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3-Methoxy-*N*-*p*-tolylbenzohydroxamic acid

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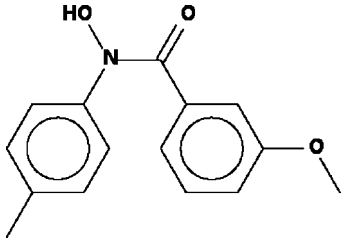
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.121; data-to-parameter ratio = 13.4.

Two molecules of the title compound, $C_{15}H_{15}NO_3$, are linked by a pair of O—H···$O_{carbonyl}$ hydrogen bonds over a centre of inversion to form a hydrogen-bonded dimer. With respect to the –C(=O)—N(OH)– unit, the methoxy-substituted ring is twisted by 42.2 (1)°, whereas the methyl-substituted ring is twisted by 52.2 (1)°.

Related literature

The parent *N*-phenylbenzohydroxamic acid exists in the *cis* form as well as the common *trans* form (see Yamasaki *et al.*, 2006). For the synthesis and spectroscopic data of the title compound, see: Agrawal & Tandon (1971, 1972, 1973).



Experimental

Crystal data

$C_{15}H_{15}NO_3$
M_r = 257.28
Monoclinic, $P2_1/c$
a = 11.332 (1) Å
b = 7.939 (1) Å
c = 15.567 (2) Å
β = 106.397 (2)°
V = 1343.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.47 × 0.36 × 0.27 mm

Data collection

Bruker APEX diffractometer
Absorption correction: none
6832 measured reflections
2378 independent reflections
1748 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.121
S = 1.06
2378 reflections
178 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 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
O1—H1···O2^{i}	0.86 (1)	1.99 (2)	2.699 (2)	139 (2)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

The authors acknowledge Professor Bohari M. Yamin's assistance with the diffraction measurements. The authors thank Universiti Kebangsaan Malaysia and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AV2005).

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supporting information

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S1. Experimental

3-Methoxybenzoyl chloride (1.4 g, 0.01 mmol) dissolved in ether was added to *N*-(4-tolyl)hydroxylamine (1.0 g, 0.01 mmol) dissolved in ether in the presence of sodium bicarbonate (0.7 g, 0.01 mmol). The reaction was carried out in an ice-bath. The solid that formed on removal of the solvent was extracted with ethyl acetate (10 ml). The solution yielded crystals after being set aside in a refrigerator.

S2. Refinement

The carbon-bound H atoms were placed at calculated positions (C–H 0.93– 0.97 Å), and were included in the refinement in the riding model approximation with *U*(H) set to 1.2–1.5U_{eq}(C). The hydropxy hydrogen atom was located in a difference Fouier map, and was refined with a distance restraint of O–H 0.85±0.01 Å.

