

1,2-Bis(2-furylmethylene)hydrazine

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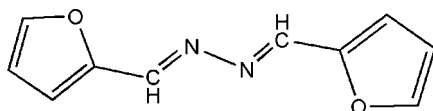
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.105; data-to-parameter ratio = 12.8.

Crystals of the title compound, $\text{C}_{10}\text{H}_8\text{N}_2\text{O}_2$, were obtained from a condensation reaction of hydrazine hydrate with furfural. In the crystal structure, the molecule is centrosymmetric and almost planar and the furan rings are parallel by symmetry.

Related literature

For background, see: Casellato & Vigato (1977); For related structures, see: Fan *et al.* (2008); Shan *et al.* (2004); Shan, Tian *et al.* (2008); Shan, Wang *et al.* (2008).



Experimental

Crystal data

$\text{C}_{10}\text{H}_8\text{N}_2\text{O}_2$	$V = 938.6$ (5) Å ³
$M_r = 188.18$	$Z = 4$
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 6.877$ (2) Å	$\mu = 0.10$ mm ⁻¹
$b = 8.996$ (3) Å	$T = 298$ (2) K
$c = 15.171$ (4) Å	$0.50 \times 0.40 \times 0.40$ mm

Data collection

Bruker SMART 1K CCD diffractometer	4129 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2000)	829 independent reflections
$T_{\min} = 0.954$, $T_{\max} = 0.963$	677 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.081$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	65 parameters
$wR(F^2) = 0.105$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.16$ e Å ⁻³
829 reflections	$\Delta\rho_{\text{min}} = -0.25$ e Å ⁻³

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL/PC (Sheldrick, 2008); software used to prepare material for publication: SHELXTL/PC.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2802).

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supplementary materials

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Comment

Schiff bases have been studied for decades (Casellato & Vigato 1977) and they are still one of the most prevalent mixed-donor ligands in coordination chemistry, with numerous applications including single-molecule magnetism, materials science and catalysis. Here, the synthesis and crystal structure of the title compound (I) are reported.

The molecule of (I) is centrosymmetric, with the midpoint of the N—N bond located on the inversion center. The C5—N1 double bond distance of 1.272 (2) Å is shorter than the C=N bond distance found in related hydrazone structures, *i.e.* 1.295 (2) Å in (*E*)-3-methoxyacetophenone 4-nitrophenylhydrazone (Fan *et al.*, 2008), 1.298 (2) Å in (*E*)-2-furylmethylketone 2,4-dinitrophenylhydrazone (Shan, Tian *et al.*, 2008) and 1.293 (2) Å in benzylideneacetone 2,4-dinitrophenylhydrazone (Shan *et al.*, 2004). It is indistinguishable from the length of 1.273 (1) Å in 2-methylbenzaldehyde 2-methylbenzylidenehydrazone (Shan, Wang *et al.*, 2008).

Experimental

Hydrazine hydrate (35% solution in water, 0.71 g, 5 mmol) and furfural (0.96 g, 10 mmol) were mixed, at the same time adding 2 or 3 drops of formic acid, and stirred at room temperature in 30 ml of ethanol solution for 2 days, and then the filtrate was kept open to slowly evaporate for a few days, depositing yellow blocks of (I).

Refinement

The H atoms attached were placed in geometrically idealized positions (C—H = 0.93 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

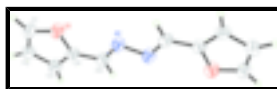


Fig. 1. A view of the molecular structure of (I) with displacement ellipsoids for the non-hydrogen atoms drawn at the 30% probability level (arbitrary spheres for the H atoms). The unlabelled atoms are generated by the symmetry operation (1-*x*, 2-*y*, -*z*).

