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2,2'-o-Phenylenediacetonitrile

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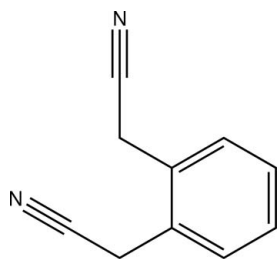
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 Key indicators: single-crystal X-ray study; $T = 93$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.051; wR factor = 0.133; data-to-parameter ratio = 15.2.

In the title compound, $\text{NCCH}_2\text{C}_6\text{H}_4\text{CH}_2\text{CN}$, the bond lengths and angles are within normal ranges. The benzene ring makes dihedral angles of 4.94 (8) and 77.04 (8)° with the $\text{C}-\text{C}-\text{C}-\text{N}$ mean planes. Weak non-conventional $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds are effective in the stabilization of the crystal structure. The weak $\text{C}-\text{H}\cdots\text{N}$ contacts form antiparallel chains running in the $a + c$ direction, and ring systems with two N-atom acceptors and four H-atom donors.

Related literature

For reactions of Woollins' Reagent see: Gray *et al.* (2005); Hua *et al.* (2006, 2009); Hua & Woollins (2009). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_8\text{N}_2$
 $M_r = 156.18$
 Monoclinic, $P2_1/n$
 $a = 8.3882$ (18) Å
 $b = 8.1605$ (15) Å
 $c = 11.993$ (2) Å

 $\beta = 101.890$ (6)°
 $V = 803.4$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

 $\mu = 0.08$ mm⁻¹
 $T = 93$ K
 $0.30 \times 0.25 \times 0.15$ mm

Data collection

 Rigaku Mercury CCD diffractometer
 Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2004)
 $T_{\min} = 0.977$, $T_{\max} = 0.988$

 5271 measured reflections
 1660 independent reflections
 1330 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.133$
 $S = 1.09$
 1660 reflections

 109 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C9}-\text{H9A}\cdots\text{N1}^{\text{i}}$	0.99	2.57	3.5605 (18)	176
$\text{C9}-\text{H9B}\cdots\text{N1}^{\text{ii}}$	0.99	2.56	3.5210 (17)	165

 Symmetry codes: (i) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2203).

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supporting information

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2,2'-*o*-Phenylenediacetonitrile

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S1. Comment

Recently, we have continued our studies exploring the reactivity of Woollins reagent towards different organic substituents (Gray *et al.* 2005, Hua *et al.* 2006 and 2009; Hua & Woollins 2009). Thereby 2,2'-(1,2-phenylene)diacetonitrile represents one of the starting materials. Single crystals of 2,5-dihydroxybenzaldehyde for X-ray crystallographic analysis were obtained by recrystallization from dichloromethane-hexane solution.

The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The benzene ring makes the dihedral angles of 4.94 (8) and 77.04 (8)° with the mean planes of C1—C7—C8—N1 and C2—C9—C10—N2 respectively. The antiparallel chains running in *a*+*c* direction are generated through the weak C—H···N contacts, glide plane and inversion symmetry operations [see Fig. 2 and Table 1]. Inversion symmetry forms also C—H···N ring systems consisting of two N acceptors and four H atom donors, where the centroid-centroid distance between the inversion-related benzene ring planes is 3.6809 (10) Å, the perpendicular plane to plane distance is 3.364 Å, and the slippage between the planes is 1.495 Å.

S2. Experimental

Commercially available 2,2'-(1,2-phenylene)diacetonitrile was recrystallized from dichloromethane-hexane.

S3. Refinement

H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and were treated as riding on their parent C atoms with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$.