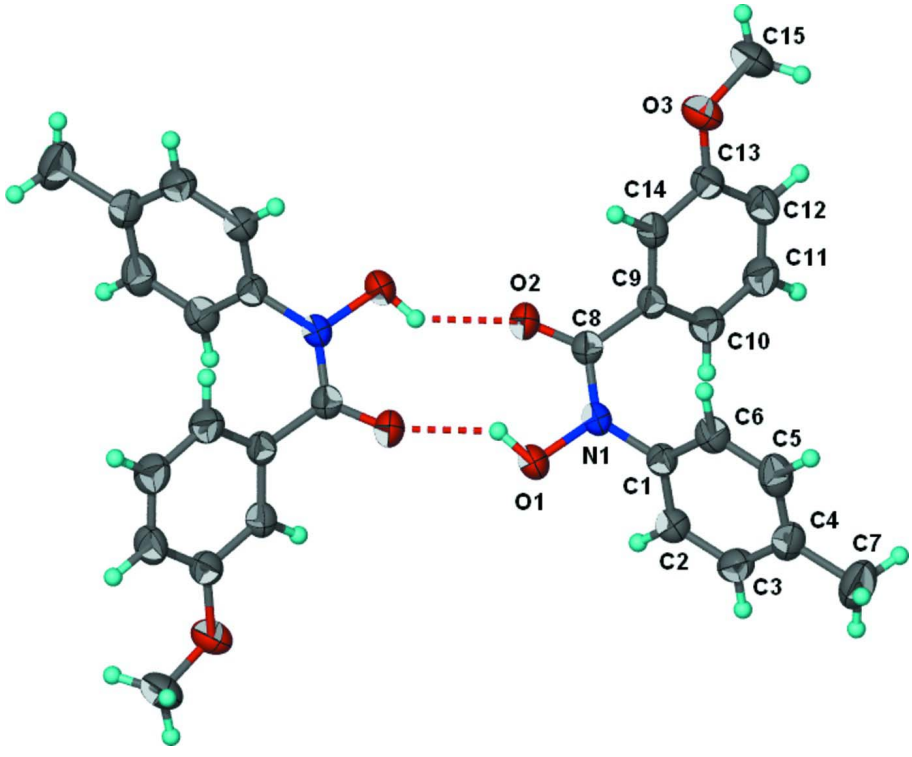

Figure 1

Thermal ellipsoid plot of two molecules of $C_{15}H_{15}NO_3$. Displacement ellipsoids are drawn at the 50% probability level, and H atoms are shown as spheres of arbitrary radii.

3-Methoxy-*N*-*p*-tolylbenzohydroxamic acid

Crystal data

$C_{15}H_{15}NO_3$
$M_r = 257.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.332$ (1) Å
$b = 7.939$ (1) Å
$c = 15.567$ (2) Å
$\beta = 106.397$ (2)°
$V = 1343.5$ (2) Å^3
$Z = 4$
$F(000) = 544$
$D_x = 1.272$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7258 reflections
$\theta = 2.8$–$21.7°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Irregular block, colorless
0.47 × 0.36 × 0.27 mm

Data collection

Bruker APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
6832 measured reflections
2378 independent reflections
1748 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 25.1°$, $\theta_{min} = 1.9°$
$h = -13 \rightarrow 13$
$k = -9 \rightarrow 9$
$l = -14 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.121$
$S = 1.06$
2378 reflections
178 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.1129P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.50151 (12)	0.30374 (18)	0.40770 (9)	0.0623 (4)
H1	0.494 (2)	0.390 (2)	0.4393 (15)	0.108 (9)*
O2	0.62368 (12)	0.42304 (16)	0.56320 (8)	0.0635 (4)
O3	0.94373 (12)	0.18762 (18)	0.82493 (8)	0.0681 (4)
N1	0.60896 (13)	0.22480 (18)	0.45952 (9)	0.0484 (4)
C1	0.62980 (15)	0.0686 (2)	0.42159 (11)	0.0436 (4)
C2	0.63120 (16)	0.0638 (2)	0.33340 (12)	0.0521 (5)
H2	0.6186	0.1618	0.2993	0.063*
C3	0.65135 (16)	−0.0866 (3)	0.29608 (13)	0.0585 (5)
H3	0.6524	−0.0890	0.2366	0.070*
C4	0.67003 (16)	−0.2340 (2)	0.34493 (15)	0.0588 (5)
C5	0.66643 (17)	−0.2264 (2)	0.43301 (15)	0.0616 (6)
H5	0.6788	−0.3245	0.4670	0.074*

