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N,*N*-Dicyclohexylnitramine

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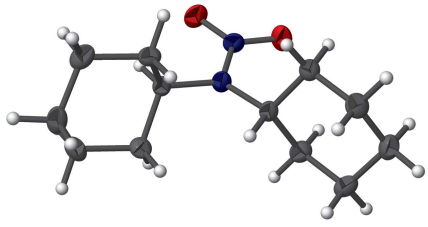
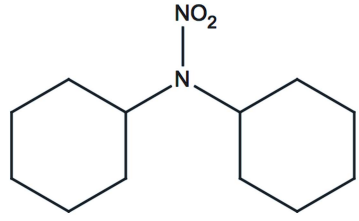
Molecules of the title compound, $C_{12}H_{22}N_2O_2$, are composed of an nitramine group substituted by two cyclohexane rings. The cyclohexane rings have chair conformations, with the exocyclic C—N bonds in axial orientations. In the crystal, C—H···O hydrogen bonds connect the molecules into $C(6)$ [$\bar{1}01$] zigzag chains.

3D view



Chemical scheme



Structure description

The study of the chemistry of aliphatic nitrosoamines began in 1863 when Geuther obtained *N*-nitrosodiethylamine by the reaction of diethylamine hydrochloride with sodium nitrite (Taylor & Price, 1929). Many nitrosamines showed carcinogenic properties for animals but it is difficult to determine these properties for humans because of the very low typical absorption. For this reason, determination of the danger for humans is very complicated (Crosby & Sawyer, 1976).

The amide group is substituted by two cyclohexane rings (Fig. 1). The N1—N2 bond length is notably shorter [1.3509 (13) Å] than the distance characteristic of an N—N single bond (1.42 Å) but longer than the distance of an N═N double bond (1.24 Å), indicating partial double-bond character. The geometry of the nitramine group is typical, and corresponds well with similar compounds (Prezhdo *et al.*, 2001*b*; Zarychta *et al.*, 2005*a*,*b*, 2011). Both cyclohexane rings have chair conformations with the exocyclic C—N bonds in axial orientations.

The crystal structure features weak C—H···O interactions, which connect the molecules into [$\bar{1}01$] $C_1^1(6)$ zigzag chains (Table 1). The packing is shown in Fig. 2.

Synthesis and crystallization

N,*N*-dimethylnitramine was prepared as follows (Prezhdo *et al.*, 2001*a*). To a solution of 1.05 g of *N*-nitrosodicyclohexylamine in 50 ml of acetone was added 25 ml of a buffer

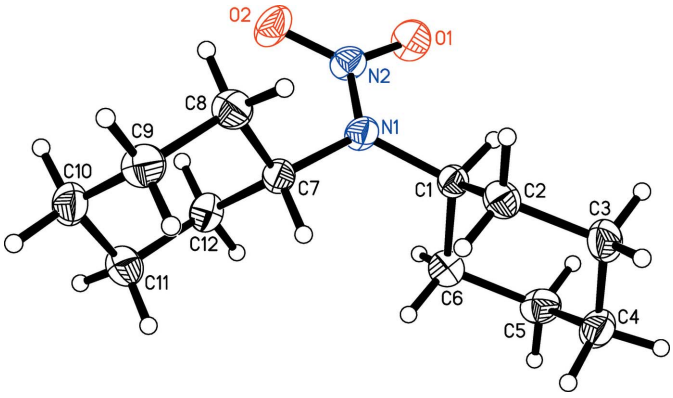


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

solution containing 6 g of K_2HPO_4 and 0.7 g of KH_2PO_4 and then, in four portions over a period of 4 h, 3 g of oxone ($2KHSO_5{\cdot}KHSO_4{\cdot}K_2SO_4$) and stirred for the next 3 h. The product was extracted with methylene chloride (4 × 25 ml). The extract was dried over anhydrous magnesium sulfate, the solvent was evaporated, and the residue was recrystallized from iso-octane solution. Yield = 0.8 g, m.p. 137–138°C.

