

(*E*)-1,1'-(Diazene-1,2-diyl)bis(cyclohexane-1-carbonitrile)

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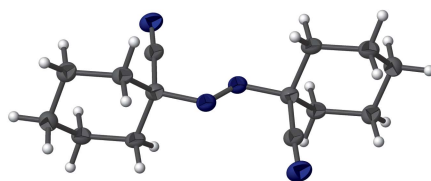
Keywords: crystal structure; VAZO-88; radical initiator; diazene; carbonitrile; cyclohexane.

CCDC reference: 1536557

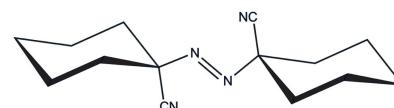
Structural data: full structural data are available from iucrdata.iucr.org

The whole molecule of the title compound, $C_{14}H_{20}N_4$, is generated by inversion symmetry. The mid-point of the $N=N$ bond is situated on the inversion centre. The conformation about this central $N=N$ bond is *E*. The carbonitrile groups occupy axial positions on the cyclohexane rings. In the crystal, there are no significant intermolecular interactions present.

3D view



Chemical scheme



Structure description

The title compound, known as VAZO-88, is a well known radical initiator that has also been used as a ligand in coordination chemistry (Chainok *et al.*, 2010).

The whole molecule of the title compound, illustrated in Fig. 1, is generated by inversion symmetry. The mid-point of the $N=N$ bond is situated on the inversion centre. The conformation about the central $N1=N1'$ bond, of 1.231 (2) Å, is *E*. The carbonitrile groups occupy axial positions on the cyclohexane rings and the $C2-C3\equiv N4$ bond angle is 176.58 (18)°, with bond length $C3\equiv N4$ being 1.147 (2) Å.

In the crystal, molecules stack along the *a*-axis direction (Fig. 2), but there are no significant intermolecular interactions present.

Synthesis and crystallization

During an attempted radical cyclization reaction, using commercially available VAZO-88, we obtained a few colourless crystals of the title compound, on slow evaporation of a hexane solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

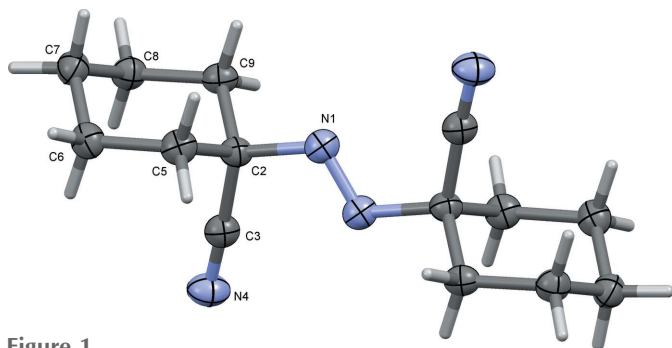


Figure 1
The molecular structure of the title compound, with the atom labelling and displacement ellipsoids drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by the symmetry operation $(-x + 1, -y + 1, -z + 1)$.

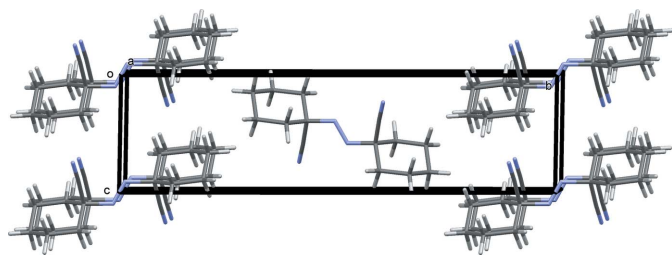


Figure 2
A view along the a axis of the crystal packing of the title compound.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{20}N_4$
M_r	244.34
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	5.8740 (19), 21.077 (7), 5.974 (2)
β (°)	110.372 (13)
V (Å ³)	693.4 (4)
Z	2
Radiation type	Cu $K\alpha$
μ (mm ⁻¹)	0.57
Crystal size (mm)	0.16 × 0.13 × 0.12
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (SADABS; Bruker, 2015)
T_{min}, T_{max}	0.666, 0.753
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7548, 1299, 1213
R_{int}	0.033
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.608
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.059, 0.160, 1.06
No. of reflections	1299
No. of parameters	82
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.47, -0.16

Computer programs: APEX3 and SAINT (Bruker, 2015), SHELXS2014 (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015), Mercury (Macrae *et al.*, 2008) and OLEX2 (Dolomanov *et al.*, 2009).