C6	0.64499 (16)	−0.0772 (2)	0.47175 (13)	0.0532 (5)
H6	0.6409	−0.0751	0.5306	0.064*
C7	0.6942 (2)	−0.3978 (3)	0.30416 (18)	0.0897 (8)
H7A	0.6333	−0.4791	0.3082	0.135*
H7B	0.7744	−0.4387	0.3359	0.135*
H7C	0.6904	−0.3802	0.2424	0.135*
C8	0.66747 (16)	0.2968 (2)	0.53701 (11)	0.0453 (4)
C9	0.78932 (15)	0.2288 (2)	0.58918 (11)	0.0429 (4)
C10	0.88037 (16)	0.1831 (2)	0.55018 (13)	0.0546 (5)
H10	0.8647	0.1839	0.4882	0.065*
C11	0.99379 (17)	0.1367 (3)	0.60382 (14)	0.0639 (6)
H11	1.0548	0.1060	0.5775	0.077*
C12	1.01962 (17)	0.1344 (3)	0.69602 (14)	0.0606 (5)
H12	1.0968	0.1015	0.7314	0.073*
C13	0.92907 (17)	0.1818 (2)	0.73517 (12)	0.0502 (5)
C14	0.81453 (16)	0.2289 (2)	0.68151 (12)	0.0468 (4)
H14	0.7537	0.2610	0.7077	0.056*
C15	1.0630 (2)	0.1592 (4)	0.88294 (15)	0.0953 (9)
H15A	1.0606	0.1657	0.9440	0.143*
H15B	1.1184	0.2432	0.8725	0.143*
H15C	1.0911	0.0495	0.8717	0.143*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0614 (9)	0.0629 (9)	0.0531 (8)	0.0250 (7)	0.0004 (7)	−0.0025 (7)
O2	0.0637 (8)	0.0571 (8)	0.0624 (8)	0.0228 (7)	0.0058 (7)	−0.0098 (6)
O3	0.0640 (9)	0.0829 (10)	0.0491 (8)	0.0086 (7)	0.0024 (7)	0.0036 (7)
N1	0.0468 (9)	0.0480 (9)	0.0461 (9)	0.0125 (7)	0.0061 (7)	0.0007 (7)
C1	0.0391 (9)	0.0429 (10)	0.0466 (10)	0.0025 (7)	0.0086 (8)	−0.0022 (8)
C2	0.0516 (11)	0.0528 (11)	0.0498 (11)	0.0051 (8)	0.0109 (9)	−0.0004 (9)
C3	0.0513 (11)	0.0701 (14)	0.0514 (11)	0.0044 (10)	0.0104 (9)	−0.0129 (10)
C4	0.0418 (10)	0.0537 (12)	0.0753 (14)	−0.0004 (9)	0.0076 (10)	−0.0191 (10)
C5	0.0546 (12)	0.0439 (11)	0.0822 (15)	0.0003 (9)	0.0123 (11)	0.0040 (10)
C6	0.0535 (11)	0.0508 (11)	0.0543 (11)	0.0023 (9)	0.0134 (9)	0.0051 (9)
C7	0.0737 (15)	0.0686 (15)	0.121 (2)	0.0060 (12)	0.0177 (14)	−0.0378 (14)
C8	0.0469 (10)	0.0425 (10)	0.0464 (10)	0.0050 (8)	0.0129 (9)	0.0009 (8)
C9	0.0425 (9)	0.0355 (9)	0.0492 (10)	0.0021 (7)	0.0103 (8)	−0.0015 (7)
C10	0.0479 (11)	0.0607 (12)	0.0549 (12)	0.0027 (9)	0.0142 (9)	−0.0065 (9)
C11	0.0469 (11)	0.0752 (14)	0.0718 (14)	0.0071 (10)	0.0202 (10)	−0.0125 (11)
C12	0.0430 (10)	0.0606 (12)	0.0713 (15)	0.0075 (9)	0.0051 (10)	−0.0037 (10)
C13	0.0520 (11)	0.0425 (10)	0.0514 (12)	0.0008 (8)	0.0071 (9)	−0.0011 (8)
C14	0.0434 (10)	0.0445 (10)	0.0525 (11)	0.0043 (8)	0.0136 (8)	0.0001 (8)
C15	0.0872 (18)	0.118 (2)	0.0605 (14)	0.0368 (15)	−0.0128 (13)	−0.0057 (13)

Geometric parameters (Å, º)

