine and carbonyl groups.

Related literature

For benzodioxole derivatives, see: Ullrich *et al.* (2004); Gates & Gillon (1974); Arndt & Franke (1977); Joshi *et al.* (2005); Jae *et al.* (2001); Leite *et al.* (2004).



Experimental

Crystal data

$C_{11}H_{13}NO_4$
M_r = 223.22
Monoclinic, $P2_1/n$
a = 9.505 (6) Å
b = 9.669 (7) Å
c = 12.355 (8) Å
β = 94.326 (11)°
V = 1132.2 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 K
0.24 × 0.18 × 0.16 mm

Data collection

Siemens SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
T_{min} = 0.976, T_{max} = 0.984
5692 measured reflections
2003 independent reflections
1615 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.098
S = 1.03
2003 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.01	2.819 (3)	157

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

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

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2301).

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supplementary materials

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2,2-Dimethyl-1,3-benzodioxol-4-yl *N*-methylcarbamate

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Comment

Benzodioxoles derivatives can be used as inhibitors of mono-oxygenase enzymes (Ullrich *et al.*, 2004), pesticides or pesticide intermediates (Gates & Gillon, 1974), herbicides (Arndt & Franke, 1977), antioxidants (Joshi *et al.*, 2005), antimicrobials (Jae *et al.*, 2001) and medicines (Leite *et al.*, 2004). As a part of our continuing interest in the synthesis of benzodioxole derivatives, we have isolated the title compound from the reaction of isocyanatomethane, triethylamine and 2,2-dimethylbenzo[*d*][1,3]dioxol-4-ol, as colorless crystals suitable for X-ray analysis.

As shown in Fig. 1, the molecule is built-up of a five-membered ring and a six-membered ring. Atoms C4, O3, O4, C5, and C6 are coplanar, which is illustrated clearly by the torsion angle O3—C4—C5—C6 [-179.39 (14)°], C3—C4—C5—O4 [177.83 (13)°], C3—C4—C5—C6 [-0.5 (2)°], and O3—C4—C5—O4 [-1.11 (17)°]. In the crystal structure, amino groups and carbonyl groups are involved in the hydrogen-bond network. Carbonyl atom O1 acts as an acceptor, forming the intramolecular hydrogen bonds depicted in Fig. 2.

Experimental

The title compound was synthesized from a mixture of triethylamine (2 mmol, 0.2 g), isocyanatomethane (0.11 mol, 6.3 g) and 2,2-dimethylbenzo[*d*][1,3]dioxol-4-ol (0.1 mol, 16.6 g). The resulting compound was dissolved in 20 ml of ethanol and 2 ml of water, and refluxed for 10 min. The system was cooled to room temperature and colorless crystals were collected after two weeks.

Refinement

Amine H atom H1 was found in a difference map, and its position fixed. Other H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.93 (aromatic CH) or 0.96 Å (methyl CH_3), with U_{iso}(H) values fixed to 1.2 or 1.5 times U_{eq} of the parent atoms.

Figures

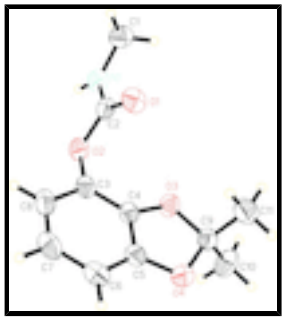

Fig. 1. The asymmetric unit of the title molecule with atom labels, showing 40% probability displacement ellipsoids.

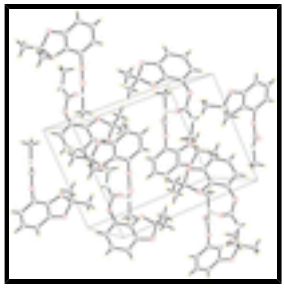

Fig. 2. Part of the crystal structure, with hydrogen bonds shown as dashed lines.

