Crystal structure of 3-(3,4-dimethyl-anilino)-2-benzofuran-1(3H)-one

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In the title compound, $C_{16}H_{15}NO_2$, the 2-benzofuran-1(3H)-one and 3,4-dimethylaniline fragments are oriented with a dihedral angle of 89.12 (5)°. N—H···O hydrogen-bond interactions join molecules into C(6) chains propagating along the a axis. In addition, there are π–π stacking interactions between the 2-benzofuranone benzene rings [centroid–centroid distance = 3.7870 (13) Å] and C—H···π interactions between one of the methyl groups and the 3,4-dimethylaniline benzene ring.

1. Related literature

For related crystal structures, see: Li et al. (2009); Odabaşoğlu & Büyükgüngör (2006a,b, 2007a,b). For graph-set notation, see: Bernstein et al. (1995).
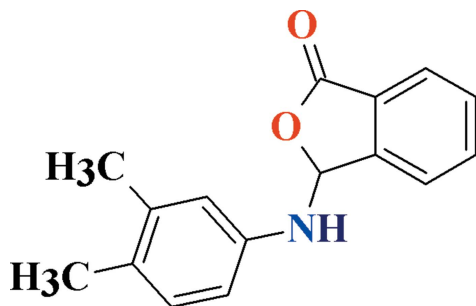
2. Experimental

2.1. Crystal data

$C_{16}H_{15}NO_2$

$M_r = 253.29$

Orthorhombic, Pbca
$a = 7.3386$ (7) Å
$b = 14.9133$ (11) Å
$c = 24.3322$ (18) Å
$V = 2663.0$ (4) Å^3

$Z = 8$
Mo Kα radiation
$\mu = 0.08$ mm^{-1}
$T = 296$ K
$0.38 \times 0.23 \times 0.16$ mm

2.2. Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2005)
$T_{min} = 0.970$, $T_{max} = 0.988$

20839 measured reflections
2906 independent reflections
1660 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.144$
$S = 1.02$
2906 reflections

174 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

Cg3 is the centroid of the C9—C14

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2[i]	0.86	2.31	3.025 (2)	141
C15—H15B···Cg3[ii]	0.96	2.88	3.661 (3)	139

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL2014 (Sheldrick, 2015); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012) and PLATON (Spek, 2009); software used to prepare material for publication: WinGX (Farrugia, 2012) and PLATON.

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supporting information

Crystal structure of 3-(3,4-dimethylanilino)-2-benzofuran-1(3*H*)-one

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S1. Comment

The crystal structures of 3-anilinoisobenzofuran-1(3*H*)-one (Odabaşoğlu & Büyükgüngör, 2006*a*), 3-(2,6-dimethyl-anilino)isobenzofuran-1(3*H*) -one (Odabaşoğlu & Büyükgüngör, 2006*b*), 3-(4-methylanilino)isobenzo furan-1(3*H*)-one (Odabaşoğlu & Büyükgüngör, 2007*a*), 3-(2- (hydroxymethyl)anilino)isobenzofuran-1(3*H*)-one (Odabaşoğlu & Büyükgüngör, 2007*b*), and 3-((3-oxo-1,3-dihydroisobenzofuran-1-yl)amino) benzoic acid Li *et al.*, 2009) have been published which are related to the title compound (I, Fig. 1). The title compound was synthesized for the biological studies and for the preparation of further derivitives.

The benzofuran ring A (C1–C8/O1/O2) and the 3,4-dmethylaniline group B (C9–C16/N1) are planar with r. m. s. deviation of 0.0209 and 0.0101 Å, respectively. The dihedral angle between A/B fragments is 89.12 (5)°. Intermolecular hydrogen bond of N—H···O type generates *C* (6) chains (Bernstein *et al.*, 1995) along the crystallographic *a* axis (Table 1, Fig. 2). The π–π interactions are observed between the 2-benzofuranone fragments [*Cg*1—*Cg*2[i] 3.6204 (12) Å; *Cg*1—*Cg*1[i] 3.8138 (13) Å; *Cg*2—*Cg*2[i] 3.7870 (13) Å, *Cg*1 - centroid of C1/C2/C7/C8/O1, *Cg*2 - centroid of C2–C7); i = - 1/2 + *x*, *y*, 1/2 - *z*]. In addition there are also C—H···π interactions (Table 1).

