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2-Methylbenzaldehyde 2-methylbenzylidenehydrazone

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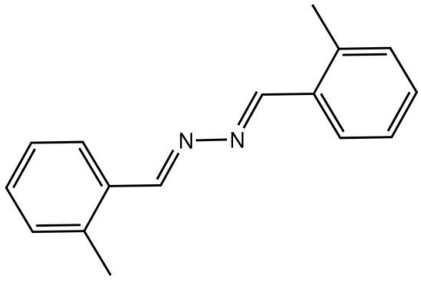
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.116; data-to-parameter ratio = 17.9.

The molecule of the title compound, $C_{16}H_{16}N_2$, is centrosymmetric and the dihedral angle between the benzene ring and the dimethylhydrazine mean plane is 16.11 (15)°.

Related literature

For background, see: Shan *et al.* (2003). For related structures, see: Fan *et al.* (2008); Shan *et al.* (2004, 2008).



Experimental

Crystal data

$C_{16}H_{16}N_2$ M_r = 236.31
Monoclinic, $P2_1/c$
a = 6.1578 (11) Å
b = 13.248 (2) Å
c = 8.8161 (16) Å
β = 105.398 (12)°
V = 693.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 295 (2) K
0.32 × 0.28 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: none
5451 measured reflections
1503 independent reflections
1168 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.115
S = 1.10
1503 reflections
84 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2750).

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supporting information

Acta Cryst. (2008). E**64**, o1386 [doi:10.1107/S160053680801934X]

2-Methylbenzaldehyde 2-methylbenzylidenehydrazone

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S1. Comment

As part of our ongong studies of hydrazone derivatives (Shan *et al.*, 2003), the title compound, (I), has been prepared and its crystal structure is reported here (Fig. 1).

The molecule of (I) is centrosymmetric, with the mid-point of the N—N bond located on an inversion center. The N=C7 double bond distance of 1.2727 (14) Å is shorter than the C=N bond distances found in related hydrazone structures, i.e. 1.295 (2)Å in (*E*)-3-methoxyacetophenone 4-nitrophenylhydrazone (Fan *et al.*, 2008), 1.2977 (18) Å in (E)-2-furyl methyl ketone 2,4-dinitrophenylhydrazone (Shan *et al.*, 2008) and 1.293 (2) Å in benzylideneacetone 2,4-dinitrophenyl-hydrazone (Shan *et al.* 2004). In (I), the terminal benzene ring is twisted with respect to the central dimethylhydrazine plane by 16.11 (15)°. The crystal packing is controlled by van der Waals forces.

S2. Experimental

Hydrazine hydrate (0.10 g, 2 mmol) was dissolved in ethanol (10 ml), then acetic acid (0.1 ml) was added slowly to the ethanol solution with stirring. The solution was heated at 333 K for several minutes until the solution cleared. 2-Methyl-benzaldehyde (0.24 g, 2 mmol) was then dropped slowly into the solution, and the mixture was kept at 333 K with continuous stirring for 2 h. After the solution had cooled to room temperature yellow powder appeared. The crude title compound was separated and washed with water three times. Recrystallization from an absolute ethanol yielded yellow plates of (I).

S3. Refinement

Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and the torsion angle was refined to fit the electron density with U_{iso}(H) = 1.5U_{eq}(C). The other H atoms were placed in calculated positions with C—H = 0.93 and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

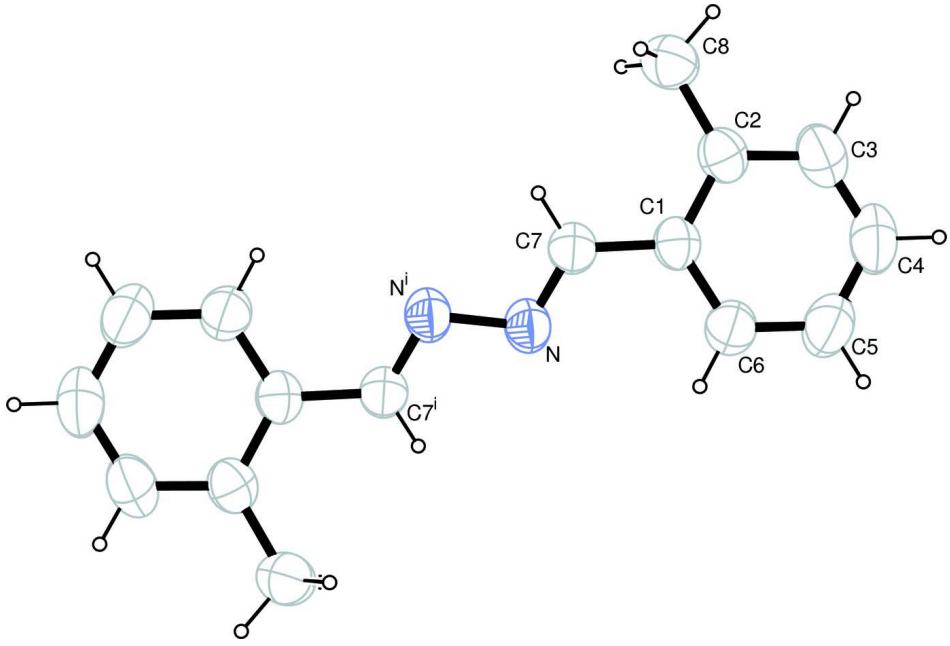

Figure 1

The molecular structure of (I) with 50% probability displacement ellipsoids for non-H atoms. Symmetry code: (i) -x, 1-y, -z.