O1—N1	1.4041 (18)	C7—H7A	0.9600
O1—H1	0.86 (1)	C7—H7B	0.9600
O2—C8	1.238 (2)	C7—H7C	0.9600
O3—C13	1.361 (2)	C8—C9	1.491 (2)
O3—C15	1.416 (2)	C9—C10	1.385 (2)
N1—C8	1.330 (2)	C9—C14	1.384 (2)
N1—C1	1.422 (2)	C10—C11	1.371 (3)
C1—C2	1.378 (2)	C10—H10	0.9300
C1—C6	1.380 (2)	C11—C12	1.382 (3)
C2—C3	1.375 (3)	C11—H11	0.9300
C2—H2	0.9300	C12—C13	1.385 (3)
C3—C4	1.379 (3)	C12—H12	0.9300
C3—H3	0.9300	C13—C14	1.382 (2)
C4—C5	1.385 (3)	C14—H14	0.9300
C4—C7	1.506 (3)	C15—H15A	0.9600
C5—C6	1.381 (3)	C15—H15B	0.9600
C5—H5	0.9300	C15—H15C	0.9600
C6—H6	0.9300		
N1—O1—H1	103.5 (18)	H7B—C7—H7C	109.5
C13—O3—C15	117.87 (16)	O2—C8—N1	120.20 (15)
C8—N1—O1	117.30 (14)	O2—C8—C9	120.62 (15)
C8—N1—C1	130.73 (14)	N1—C8—C9	119.10 (15)
O1—N1—C1	111.69 (13)	C10—C9—C14	119.63 (16)
C2—C1—C6	120.42 (16)	C10—C9—C8	123.08 (16)
C2—C1—N1	119.18 (15)	C14—C9—C8	116.95 (15)
C6—C1—N1	120.38 (16)	C11—C10—C9	119.33 (18)
C3—C2—C1	119.64 (17)	C11—C10—H10	120.3
C3—C2—H2	120.2	C9—C10—H10	120.3
C1—C2—H2	120.2	C10—C11—C12	121.57 (18)
C2—C3—C4	121.47 (18)	C10—C11—H11	119.2
C2—C3—H3	119.3	C12—C11—H11	119.2
C4—C3—H3	119.3	C11—C12—C13	119.17 (18)
C3—C4—C5	117.80 (17)	C11—C12—H12	120.4
C3—C4—C7	121.2 (2)	C13—C12—H12	120.4
C5—C4—C7	121.0 (2)	O3—C13—C14	115.67 (16)
C6—C5—C4	121.85 (18)	O3—C13—C12	124.76 (17)
C6—C5—H5	119.1	C14—C13—C12	119.57 (17)
C4—C5—H5	119.1	C13—C14—C9	120.72 (16)
C1—C6—C5	118.78 (18)	C13—C14—H14	119.6
C1—C6—H6	120.6	C9—C14—H14	119.6
C5—C6—H6	120.6	O3—C15—H15A	109.5
C4—C7—H7A	109.5	O3—C15—H15B	109.5
C4—C7—H7B	109.5	H15A—C15—H15B	109.5
H7A—C7—H7B	109.5	O3—C15—H15C	109.5
C4—C7—H7C	109.5	H15A—C15—H15C	109.5

H7A—C7—H7C	109.5	H15B—C15—H15C	109.5
C8—N1—C1—C2	−133.3 (2)	C1—N1—C8—C9	13.6 (3)
O1—N1—C1—C2	53.0 (2)	O2—C8—C9—C10	−133.46 (18)
C8—N1—C1—C6	48.5 (3)	N1—C8—C9—C10	43.3 (2)
O1—N1—C1—C6	−125.21 (17)	O2—C8—C9—C14	39.8 (2)
C6—C1—C2—C3	−1.8 (3)	N1—C8—C9—C14	−143.42 (16)
N1—C1—C2—C3	179.96 (15)	C14—C9—C10—C11	0.8 (3)
C1—C2—C3—C4	0.2 (3)	C8—C9—C10—C11	173.91 (18)
C2—C3—C4—C5	0.8 (3)	C9—C10—C11—C12	0.0 (3)
C2—C3—C4—C7	−178.94 (18)	C10—C11—C12—C13	−0.7 (3)
C3—C4—C5—C6	0.0 (3)	C15—O3—C13—C14	−172.98 (19)
C7—C4—C5—C6	179.68 (18)	C15—O3—C13—C12	6.5 (3)
C2—C1—C6—C5	2.5 (3)	C11—C12—C13—O3	−178.85 (18)
N1—C1—C6—C5	−179.29 (15)	C11—C12—C13—C14	0.6 (3)
C4—C5—C6—C1	−1.6 (3)	O3—C13—C14—C9	179.64 (15)
O1—N1—C8—O2	3.7 (3)	C12—C13—C14—C9	0.1 (3)
C1—N1—C8—O2	−169.65 (16)	C10—C9—C14—C13	−0.8 (2)
O1—N1—C8—C9	−173.02 (14)	C8—C9—C14—C13	−174.38 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.86 (1)	1.99 (2)	2.699 (2)	139 (2)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.