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N-(2-Ethoxyphenyl)formamide

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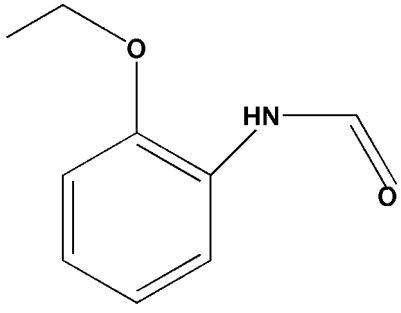
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.120; data-to-parameter ratio = 17.8.

The title compound, $C_9H_{11}NO_2$, was obtained as an unexpected product in an attempt to synthesize a triazene ligand. The title molecule is almost planar, with the formamide and ethoxy groups oriented at 2.7 (3) and 12.9 (2)°, respectively, with respect to the mean plane of the benzene ring. In the crystal, molecules are linked by intermolecular N—H···O hydrogen bonds, forming a chain along the *a* axis. Weak C—H···π interactions with an H···π distance of 2.78 Å reinforce the crystal packing, resulting in a three-dimensional network.

Related literature

For preparation of several trizene compounds in our laboratory, see: Melardi *et al.* (2011). For similar crystal structures, see: Landman *et al.* (2011); Chitanda *et al.* (2008); Hu *et al.* (2010).



Experimental

Crystal data

$C_9H_{11}NO_2$
M_r = 165.19
Orthorhombic, *Pbca*
a = 7.9079 (4) Å
b = 14.1253 (6) Å
c = 15.9555 (7) Å
V = 1782.25 (14) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.45 × 0.23 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.671, T_{max} = 0.746
8725 measured reflections
1961 independent reflections
1248 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.120
S = 1.03
1961 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

Cg is the centroid of the C3–C9 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.24	2.9741 (18)	144
C2—H2A···Cg^{ii}	0.97	2.78	3.5853 (19)	141

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

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: *SHELXL97* (Sheldrick, 2008); molecular graphics: XPW in *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: PV2505).

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supplementary materials

Acta Cryst. (2012). E**68**, o505 [**doi:10.1107/S160053681200205X**]

N-(2-Ethoxyphenyl)formamide

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Comment

The preparation of several trizene compounds as ligands in our laboratory has already been reported (Melardi *et al.*, 2011). However, the title compound was formed as an unexpected product in an attempt for the synthesis of a triazene ligand, 1-(2-methylphenyl)-3(2-ethoxyphenyl)triazene). In this article, we report the synthesis and crystal structure of the title compound.

The title molecule (Fig. 1) is almost planar with formamide and ethoxy groups oriented at 2.7 (3) and 12.9 (2)°, respectively, with respect to the mean-plane of the benzene ring. The bond lengths and angles in the title molecule are in accord with the corresponding bond lengths and angles reported in a few similar structures (Landman *et al.*, 2011; Chitanda *et al.*, 2008; Hu *et al*., 2010).

In the crystal structure molecules are linked by intermolecular N—H···O hydrogen bonds (Table 1) to form a chain along the *a*-axis. Weak edge-to-face C—H···*Cg*1 stacking interaction between an ethoxy hydrogen and a benzene ring with H···π distance of 2.78 Å, (*Cg*1 is the center of benzene ring atoms C3/C4/C6—C9) reinforce the crystal packing resulting in a three-dimensional network (Fig. 2).

Experimental

The title compound was obtained as an unexpected product in an attempt for the synthesis of a triazene ligand, 1-(2-methylphenyl)-3(2-ethoxyphenyl)triazene). A 100 ml flask was charged with 10 g of ice and 15 ml of water and then cooled to 273 K in an ice-bath. To this were added 2-methylaniline (0.215 g, 2 mmol), hydrochloric acid (36.5%, 2 mmol) and 2 ml water. To this solution was then added a solution containing $NaNO_2$ (0.16 g, 2 mmol) in 2 ml water during a 15 min period. After mixing for 15 min, the obtained solution was added to a solution of o-phenetidin (0.261 ml, 2 mmol), 2 ml methanol and 2 ml water. After that a solution containing sodium acetate (2.95 g, 36 mmol) in 10 ml water was added. After mixing for 24 h the colorless material was filtered off and dissolved in DMSO. By recrystallization from DMSO, the crystals of the title compound were obtained instead of the expected triazene.