S2. Experimental

Equimolar quantities of 3,4-dimethylaniline (0.605 g. 5 mmol) and 2-formylbenzoic acid (0.751 g, 5 mmol) were stirred in methanol for 2 h. The solution was kept at room temperature for crystallization which afforded light brown needles after 48 h.

S3. Refinement

The H atoms were positioned geometrically (C–H = 0.93–0.96 Å, N—H= 0.86 Å) and refined as riding on their carriers with $U_{iso}(H) = xU_{eq}(C, N)$, where x = 1.5 for methyl and x =1.2 for other H-atoms.
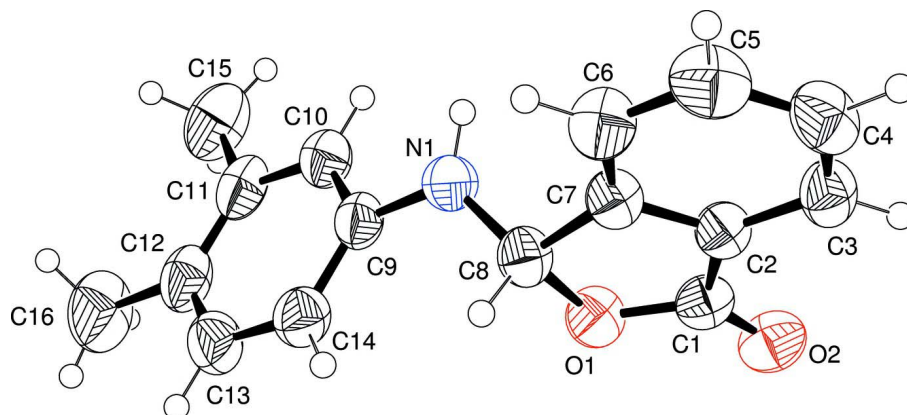
Figure 1

Molecular structure with the atom numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H-atoms are shown by small circles of arbitrary radii.
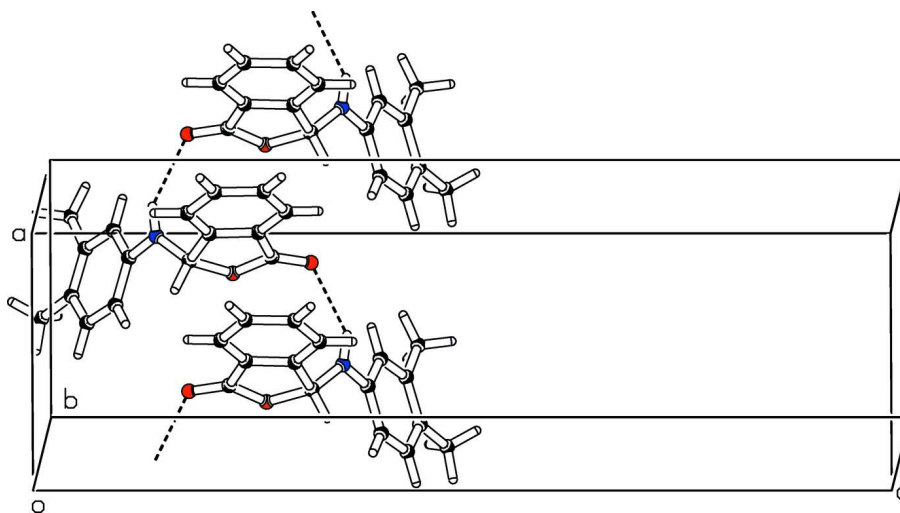
Figure 2

The chains of molecules along the *a* axis *via* N—H···O hydrogen bond (*PLATON*; Spek, 2009).