Funding information

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References

Bruker (2015). APEX3, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.

- Chainok, K., Neville, S. M., Moubaraki, B., Batten, S. R., Murray, K. S., Forsyth, C. M. & Cashion, J. D. (2010). *Dalton Trans.* **39**, 10900–10909.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **A71**, 3–8.

full crystallographic data

IUCrData (2017). 2, x170369 [https://doi.org/10.1107/S2414314617003698]

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(*E*-1,1'-(Diazene-1,2-diyl)bis(cyclohexane-1-carbonitrile))*Crystal data*

$C_{14}H_{20}N_4$	$F(000) = 264$
$M_r = 244.34$	$D_x = 1.170 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$
$a = 5.8740 (19) \text{ \AA}$	Cell parameters from 4485 reflections
$b = 21.077 (7) \text{ \AA}$	$\theta = 4.2\text{--}69.5^\circ$
$c = 5.974 (2) \text{ \AA}$	$\mu = 0.57 \text{ mm}^{-1}$
$\beta = 110.372 (13)^\circ$	$T = 100 \text{ K}$
$V = 693.4 (4) \text{ \AA}^3$	Block, colourless
$Z = 2$	$0.16 \times 0.13 \times 0.12 \text{ mm}$

Data collection

Bruker APEXII CCD diffractometer	$T_{\min} = 0.666, T_{\max} = 0.753$
Radiation source: microfocus sealed X-ray tube, Incoatec I μ s	7548 measured reflections
Mirror optics monochromator	1299 independent reflections
Detector resolution: 7.9 pixels mm^{-1}	1213 reflections with $I > 2\sigma(I)$
ω and ϕ scans	$R_{\text{int}} = 0.033$
Absorption correction: multi-scan (SADABS; Bruker, 2015)	$\theta_{\max} = 69.7^\circ, \theta_{\min} = 8.2^\circ$
	$h = -7 \rightarrow 7$
	$k = -25 \rightarrow 25$
	$l = -7 \rightarrow 7$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.059$	$w = 1/[\sigma^2(F_o^2) + (0.0875P)^2 + 0.4078P]$
$wR(F^2) = 0.160$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\max} < 0.001$
1299 reflections	$\Delta\rho_{\max} = 0.47 \text{ e \AA}^{-3}$
82 parameters	$\Delta\rho_{\min} = -0.16 \text{ e \AA}^{-3}$
0 restraints	
Primary atom site location: dual	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C2	0.5458 (3)	0.42030 (8)	0.4069 (3)	0.0259 (4)
C3	0.7721 (3)	0.40938 (8)	0.6143 (3)	0.0286 (4)
C5	0.3418 (3)	0.37594 (8)	0.4188 (3)	0.0295 (4)
H5A	0.3201	0.3814	0.5748	0.035*
H5B	0.1876	0.3878	0.2920	0.035*
C6	0.3981 (3)	0.30654 (8)	0.3880 (3)	0.0309 (5)
H6A	0.5404	0.2930	0.5261	0.037*
H6B	0.2580	0.2800	0.3846	0.037*
C7	0.4513 (3)	0.29635 (8)	0.1585 (3)	0.0320 (5)
H7A	0.3027	0.3049	0.0194	0.038*
H7B	0.4982	0.2516	0.1493	0.038*
C8	0.6554 (3)	0.33973 (8)	0.1480 (3)	0.0306 (5)
H8A	0.8083	0.3281	0.2772	0.037*
H8B	0.6797	0.3337	-0.0065	0.037*
C9	0.5977 (3)	0.40919 (8)	0.1749 (3)	0.0280 (4)
H9A	0.4544	0.4221	0.0368	0.034*
H9B	0.7368	0.4358	0.1757	0.034*
N1	0.4544 (3)	0.48574 (7)	0.4049 (3)	0.0285 (4)
N4	0.9502 (3)	0.39882 (8)	0.7674 (3)	0.0382 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0226 (8)	0.0294 (9)	0.0254 (8)	0.0012 (6)	0.0079 (7)	-0.0005 (6)
C3	0.0255 (9)	0.0331 (9)	0.0291 (9)	0.0010 (7)	0.0118 (8)	-0.0008 (7)
C5	0.0215 (8)	0.0357 (10)	0.0333 (9)	-0.0010 (7)	0.0120 (7)	-0.0009 (7)
C6	0.0252 (9)	0.0316 (10)	0.0364 (10)	-0.0015 (7)	0.0114 (7)	0.0020 (7)
C7	0.0306 (9)	0.0295 (9)	0.0341 (9)	-0.0002 (7)	0.0089 (7)	-0.0023 (7)
C8	0.0307 (9)	0.0336 (10)	0.0298 (9)	0.0015 (7)	0.0133 (7)	-0.0010 (7)
C9	0.0247 (9)	0.0347 (10)	0.0255 (9)	-0.0012 (6)	0.0099 (7)	-0.0005 (7)
N1	0.0247 (8)	0.0320 (8)	0.0301 (7)	-0.0008 (5)	0.0111 (6)	-0.0023 (5)
N4	0.0284 (9)	0.0505 (10)	0.0323 (8)	0.0056 (7)	0.0063 (7)	0.0012 (7)