Refinement

The H atoms were placed in calculated positions and refined as riding, with N—H = 0.86 Å and C—H = 0.93, 0.96 and 0.97 Å for aryl, methy and methylene type H-atoms, respectively, with U_{iso}(H) = 1.2–1.5 U_{eq}(C/N).

Figures

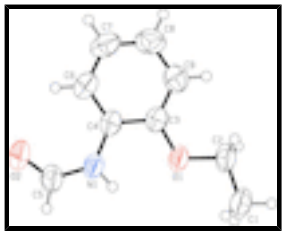

Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

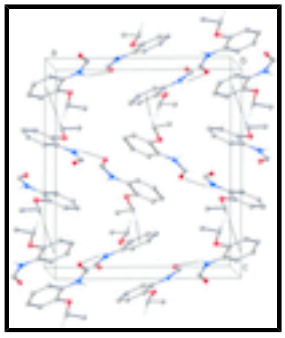

Fig. 2. A view of the N—-H···O hydrogen bonds and and C—H···π interactions (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen-bonding were omitted for clarity.

N-(2-Ethoxyphenyl)formamide

Crystal data

$C_9H_{11}NO_2$	$F(000) = 704$
$M_r = 165.19$	$D_x = 1.231$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 2150 reflections
$a = 7.9079$ (4) Å	θ = 2.6–23.1°
$b = 14.1253$ (6) Å	μ = 0.09 mm^{-1}
$c = 15.9555$ (7) Å	$T = 296$ K
$V = 1782.25$ (14) Å^3	Cubic, colourless
$Z = 8$	0.45 × 0.23 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer	1961 independent reflections
Radiation source: fine-focus sealed tube	1248 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.024$
φ and ω scans	$\theta_{max} = 27.1°$, $\theta_{min} = 3.2°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)	$h = -10 \rightarrow 10$
$T_{min} = 0.671$, $T_{max} = 0.746$	$k = -18 \rightarrow 17$
8725 measured reflections	$l = -20 \rightarrow 15$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.120$	H-atom parameters constrained
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.278P]$ where $P = (F_o^2 + 2F_c^2)/3$
1961 reflections	$(\Delta/\sigma)_{max} < 0.001$
110 parameters	$\Delta\rho_{max} = 0.12$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.14$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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}
O1	0.14029 (12)	0.40133 (8)	0.12756 (7)	0.0571 (3)
N1	−0.12270 (15)	0.33831 (10)	0.04588 (8)	0.0525 (4)
H1	−0.0300	0.3086	0.0561	0.063*
O2	−0.37570 (15)	0.31796 (10)	−0.02102 (9)	0.0808 (4)
C1	0.3874 (2)	0.33886 (16)	0.18902 (13)	0.0842 (7)
H1A	0.3183	0.2875	0.2085	0.126*
H1B	0.4759	0.3507	0.2288	0.126*
H1C	0.4361	0.3226	0.1358	0.126*
C2	0.2815 (2)	0.42535 (14)	0.17963 (11)	0.0653 (5)
H2A	0.2425	0.4468	0.2340	0.078*
H2B	0.3469	0.4758	0.1540	0.078*
C3	0.00875 (19)	0.46374 (11)	0.12280 (10)	0.0489 (4)
C4	−0.13409 (18)	0.43045 (11)	0.07964 (9)	0.0466 (4)
C5	−0.23581 (19)	0.29108 (14)	0.00042 (10)	0.0597 (5)
H5	−0.2045	0.2307	−0.0170	0.072*
C6	−0.2759 (2)	0.48701 (12)	0.07355 (11)	0.0585 (5)
H6	−0.3704	0.4656	0.0446	0.070*
C7	−0.2773 (2)	0.57530 (14)	0.11042 (14)	0.0732 (6)