3-(3,4-Dimethylanilino)-2-benzofuran-1(3*H*)-one

Crystal data

$C_{16}H_{15}NO_2$	$D_x = 1.264$ Mg m^{-3}
$M_r = 253.29$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, *Pbca*	Cell parameters from 4689 reflections
$a = 7.3386$ (7) Å	$\theta = 1.4–27.0°$
$b = 14.9133$ (11) Å	$\mu = 0.08$ mm^{-1}
$c = 24.3322$ (18) Å	$T = 296$ K
$V = 2663.0$ (4) Å^3	Needle, light brown
$Z = 8$	$0.38 \times 0.23 \times 0.16$ mm
$F(000) = 1072$	

Data collection

Bruker Kappa APEXII CCD diffractometer	20839 measured reflections
Radiation source: fine-focus sealed tube	2906 independent reflections
Graphite monochromator	1660 reflections with $I > 2\sigma(I)$
Detector resolution: 7.80 pixels mm⁻¹	$R_{int} = 0.042$
ω scans	$\theta_{max} = 27.0°$, $\theta_{min} = 2.7°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -5 \rightarrow 9$
$T_{min} = 0.970$, $T_{max} = 0.988$	$k = -19 \rightarrow 18$
	$l = -31 \rightarrow 31$

Detector resolution: 7.80 pixels mm^{-1}

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.144$	
$S = 1.02$	H-atom parameters constrained
2906 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2 + 0.3907P]$ where $P = (F_o^2 + 2F_c^2)/3$
174 parameters	
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.27$ e Å⁻³
	$\Delta\rho_{min} = -0.25$ e Å⁻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 (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
O1	0.80336 (19)	0.14473 (8)	0.23068 (5)	0.0528 (4)
O2	0.8396 (2)	0.16231 (9)	0.32129 (5)	0.0614 (4)
N1	0.9466 (2)	0.15132 (10)	0.14175 (6)	0.0533 (5)
H1	1.0572	0.1688	0.1371	0.064*
C1	0.8541 (3)	0.19202 (12)	0.27513 (7)	0.0457 (5)
C2	0.9241 (3)	0.27938 (11)	0.25746 (7)	0.0424 (5)
C3	0.9848 (3)	0.35141 (12)	0.28845 (8)	0.0523 (5)
H3	0.9871	0.3488	0.3266	0.063*
C4	1.0416 (3)	0.42698 (13)	0.26083 (9)	0.0610 (6)
H4	1.0827	0.4765	0.2805	0.073*
C5	1.0381 (3)	0.42990 (13)	0.20393 (10)	0.0651 (6)
H5	1.0775	0.4815	0.1860	0.078*
C6	0.9774 (3)	0.35782 (12)	0.17306 (8)	0.0575 (6)
H6	0.9761	0.3602	0.1349	0.069*
C7	0.9189 (3)	0.28247 (11)	0.20076 (7)	0.0443 (5)
C8	0.8370 (3)	0.19769 (12)	0.17912 (7)	0.0472 (5)