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

C2—C3	1.486 (2)	C6—C7	1.524 (3)
C2—C5	1.541 (2)	C7—H7A	0.9900
C2—C9	1.537 (2)	C7—H7B	0.9900
C2—N1	1.479 (2)	C7—C8	1.526 (2)
C3—N4	1.147 (2)	C8—H8A	0.9900
C5—H5A	0.9900	C8—H8B	0.9900
C5—H5B	0.9900	C8—C9	1.524 (2)
C5—C6	1.525 (2)	C9—H9A	0.9900
C6—H6A	0.9900	C9—H9B	0.9900
C6—H6B	0.9900	N1—N1 ⁱ	1.231 (3)

C3—C2—C5	110.60 (14)	C6—C7—H7A	109.4
C3—C2—C9	109.11 (14)	C6—C7—H7B	109.4
C9—C2—C5	110.54 (14)	C6—C7—C8	111.24 (14)
N1—C2—C3	111.60 (13)	H7A—C7—H7B	108.0
N1—C2—C5	106.26 (13)	C8—C7—H7A	109.4
N1—C2—C9	108.69 (13)	C8—C7—H7B	109.4
N4—C3—C2	176.58 (18)	C7—C8—H8A	109.3
C2—C5—H5A	109.3	C7—C8—H8B	109.3
C2—C5—H5B	109.3	H8A—C8—H8B	108.0
H5A—C5—H5B	107.9	C9—C8—C7	111.40 (14)
C6—C5—C2	111.80 (14)	C9—C8—H8A	109.3
C6—C5—H5A	109.3	C9—C8—H8B	109.3
C6—C5—H5B	109.3	C2—C9—H9A	109.4
C5—C6—H6A	109.3	C2—C9—H9B	109.4
C5—C6—H6B	109.3	C8—C9—C2	111.26 (14)
H6A—C6—H6B	108.0	C8—C9—H9A	109.4
C7—C6—C5	111.41 (14)	C8—C9—H9B	109.4
C7—C6—H6A	109.3	H9A—C9—H9B	108.0
C7—C6—H6B	109.3	N1 ⁱ —N1—C2	114.07 (18)
C2—C5—C6—C7	-54.82 (19)	C6—C7—C8—C9	-56.00 (19)
C3—C2—C5—C6	-66.44 (18)	C7—C8—C9—C2	56.17 (19)
C3—C2—C9—C8	66.81 (18)	C9—C2—C5—C6	54.51 (19)
C3—C2—N1—N1 ⁱ	-15.1 (2)	C9—C2—N1—N1 ⁱ	-135.45 (18)
C5—C2—C9—C8	-55.02 (19)	N1—C2—C5—C6	172.27 (13)
C5—C2—N1—N1 ⁱ	105.57 (19)	N1—C2—C9—C8	-171.28 (13)
C5—C6—C7—C8	55.17 (19)		

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