2,2-Dimethyl-1,3-benzodioxol-4-yl *N*-methylcarbamate

Crystal data

$C_{11}H_{13}NO_4$	$F(000) = 472$
$M_r = 223.22$	$D_x = 1.310$ Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 408 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.505$ (6) Å	Cell parameters from 2406 reflections
$b = 9.669$ (7) Å	$\theta = 2.7$–$26.7°$
$c = 12.355$ (8) Å	$\mu = 0.10$ mm^{-1}
$\beta = 94.326$ (11)°	$T = 298$ K
$V = 1132.2$ (13) Å^3	Block, colorless
$Z = 4$	0.24 × 0.18 × 0.16 mm

Data collection

Siemens SMART APEX CCD area-detector diffractometer	2003 independent reflections
Radiation source: fine-focus sealed tube	1615 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.023$
φ and ω scans	$\theta_{max} = 25.1°$, $\theta_{min} = 2.7°$
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)	$h = -11 \rightarrow 10$
$T_{min} = 0.976$, $T_{max} = 0.984$	$k = -11 \rightarrow 11$
5692 measured reflections	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.098$	$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.1766P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
2003 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
146 parameters	$\Delta\rho_{min} = -0.13$ e Å^{-3}

0 restraints

0 constraints

Primary atom site location: structure-invariant direct methods

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

Extinction coefficient: 0.035 (3)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.78908 (12)	0.22049 (10)	0.73401 (10)	0.0666 (3)
O2	0.91170 (11)	0.04289 (10)	0.81309 (9)	0.0614 (3)
O3	1.07164 (11)	0.14292 (11)	0.63309 (8)	0.0624 (3)
O4	1.27901 (11)	0.26676 (13)	0.65853 (9)	0.0707 (4)
N1	0.69122 (14)	0.01063 (12)	0.74902 (10)	0.0589 (4)
H1	0.7052	−0.0723	0.7726	0.071*
C1	0.55627 (18)	0.04608 (19)	0.69670 (16)	0.0748 (5)
H1A	0.5096	0.1103	0.7412	0.112*
H1B	0.4999	−0.0360	0.6864	0.112*
H1C	0.5689	0.0876	0.6275	0.112*
C2	0.79399 (16)	0.10118 (14)	0.76185 (11)	0.0491 (4)
C3	1.03134 (16)	0.12582 (15)	0.82661 (12)	0.0537 (4)
C4	1.10295 (15)	0.16741 (14)	0.74063 (11)	0.0505 (4)
C5	1.22535 (16)	0.24235 (16)	0.75644 (12)	0.0554 (4)
C6	1.28072 (19)	0.27957 (18)	0.85695 (14)	0.0674 (5)
H6	1.3632	0.3313	0.8667	0.081*
C7	1.2084 (2)	0.23669 (19)	0.94372 (14)	0.0730 (5)
H7	1.2435	0.2597	1.0137	0.088*
C8	1.0863 (2)	0.16103 (17)	0.92966 (13)	0.0670 (5)
H8	1.0402	0.1333	0.9898	0.080*
C9	1.17105 (17)	0.22577 (17)	0.57665 (13)	0.0602 (4)
C10	1.2336 (2)	0.1358 (2)	0.49544 (16)	0.0884 (6)
H10A	1.3010	0.1879	0.4581	0.133*
H10B	1.1604	0.1029	0.4441	0.133*
H10C	1.2798	0.0586	0.5316	0.133*
C11	1.0989 (2)	0.35311 (19)	0.53297 (16)	0.0795 (5)
H11A	1.0580	0.4014	0.5908	0.119*
H11B	1.0260	0.3280	0.4785	0.119*
H11C	1.1662	0.4119	0.5015	0.119*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0786 (8)	0.0347 (6)	0.0860 (8)	0.0026 (5)	0.0022 (6)	0.0060 (5)
O2	0.0715 (7)	0.0454 (6)	0.0679 (7)	0.0011 (5)	0.0085 (5)	0.0166 (5)
O3	0.0668 (7)	0.0740 (7)	0.0466 (6)	−0.0115 (5)	0.0049 (5)	−0.0029 (5)
O4	0.0564 (6)	0.0921 (9)	0.0635 (7)	−0.0089 (6)	0.0032 (5)	0.0079 (6)
N1	0.0698 (8)	0.0357 (6)	0.0725 (9)	−0.0011 (6)	0.0139 (7)	0.0037 (6)
C1	0.0679 (11)	0.0654 (11)	0.0919 (13)	−0.0016 (9)	0.0109 (10)	0.0018 (9)
C2	0.0669 (9)	0.0353 (7)	0.0470 (8)	0.0064 (7)	0.0154 (7)	−0.0002 (6)