H8	0.7199	0.2115	0.1617	0.057*
C9	0.8834 (3)	0.07697 (11)	0.11132 (7)	0.0463 (5)
C10	1.0086 (3)	0.01250 (12)	0.09499 (7)	0.0521 (5)
H10	1.1299	0.0189	0.1054	0.062*
C11	0.9584 (4)	−0.06119 (12)	0.06359 (7)	0.0572 (6)
C12	0.7769 (4)	−0.07110 (13)	0.04754 (7)	0.0618 (6)
C13	0.6529 (3)	−0.00635 (14)	0.06404 (8)	0.0632 (6)
H13	0.5317	−0.0124	0.0535	0.076*
C14	0.7027 (3)	0.06721 (13)	0.09569 (7)	0.0553 (6)
H14	0.6161	0.1093	0.1063	0.066*
C15	1.1015 (4)	−0.12910 (15)	0.04791 (10)	0.0846 (8)
H15A	1.2147	−0.1140	0.0654	0.127*
H15B	1.1175	−0.1289	0.0087	0.127*
H15C	1.0635	−0.1877	0.0596	0.127*
C16	0.7133 (4)	−0.15104 (15)	0.01424 (10)	0.0953 (9)
H16A	0.5847	−0.1462	0.0076	0.143*
H16B	0.7378	−0.2052	0.0342	0.143*
H16C	0.7769	−0.1524	−0.0202	0.143*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0623 (10)	0.0370 (7)	0.0592 (8)	−0.0047 (6)	0.0027 (7)	−0.0033 (6)
O2	0.0646 (11)	0.0614 (9)	0.0581 (8)	0.0045 (7)	0.0074 (7)	0.0143 (7)
N1	0.0515 (11)	0.0518 (9)	0.0566 (9)	−0.0076 (8)	0.0071 (8)	−0.0179 (7)
C1	0.0457 (12)	0.0391 (10)	0.0524 (11)	0.0075 (9)	0.0045 (9)	0.0000 (9)
C2	0.0434 (12)	0.0356 (9)	0.0481 (10)	0.0067 (9)	−0.0002 (9)	−0.0037 (8)
C3	0.0577 (14)	0.0450 (11)	0.0542 (10)	0.0084 (10)	−0.0029 (10)	−0.0122 (9)
C4	0.0638 (16)	0.0385 (11)	0.0809 (15)	−0.0005 (10)	−0.0047 (12)	−0.0147 (10)
C5	0.0725 (17)	0.0377 (11)	0.0851 (15)	−0.0050 (11)	0.0028 (13)	0.0061 (10)
C6	0.0714 (16)	0.0458 (11)	0.0552 (11)	−0.0007 (11)	0.0001 (11)	0.0060 (9)
C7	0.0481 (12)	0.0355 (9)	0.0493 (10)	0.0038 (9)	−0.0016 (9)	−0.0025 (8)
C8	0.0524 (13)	0.0405 (9)	0.0487 (10)	0.0021 (9)	−0.0026 (9)	−0.0040 (8)
C9	0.0561 (14)	0.0444 (10)	0.0385 (9)	−0.0047 (10)	0.0014 (9)	−0.0042 (8)
C10	0.0591 (14)	0.0495 (11)	0.0476 (10)	0.0010 (11)	0.0013 (10)	−0.0049 (9)
C11	0.0845 (18)	0.0456 (11)	0.0417 (10)	0.0002 (12)	0.0104 (11)	−0.0029 (8)
C12	0.0906 (19)	0.0489 (12)	0.0460 (10)	−0.0145 (13)	0.0064 (12)	−0.0084 (9)
C13	0.0706 (17)	0.0646 (13)	0.0543 (11)	−0.0185 (13)	−0.0032 (11)	−0.0076 (10)
C14	0.0604 (15)	0.0512 (11)	0.0542 (11)	−0.0034 (11)	0.0041 (10)	−0.0084 (9)
C15	0.119 (2)	0.0601 (13)	0.0749 (14)	0.0175 (14)	0.0172 (16)	−0.0143 (12)
C16	0.135 (3)	0.0685 (15)	0.0821 (15)	−0.0301 (16)	0.0022 (17)	−0.0282 (13)

Geometric parameters (Å, º)

O1—C1	1.344 (2)	C8—H8	0.9800
O1—C8	1.503 (2)	C9—C14	1.387 (3)
O2—C1	1.212 (2)	C9—C10	1.388 (3)
N1—C8	1.397 (2)	C10—C11	1.388 (3)