2-Methylbenzaldehyde 2-methylbenzylidenehydrazone

Crystal data

$C_{16}H_{16}N_2$
$M_r = 236.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.1578\ (11)$ Å
$b = 13.248\ (2)$ Å
$c = 8.8161\ (16)$ Å
$\beta = 105.398\ (12)°$
$V = 693.4\ (2)$ Å^3
$Z = 2$
$F(000) = 252$
$D_x = 1.132$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2246 reflections
$\theta = 3.0–25.5°$
$\mu = 0.07$ mm^{-1}
$T = 295$ K
Plate, yellow
0.32 × 0.28 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.00 pixels mm^{-1}
ω scans
5451 measured reflections
1503 independent reflections
1168 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.9°$
$h = -7\rightarrow7$
$k = -15\rightarrow16$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.115$
$S = 1.10$
1503 reflections
84 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.0481P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.099 (12)

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}
N	0.03959 (16)	0.45944 (7)	0.05193 (11)	0.0545 (3)
C1	0.33920 (18)	0.40583 (8)	0.26999 (12)	0.0495 (3)
C2	0.56653 (19)	0.41758 (9)	0.35123 (13)	0.0553 (3)
C3	0.6648 (2)	0.34407 (11)	0.46155 (15)	0.0731 (4)
H3	0.8162	0.3499	0.5151	0.088*
C4	0.5435 (3)	0.26319 (11)	0.49318 (18)	0.0813 (5)
H4	0.6129	0.2158	0.5682	0.098*
C5	0.3198 (3)	0.25229 (10)	0.41412 (16)	0.0754 (4)
H5	0.2377	0.1977	0.4356	0.090*
C6	0.2186 (2)	0.32277 (9)	0.30309 (14)	0.0617 (4)
H6	0.0678	0.3151	0.2492	0.074*
C7	0.22666 (19)	0.47938 (8)	0.15044 (13)	0.0514 (3)
H7	0.2935	0.5419	0.1466	0.062*
C8	0.7057 (2)	0.50502 (11)	0.32086 (16)	0.0720 (4)
H8A	0.6477	0.5668	0.3517	0.108*
H8B	0.6986	0.5078	0.2108	0.108*
H8C	0.8593	0.4963	0.3807	0.108*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.0577 (6)	0.0502 (6)	0.0522 (6)	0.0060 (4)	0.0086 (4)	0.0054 (4)
C1	0.0564 (6)	0.0494 (6)	0.0427 (6)	0.0049 (5)	0.0129 (5)	−0.0008 (5)
C2	0.0563 (7)	0.0624 (7)	0.0471 (6)	0.0058 (5)	0.0137 (5)	−0.0003 (5)
C3	0.0655 (8)	0.0849 (10)	0.0623 (8)	0.0139 (7)	0.0055 (6)	0.0119 (7)
C4	0.0945 (11)	0.0741 (9)	0.0686 (9)	0.0164 (8)	0.0097 (8)	0.0235 (7)
C5	0.0976 (11)	0.0571 (8)	0.0700 (9)	−0.0035 (7)	0.0198 (8)	0.0121 (6)
C6	0.0682 (8)	0.0567 (7)	0.0570 (7)	−0.0038 (6)	0.0112 (6)	0.0030 (5)
C7	0.0549 (6)	0.0492 (6)	0.0495 (6)	0.0012 (5)	0.0126 (5)	0.0009 (5)

C8	0.0568 (7)	0.0860 (10)	0.0711 (8)	−0.0059 (6)	0.0135 (6)	0.0053 (7)

Geometric parameters (Å, º)

N—C7	1.2727 (14)	C4—C5	1.376 (2)
N—N^{i}	1.4121 (17)	C4—H4	0.9300
C1—C6	1.4007 (16)	C5—C6	1.3764 (17)
C1—C2	1.4016 (16)	C5—H5	0.9300
C1—C7	1.4672 (15)	C6—H6	0.9300
C2—C3	1.3956 (17)	C7—H7	0.9300
C2—C8	1.5065 (17)	C8—H8A	0.9600
C3—C4	1.3761 (19)	C8—H8B	0.9600
C3—H3	0.9300	C8—H8C	0.9600
C7—N—N^{i}	112.25 (11)	C4—C5—H5	120.3
C6—C1—C2	119.57 (10)	C6—C5—H5	120.3
C6—C1—C7	119.74 (10)	C5—C6—C1	121.08 (12)
C2—C1—C7	120.70 (10)	C5—C6—H6	119.5
C3—C2—C1	117.90 (12)	C1—C6—H6	119.5
C3—C2—C8	119.92 (11)	N—C7—C1	121.44 (11)
C1—C2—C8	122.18 (10)	N—C7—H7	119.3
C4—C3—C2	121.77 (13)	C1—C7—H7	119.3
C4—C3—H3	119.1	C2—C8—H8A	109.5
C2—C3—H3	119.1	C2—C8—H8B	109.5
C5—C4—C3	120.20 (12)	H8A—C8—H8B	109.5
C5—C4—H4	119.9	C2—C8—H8C	109.5
C3—C4—H4	119.9	H8A—C8—H8C	109.5
C4—C5—C6	119.48 (13)	H8B—C8—H8C	109.5

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