C3	0.0642 (9)	0.0450 (8)	0.0520 (9)	0.0082 (7)	0.0046 (7)	0.0053 (6)
C4	0.0594 (9)	0.0461 (8)	0.0454 (8)	0.0054 (7)	0.0009 (7)	−0.0001 (6)
C5	0.0547 (9)	0.0559 (8)	0.0547 (9)	0.0069 (7)	−0.0012 (7)	0.0018 (7)
C6	0.0635 (10)	0.0667 (11)	0.0695 (11)	0.0067 (8)	−0.0119 (9)	−0.0060 (8)
C7	0.0874 (13)	0.0752 (11)	0.0536 (10)	0.0159 (10)	−0.0133 (9)	−0.0085 (8)
C8	0.0877 (12)	0.0655 (10)	0.0479 (9)	0.0156 (9)	0.0055 (8)	0.0062 (7)
C9	0.0618 (9)	0.0676 (10)	0.0516 (9)	−0.0041 (8)	0.0069 (7)	0.0054 (7)
C10	0.1012 (15)	0.0889 (14)	0.0794 (13)	−0.0004 (11)	0.0356 (11)	−0.0040 (10)
C11	0.0805 (12)	0.0732 (12)	0.0827 (13)	0.0011 (10)	−0.0081 (10)	0.0106 (9)

Geometric parameters (Å, °)

O1—C2	1.2037 (18)	C4—C5	1.372 (2)
O2—C2	1.3650 (18)	C5—C6	1.360 (2)
O2—C3	1.3911 (19)	C6—C7	1.380 (3)
O3—C4	1.3603 (19)	C6—H6	0.9300
O3—C9	1.4560 (19)	C7—C8	1.372 (3)
O4—C5	1.368 (2)	C7—H7	0.9300
O4—C9	1.441 (2)	C8—H8	0.9300
N1—C2	1.312 (2)	C9—C10	1.485 (2)
N1—C1	1.434 (2)	C9—C11	1.490 (2)
N1—H1	0.8600	C10—H10A	0.9600
C1—H1A	0.9600	C10—H10B	0.9600
C1—H1B	0.9600	C10—H10C	0.9600
C1—H1C	0.9600	C11—H11A	0.9600
C3—C4	1.365 (2)	C11—H11B	0.9600
C3—C8	1.382 (2)	C11—H11C	0.9600
C2—O2—C3	116.85 (11)	C7—C6—H6	121.7
C4—O3—C9	105.66 (12)	C8—C7—C6	121.86 (16)
C5—O4—C9	106.26 (12)	C8—C7—H7	119.1
C2—N1—C1	121.84 (14)	C6—C7—H7	119.1
C2—N1—H1	119.1	C7—C8—C3	120.35 (16)
C1—N1—H1	119.1	C7—C8—H8	119.8
N1—C1—H1A	109.5	C3—C8—H8	119.8
N1—C1—H1B	109.5	O4—C9—O3	105.58 (12)
H1A—C1—H1B	109.5	O4—C9—C10	109.58 (15)
N1—C1—H1C	109.5	O3—C9—C10	107.98 (14)
H1A—C1—H1C	109.5	O4—C9—C11	108.10 (14)
H1B—C1—H1C	109.5	O3—C9—C11	109.30 (14)
O1—C2—N1	126.32 (15)	C10—C9—C11	115.83 (16)
O1—C2—O2	122.75 (14)	C9—C10—H10A	109.5
N1—C2—O2	110.93 (13)	C9—C10—H10B	109.5
C4—C3—C8	117.95 (16)	H10A—C10—H10B	109.5
C4—C3—O2	121.84 (13)	C9—C10—H10C	109.5
C8—C3—O2	120.08 (14)	H10A—C10—H10C	109.5
O3—C4—C3	128.60 (14)	H10B—C10—H10C	109.5
O3—C4—C5	110.59 (13)	C9—C11—H11A	109.5
C3—C4—C5	120.80 (14)	C9—C11—H11B	109.5
C6—C5—O4	128.11 (16)	H11A—C11—H11B	109.5