N1—C9	1.412 (2)	C10—H10	0.9300
N1—H1	0.8600	C11—C12	1.395 (3)
C1—C2	1.465 (3)	C11—C15	1.509 (3)
C2—C7	1.381 (2)	C12—C13	1.386 (3)
C2—C3	1.386 (2)	C12—C16	1.515 (3)
C3—C4	1.377 (3)	C13—C14	1.389 (3)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.385 (3)	C14—H14	0.9300
C4—H4	0.9300	C15—H15A	0.9600
C5—C6	1.385 (3)	C15—H15B	0.9600
C5—H5	0.9300	C15—H15C	0.9600
C6—C7	1.379 (2)	C16—H16A	0.9600
C6—H6	0.9300	C16—H16B	0.9600
C7—C8	1.496 (2)	C16—H16C	0.9600
C1—O1—C8	110.53 (13)	C14—C9—C10	118.79 (17)
C8—N1—C9	122.76 (17)	C14—C9—N1	122.70 (17)
C8—N1—H1	118.6	C10—C9—N1	118.48 (19)
C9—N1—H1	118.6	C9—C10—C11	122.0 (2)
O2—C1—O1	121.98 (17)	C9—C10—H10	119.0
O2—C1—C2	128.89 (17)	C11—C10—H10	119.0
O1—C1—C2	109.12 (15)	C10—C11—C12	119.4 (2)
C7—C2—C3	121.78 (16)	C10—C11—C15	119.1 (2)
C7—C2—C1	108.25 (15)	C12—C11—C15	121.5 (2)
C3—C2—C1	129.96 (17)	C13—C12—C11	118.15 (18)
C4—C3—C2	117.78 (18)	C13—C12—C16	120.0 (2)
C4—C3—H3	121.1	C11—C12—C16	121.8 (2)
C2—C3—H3	121.1	C12—C13—C14	122.5 (2)
C3—C4—C5	120.53 (18)	C12—C13—H13	118.7
C3—C4—H4	119.7	C14—C13—H13	118.7
C5—C4—H4	119.7	C9—C14—C13	119.1 (2)
C6—C5—C4	121.57 (19)	C9—C14—H14	120.4
C6—C5—H5	119.2	C13—C14—H14	120.4
C4—C5—H5	119.2	C11—C15—H15A	109.5
C7—C6—C5	117.87 (18)	C11—C15—H15B	109.5
C7—C6—H6	121.1	H15A—C15—H15B	109.5
C5—C6—H6	121.1	C11—C15—H15C	109.5
C6—C7—C2	120.46 (16)	H15A—C15—H15C	109.5
C6—C7—C8	129.93 (16)	H15B—C15—H15C	109.5
C2—C7—C8	109.55 (15)	C12—C16—H16A	109.5
N1—C8—C7	114.58 (17)	C12—C16—H16B	109.5
N1—C8—O1	112.19 (14)	H16A—C16—H16B	109.5
C7—C8—O1	102.49 (13)	C12—C16—H16C	109.5
N1—C8—H8	109.1	H16A—C16—H16C	109.5
C7—C8—H8	109.1	H16B—C16—H16C	109.5
O1—C8—H8	109.1		
C8—O1—C1—O2	−179.85 (17)	C2—C7—C8—N1	−123.94 (17)

C8—O1—C1—C2	0.2 (2)	C6—C7—C8—O1	−179.40 (19)
O2—C1—C2—C7	178.4 (2)	C2—C7—C8—O1	−2.2 (2)
O1—C1—C2—C7	−1.6 (2)	C1—O1—C8—N1	124.55 (17)
O2—C1—C2—C3	−2.8 (4)	C1—O1—C8—C7	1.1 (2)
O1—C1—C2—C3	177.13 (18)	C8—N1—C9—C14	30.1 (3)
C7—C2—C3—C4	−0.4 (3)	C8—N1—C9—C10	−151.93 (17)
C1—C2—C3—C4	−179.03 (19)	C14—C9—C10—C11	0.0 (3)
C2—C3—C4—C5	−0.2 (3)	N1—C9—C10—C11	−178.05 (16)
C3—C4—C5—C6	0.2 (3)	C9—C10—C11—C12	0.3 (3)
C4—C5—C6—C7	0.4 (3)	C9—C10—C11—C15	−179.31 (18)
C5—C6—C7—C2	−1.0 (3)	C10—C11—C12—C13	−0.3 (3)
C5—C6—C7—C8	176.0 (2)	C15—C11—C12—C13	179.33 (18)
C3—C2—C7—C6	1.0 (3)	C10—C11—C12—C16	−178.75 (18)
C1—C2—C7—C6	179.92 (18)	C15—C11—C12—C16	0.9 (3)
C3—C2—C7—C8	−176.51 (17)	C11—C12—C13—C14	0.0 (3)
C1—C2—C7—C8	2.4 (2)	C16—C12—C13—C14	178.46 (19)
C9—N1—C8—C7	−171.10 (16)	C10—C9—C14—C13	−0.3 (3)
C9—N1—C8—O1	72.6 (2)	N1—C9—C14—C13	177.65 (16)
C6—C7—C8—N1	58.8 (3)	C12—C13—C14—C9	0.3 (3)

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the C9—C14

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2ⁱ	0.86	2.31	3.025 (2)	141
C15—H15B···Cg3ⁱⁱ	0.96	2.88	3.661 (3)	139

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