H7	−0.3740	0.6126	0.1075	0.088*
C8	−0.1370 (3)	0.60824 (13)	0.15134 (14)	0.0784 (6)
H8	−0.1384	0.6683	0.1751	0.094*
C9	0.0067 (2)	0.55295 (13)	0.15765 (12)	0.0661 (5)
H9	0.1017	0.5759	0.1853	0.079*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0371 (6)	0.0752 (8)	0.0591 (7)	0.0031 (5)	−0.0080 (5)	−0.0113 (5)
N1	0.0351 (6)	0.0705 (9)	0.0520 (8)	0.0038 (6)	−0.0041 (6)	−0.0075 (7)
O2	0.0456 (7)	0.0982 (10)	0.0987 (10)	0.0009 (7)	−0.0234 (6)	−0.0143 (8)
C1	0.0558 (11)	0.1186 (17)	0.0781 (13)	0.0172 (11)	−0.0227 (10)	−0.0244 (12)
C2	0.0391 (8)	0.0894 (13)	0.0675 (11)	−0.0060 (9)	−0.0073 (8)	−0.0142 (10)
C3	0.0410 (8)	0.0582 (9)	0.0476 (9)	−0.0031 (7)	0.0054 (7)	0.0057 (7)
C4	0.0390 (7)	0.0587 (10)	0.0421 (8)	−0.0028 (7)	0.0041 (6)	0.0076 (7)
C5	0.0450 (9)	0.0784 (12)	0.0556 (10)	−0.0038 (9)	−0.0016 (8)	−0.0094 (8)
C6	0.0457 (9)	0.0660 (11)	0.0638 (10)	0.0035 (8)	−0.0006 (8)	0.0135 (9)
C7	0.0614 (12)	0.0597 (11)	0.0984 (15)	0.0127 (10)	0.0058 (11)	0.0182 (10)
C8	0.0793 (14)	0.0480 (11)	0.1079 (17)	−0.0010 (10)	0.0074 (12)	0.0047 (10)
C9	0.0584 (11)	0.0632 (11)	0.0768 (13)	−0.0131 (9)	0.0000 (9)	0.0013 (9)

Geometric parameters (Å, °)

O1—C3	1.3656 (18)	C3—C9	1.377 (2)
O1—C2	1.4328 (18)	C3—C4	1.404 (2)
N1—C5	1.331 (2)	C4—C6	1.380 (2)
N1—C4	1.411 (2)	C5—H5	0.9300
N1—H1	0.8600	C6—C7	1.379 (3)
O2—C5	1.2185 (19)	C6—H6	0.9300
C1—C2	1.488 (3)	C7—C8	1.369 (3)
C1—H1A	0.9600	C7—H7	0.9300
C1—H1B	0.9600	C8—C9	1.383 (3)
C1—H1C	0.9600	C8—H8	0.9300
C2—H2A	0.9700	C9—H9	0.9300
C2—H2B	0.9700		
C3—O1—C2	118.24 (12)	C6—C4—C3	119.61 (15)
C5—N1—C4	128.85 (14)	C6—C4—N1	123.97 (14)
C5—N1—H1	115.6	C3—C4—N1	116.40 (13)
C4—N1—H1	115.6	O2—C5—N1	127.37 (18)
C2—C1—H1A	109.5	O2—C5—H5	116.3
C2—C1—H1B	109.5	N1—C5—H5	116.3
H1A—C1—H1B	109.5	C7—C6—C4	120.02 (17)
C2—C1—H1C	109.5	C7—C6—H6	120.0
H1A—C1—H1C	109.5	C4—C6—H6	120.0
H1B—C1—H1C	109.5	C8—C7—C6	120.28 (18)
O1—C2—C1	107.60 (14)	C8—C7—H7	119.9
O1—C2—H2A	110.2	C6—C7—H7	119.9