C6—C5—C4	122.36 (15)	C9—C11—H11C	109.5
O4—C5—C4	109.50 (13)	H11A—C11—H11C	109.5
C5—C6—C7	116.66 (17)	H11B—C11—H11C	109.5
C5—C6—H6	121.7		
C1—N1—C2—O1	0.8 (2)	C3—C4—C5—C6	−0.5 (2)
C1—N1—C2—O2	−179.66 (13)	O3—C4—C5—O4	−1.11 (17)
C3—O2—C2—O1	−3.81 (19)	C3—C4—C5—O4	177.83 (13)
C3—O2—C2—N1	176.62 (12)	O4—C5—C6—C7	−177.16 (15)
C2—O2—C3—C4	−68.19 (17)	C4—C5—C6—C7	0.8 (2)
C2—O2—C3—C8	116.03 (15)	C5—C6—C7—C8	−0.4 (3)
C9—O3—C4—C3	172.56 (15)	C6—C7—C8—C3	−0.3 (3)
C9—O3—C4—C5	−8.60 (16)	C4—C3—C8—C7	0.6 (2)
C8—C3—C4—O3	178.46 (14)	O2—C3—C8—C7	176.56 (14)
O2—C3—C4—O3	2.6 (2)	C5—O4—C9—O3	−15.26 (16)
C8—C3—C4—C5	−0.3 (2)	C5—O4—C9—C10	−131.32 (15)
O2—C3—C4—C5	−176.13 (13)	C5—O4—C9—C11	101.61 (15)
C9—O4—C5—C6	−171.39 (16)	C4—O3—C9—O4	14.55 (15)
C9—O4—C5—C4	10.45 (17)	C4—O3—C9—C10	131.69 (14)
O3—C4—C5—C6	−179.39 (14)	C4—O3—C9—C11	−101.52 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.01	2.819 (3)	157

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

Fig. 1

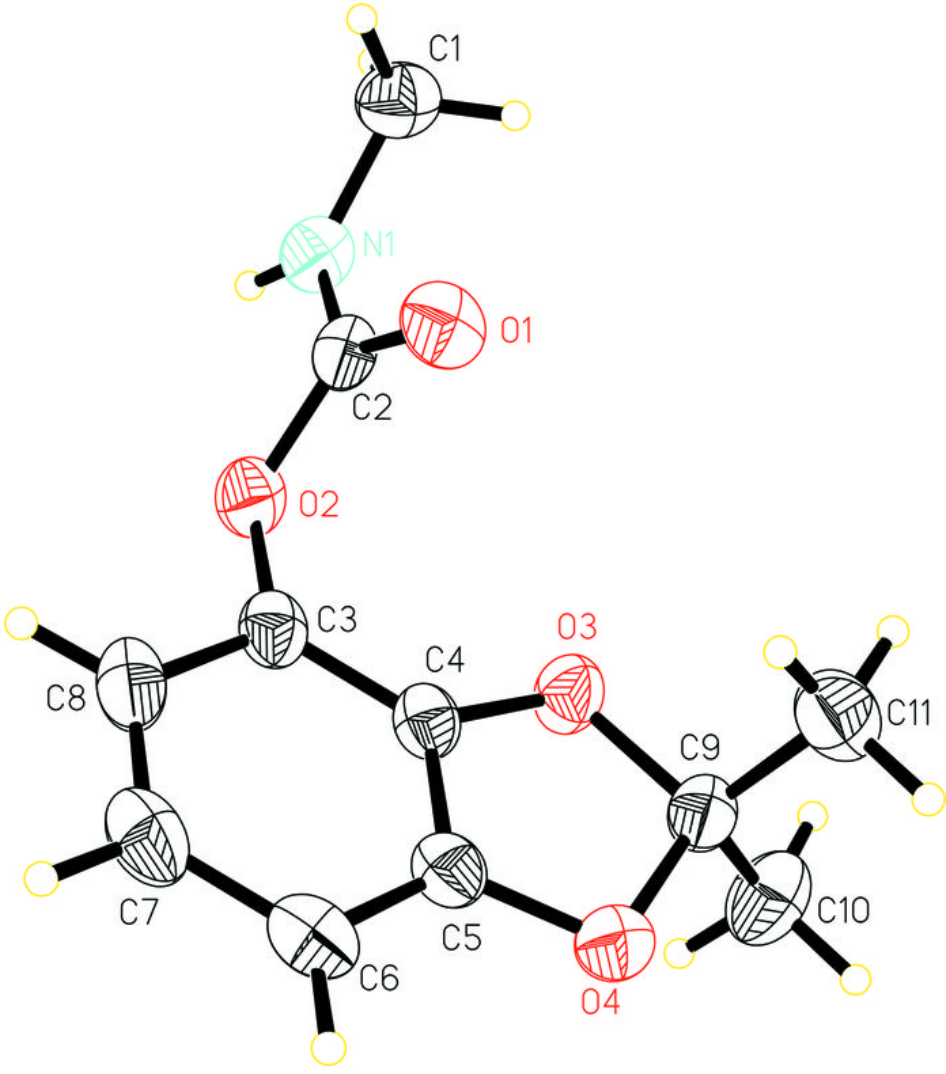

Fig. 2