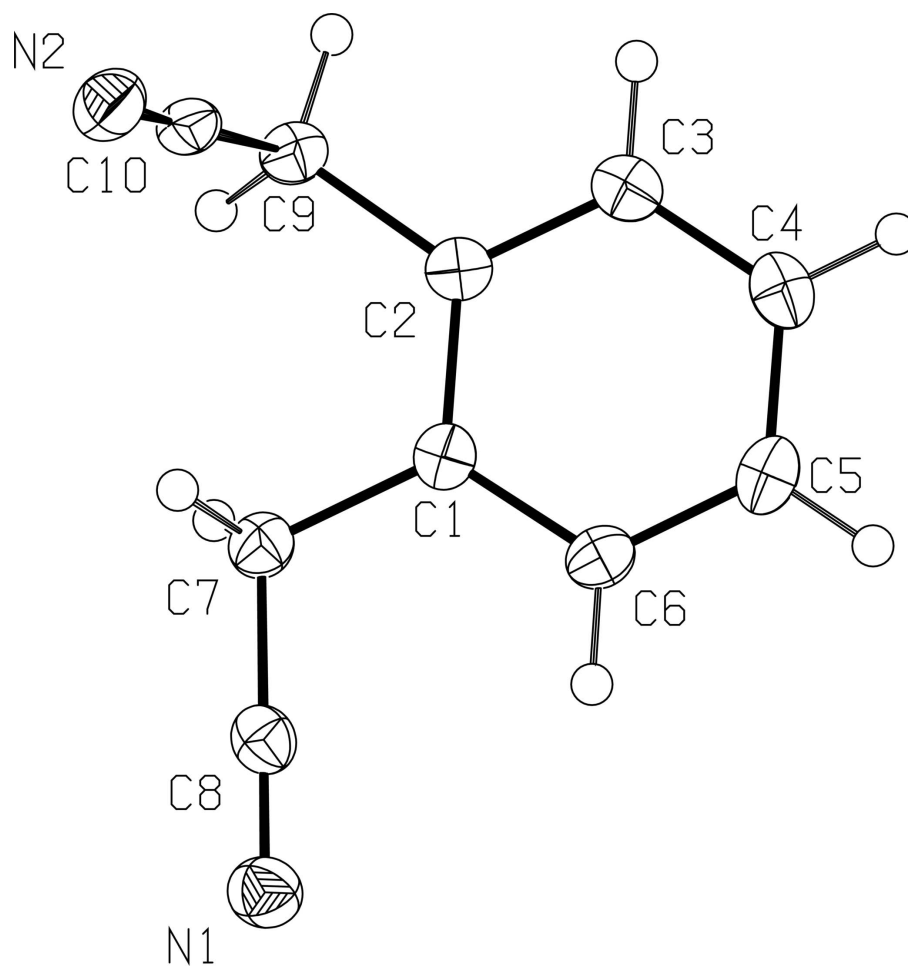


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

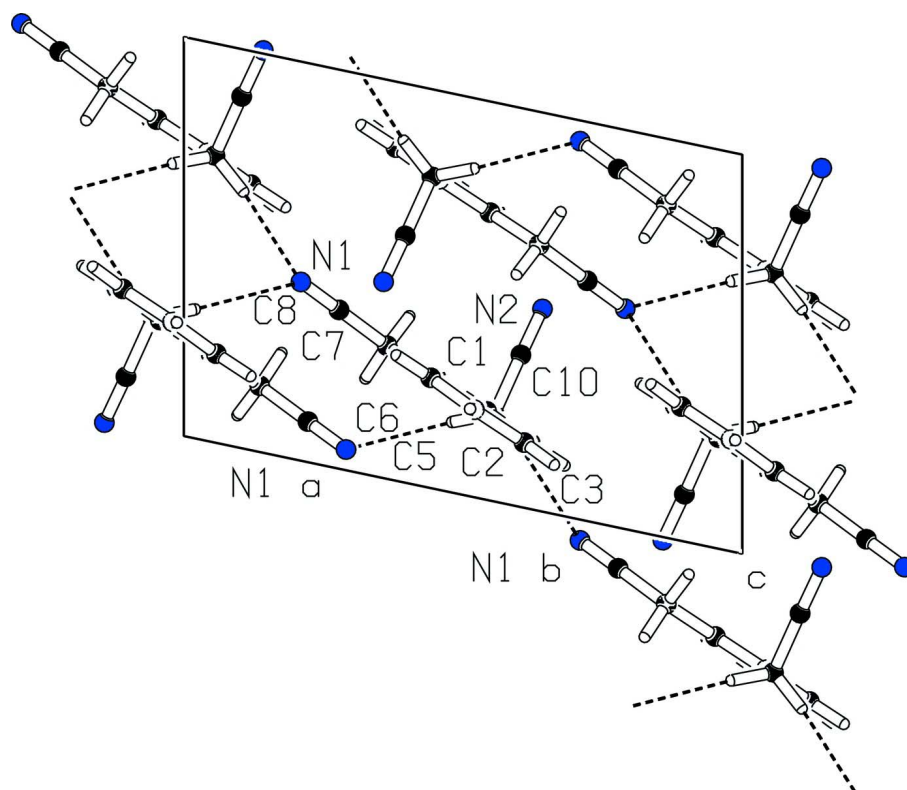


Figure 2

View along the *b* direction of the crystal packing of the title compound with non-conventional hydrogen bonding shown as dashed lines.

2,2'-*o*-Phenylenediacetonitrile

Crystal data

$C_{10}H_8N_2$

$M_r = 156.18$

Monoclinic, $P2_1/n$

$a = 8.3882$ (18) Å

$b = 8.1605$ (15) Å

$c = 11.993$ (2) Å

$\beta = 101.890$ (6)°

$V = 803.4$ (3) Å³

$Z = 4$

$F(000) = 328$

$D_x = 1.291$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2633 reflections

$\theta = 2.7$ – 28.3 °

$\mu = 0.08$ mm⁻¹

$T = 93$ K

Block, colorless

$0.30 \times 0.25 \times 0.15$ mm

Data collection

Rigaku Mercury CCD

diffractometer

Radiation source: rotating anode

Confocal monochromator

Detector resolution: 0.83 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*CrystalClear*; Rigaku, 2004)

$T_{\min} = 0.977$, $T_{\max} = 0.988$

5271 measured reflections

1660 independent reflections

1330 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 28.7$ °, $\theta_{\min} = 2.7$ °

$h = -11 \rightarrow 10$

$k = -9 \rightarrow 10$

$l = -13 \rightarrow 15$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.051$ $wR(F^2) = 0.133$ $S = 1.09$