C1—C2—H2A	110.2	C7—C8—C9	120.60 (18)
O1—C2—H2B	110.2	C7—C8—H8	119.7
C1—C2—H2B	110.2	C9—C8—H8	119.7
H2A—C2—H2B	108.5	C3—C9—C8	119.78 (17)
O1—C3—C9	125.23 (15)	C3—C9—H9	120.1
O1—C3—C4	115.08 (14)	C8—C9—H9	120.1
C9—C3—C4	119.68 (15)		
C3—O1—C2—C1	167.77 (15)	C4—N1—C5—O2	−0.8 (3)
C2—O1—C3—C9	6.7 (2)	C3—C4—C6—C7	−0.6 (2)
C2—O1—C3—C4	−172.08 (14)	N1—C4—C6—C7	178.25 (15)
O1—C3—C4—C6	178.00 (13)	C4—C6—C7—C8	1.7 (3)
C9—C3—C4—C6	−0.8 (2)	C6—C7—C8—C9	−1.2 (3)
O1—C3—C4—N1	−0.97 (19)	O1—C3—C9—C8	−177.42 (16)
C9—C3—C4—N1	−179.82 (14)	C4—C3—C9—C8	1.3 (3)
C5—N1—C4—C6	3.4 (3)	C7—C8—C9—C3	−0.3 (3)
C5—N1—C4—C3	−177.67 (15)		

Hydrogen-bond geometry (Å, °)

Cg is the centroid of the C3–C9 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.86	2.20	2.6108 (16)	109.
N1—H1···O2^{i}	0.86	2.24	2.9741 (18)	144.
C2—H2A···Cg^{ii}	0.97	2.78	3.5853 (19)	141

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

Fig. 1

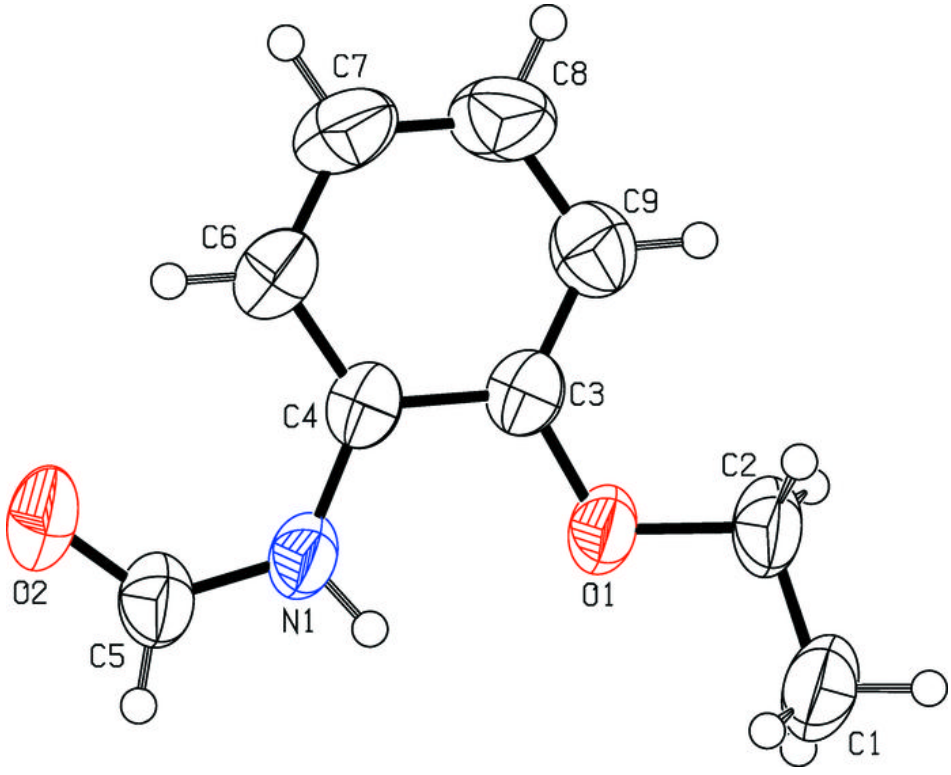

Fig. 2