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Crystal data

C₁₀H₈N₂O₂

$M_r = 188.18$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$F_{000} = 392$

$D_x = 1.332 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5207 reflections

supplementary materials

$a = 6.877 (2) \text{ \AA}$	$\theta = 2.6\text{--}25.3^\circ$
$b = 8.996 (3) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 15.171 (4) \text{ \AA}$	$T = 298 (2) \text{ K}$
$V = 938.6 (5) \text{ \AA}^3$	Block, yellow
$Z = 4$	$0.50 \times 0.40 \times 0.40 \text{ mm}$

Data collection

Bruker SMART 1K CCD diffractometer	829 independent reflections
Radiation source: fine-focus sealed tube	677 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.081$
$T = 298(2) \text{ K}$	$\theta_{\text{max}} = 25.3^\circ$
ω scans	$\theta_{\text{min}} = 2.6^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2000)	$h = -8 \rightarrow 8$
$T_{\text{min}} = 0.954$, $T_{\text{max}} = 0.963$	$k = -10 \rightarrow 10$
4129 measured reflections	$l = -18 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
829 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
65 parameters	$\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
	Extinction correction: none

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 (\AA^2)

x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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C1	-0.0467 (2)	0.88870 (18)	0.18436 (11)	0.0736 (5)
H1	-0.0937	0.8074	0.2157	0.088*
C2	-0.1336 (2)	1.01970 (19)	0.18094 (10)	0.0719 (5)
H2	-0.2492	1.0466	0.2084	0.086*
C3	-0.0168 (2)	1.10963 (17)	0.12769 (10)	0.0642 (5)
H3	-0.0400	1.2085	0.1131	0.077*
C4	0.13456 (19)	1.02755 (14)	0.10149 (8)	0.0529 (4)
C5	0.2964 (2)	1.06191 (15)	0.04628 (9)	0.0566 (4)
H5	0.3049	1.1570	0.0225	0.068*
N1	0.42950 (18)	0.96866 (13)	0.02805 (8)	0.0613 (4)
O1	0.12017 (16)	0.88799 (10)	0.13632 (7)	0.0669 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0695 (10)	0.0698 (11)	0.0815 (11)	-0.0135 (8)	0.0114 (9)	0.0137 (8)
C2	0.0637 (10)	0.0800 (12)	0.0721 (11)	0.0049 (8)	0.0084 (7)	0.0085 (8)
C3	0.0722 (11)	0.0589 (10)	0.0616 (9)	0.0093 (7)	0.0038 (7)	0.0082 (7)
C4	0.0639 (9)	0.0452 (8)	0.0496 (7)	-0.0049 (6)	-0.0044 (6)	0.0007 (6)
C5	0.0690 (10)	0.0485 (8)	0.0522 (8)	-0.0055 (7)	0.0007 (7)	0.0013 (6)
N1	0.0649 (8)	0.0541 (8)	0.0649 (8)	-0.0033 (6)	0.0084 (5)	0.0045 (5)
O1	0.0698 (8)	0.0479 (7)	0.0830 (8)	-0.0029 (5)	0.0078 (5)	0.0057 (4)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.322 (2)	C3—H3	0.9300
C1—O1	1.359 (2)	C4—O1	1.3657 (16)
C1—H1	0.9300	C4—C5	1.427 (2)
C2—C3	1.397 (2)	C5—N1	1.2721 (18)
C2—H2	0.9300	C5—H5	0.9300
C3—C4	1.3364 (19)	N1—N1 ⁱ	1.408 (2)
C2—C1—O1	111.39 (14)	C3—C4—O1	109.66 (12)
C2—C1—H1	124.3	C3—C4—C5	131.47 (13)
O1—C1—H1	124.3	O1—C4—C5	118.87 (12)
C1—C2—C3	106.18 (15)	N1—C5—C4	123.10 (13)
C1—C2—H2	126.9	N1—C5—H5	118.4
C3—C2—H2	126.9	C4—C5—H5	118.4
C4—C3—C2	107.45 (14)	C5—N1—N1 ⁱ	111.29 (15)
C4—C3—H3	126.3	C1—O1—C4	105.32 (12)
C2—C3—H3	126.3		
O1—C1—C2—C3	-0.05 (19)	O1—C4—C5—N1	-0.4 (2)
C1—C2—C3—C4	0.23 (18)	C4—C5—N1—N1 ⁱ	179.64 (14)
C2—C3—C4—O1	-0.32 (17)	C2—C1—O1—C4	-0.14 (18)
C2—C3—C4—C5	179.42 (15)	C3—C4—O1—C1	0.29 (16)
C3—C4—C5—N1	179.91 (15)	C5—C4—O1—C1	-179.49 (12)

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

Fig. 1