1660 reflections

109 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.0077P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$ *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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.77845 (13)	0.05887 (16)	0.95174 (10)	0.0215 (3)
C2	0.72184 (13)	0.13136 (16)	1.04228 (10)	0.0218 (3)
C7	0.83384 (14)	0.16768 (16)	0.86390 (10)	0.0257 (3)
H7A	0.7404	0.2350	0.8253	0.031*
H7B	0.9191	0.2433	0.9038	0.031*
C3	0.66532 (15)	0.03264 (16)	1.12009 (11)	0.0250 (3)
H3	0.6273	0.0817	1.1816	0.030*
C8	0.89813 (14)	0.07728 (16)	0.77779 (10)	0.0252 (3)
N1	0.94789 (13)	0.00673 (14)	0.70957 (10)	0.0313 (3)
C10	0.88145 (15)	0.38338 (15)	1.10361 (10)	0.0248 (4)
C9	0.71906 (14)	0.31597 (16)	1.05579 (11)	0.0249 (3)
H9A	0.6768	0.3666	0.9805	0.030*
H9B	0.6438	0.3448	1.1064	0.030*
C5	0.71958 (14)	-0.20835 (16)	1.02028 (11)	0.0278 (4)
H5	0.7192	-0.3242	1.0126	0.033*
C6	0.77646 (14)	-0.11076 (16)	0.94203 (11)	0.0248 (4)
H6	0.8146	-0.1607	0.8809	0.030*
N2	1.00792 (13)	0.43446 (14)	1.14226 (9)	0.0318 (3)
C4	0.66348 (14)	-0.13701 (17)	1.10929 (11)	0.0271 (3)
H4	0.6239	-0.2034	1.1628	0.033*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0169 (6)	0.0226 (7)	0.0238 (7)	0.0002 (4)	0.0009 (5)	0.0007 (5)

C2	0.0176 (6)	0.0247 (7)	0.0221 (7)	0.0023 (5)	0.0013 (5)	-0.0002 (5)
C7	0.0267 (7)	0.0232 (7)	0.0274 (7)	0.0013 (5)	0.0062 (5)	0.0001 (6)
C3	0.0207 (7)	0.0311 (8)	0.0220 (7)	-0.0002 (5)	0.0019 (5)	-0.0002 (6)
C8	0.0217 (7)	0.0285 (7)	0.0252 (7)	-0.0031 (5)	0.0043 (5)	0.0008 (6)
N1	0.0292 (7)	0.0337 (8)	0.0319 (7)	-0.0012 (5)	0.0085 (5)	-0.0019 (5)
C10	0.0321 (7)	0.0198 (7)	0.0243 (7)	0.0044 (5)	0.0097 (6)	-0.0012 (5)
C9	0.0256 (7)	0.0240 (8)	0.0250 (7)	0.0021 (5)	0.0049 (5)	-0.0018 (6)
C5	0.0257 (7)	0.0210 (7)	0.0342 (8)	-0.0006 (5)	0.0007 (6)	0.0018 (6)
C6	0.0241 (7)	0.0235 (8)	0.0263 (7)	0.0027 (5)	0.0040 (5)	-0.0023 (6)
N2	0.0325 (7)	0.0272 (7)	0.0359 (7)	-0.0019 (5)	0.0071 (5)	-0.0044 (5)
C4	0.0234 (7)	0.0314 (8)	0.0253 (7)	-0.0034 (5)	0.0022 (5)	0.0054 (6)

Geometric parameters (Å, °)

C1—C6	1.3890 (19)	C8—N1	1.1471 (15)
C1—C2	1.4025 (16)	C10—N2	1.1452 (15)
C1—C7	1.5218 (18)	C10—C9	1.4714 (17)
C2—C3	1.3882 (18)	C9—H9A	0.9900
C2—C9	1.5159 (18)	C9—H9B	0.9900
C7—C8	1.4599 (17)	C5—C4	1.3809 (18)
C7—H7A	0.9900	C5—C6	1.3882 (17)
C7—H7B	0.9900	C5—H5	0.9500
C3—C4	1.390 (2)	C6—H6	0.9500
C3—H3	0.9500	C4—H4	0.9500
C6—C1—C2	119.02 (11)	N2—C10—C9	178.97 (14)
C6—C1—C7	121.58 (11)	C10—C9—C2	112.32 (10)
C2—C1—C7	119.36 (12)	C10—C9—H9A	109.1
C3—C2—C1	119.52 (13)	C2—C9—H9A	109.1
C3—C2—C9	119.34 (11)	C10—C9—H9B	109.1
C1—C2—C9	121.14 (11)	C2—C9—H9B	109.1
C8—C7—C1	113.88 (11)	H9A—C9—H9B	107.9
C8—C7—H7A	108.8	C4—C5—C6	120.00 (12)
C1—C7—H7A	108.8	C4—C5—H5	120.0
C8—C7—H7B	108.8	C6—C5—H5	120.0
C1—C7—H7B	108.8	C1—C6—C5	121.00 (11)
H7A—C7—H7B	107.7	C1—C6—H6	119.5
C2—C3—C4	120.94 (12)	C5—C6—H6	119.5
C2—C3—H3	119.5	C5—C4—C3	119.53 (12)
C4—C3—H3	119.5	C5—C4—H4	120.2
N1—C8—C7	179.54 (14)	C3—C4—H4	120.2
C6—C1—C2—C3	0.04 (16)	C3—C2—C9—C10	103.33 (13)
C7—C1—C2—C3	177.52 (11)	C1—C2—C9—C10	-77.54 (13)
C6—C1—C2—C9	-179.09 (11)	C2—C1—C6—C5	0.00 (17)
C7—C1—C2—C9	-1.61 (15)	C7—C1—C6—C5	-177.43 (10)
C6—C1—C7—C8	-5.75 (16)	C4—C5—C6—C1	0.15 (18)
C2—C1—C7—C8	176.84 (10)	C6—C5—C4—C3	-0.34 (18)

C1—C2—C3—C4	-0.23 (17)	C2—C3—C4—C5	0.38 (18)
C9—C2—C3—C4	178.91 (11)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C9—H9 <i>A</i> ...N1 ⁱ	0.99	2.57	3.5605 (18)	176
C9—H9 <i>B</i> ...N1 ⁱⁱ	0.99	2.56	3.5210 (17)	165

Symmetry codes: (i) $-x+3/2, y+1/2, -z+3/2$; (ii) $x-1/2, -y+1/2, z+1/2$.