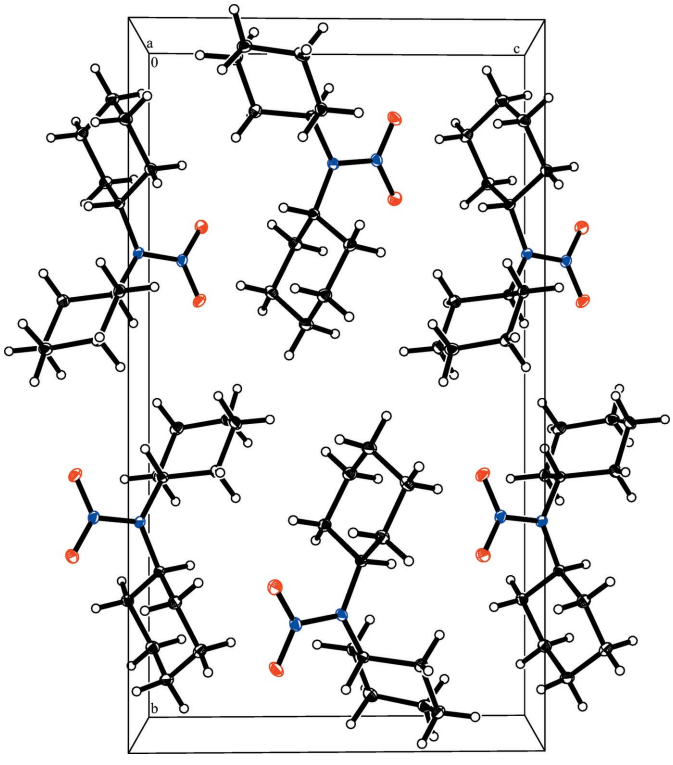


Figure 2
The crystal packing of the title compound, viewed along the *a* axis.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O2^{i}	0.97	2.60	3.5684 (14)	176

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{22}N_2O_2$
M_r	226.31
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	5.9136 (1), 19.1658 (4), 10.8946 (2)
β (°)	91.852 (2)
V (Å^3)	1234.14 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.04 × 0.04 × 0.03
Data collection	
Diffractometer	Oxford Diffraction Xcalibur CCD
Absorption correction	–
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8287, 2425, 1892
R_{int}	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.616
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.084, 1.07
No. of reflections	2425
No. of parameters	145
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.15

Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2008), *SHELXS2014* and *SHELXTL* (Sheldrick, 2008) and *SHELXL2014* (Sheldrick, 2015).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2016). **1**, x161513 [doi:10.1107/S2414314616015133]

N,*N*-Dicyclohexylnitramine

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N-Cyclohexyl-*N*-nitrocyclohexanamine

Crystal data

$C_{12}H_{22}N_2O_2$
M_r = 226.31
Monoclinic, $P2_1/n$
a = 5.9136 (1) Å
b = 19.1658 (4) Å
c = 10.8946 (2) Å
β = 91.852 (2)°
V = 1234.14 (4) Å^3
Z = 4
$F(000)$ = 496
D_x = 1.218 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8287 reflections
θ = 3.6–26.0°
μ = 0.08 mm^{-1}
T = 100 K
Plate, colourless
0.04 × 0.04 × 0.03 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 1024 x 1024 with blocks 2 x 2 pixels mm^{-1}
ω–scan
8287 measured reflections
2425 independent reflections
1892 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.0°, θ_{min} = 3.6°
h = −7→7
k = −23→23
l = −13→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.084
S = 1.07
2425 reflections
145 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.1204P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.72029 (16)	0.12483 (4)	0.64176 (8)	0.0372 (2)

O2	0.64140 (15)	0.23560 (5)	0.64377 (8)	0.0380 (2)
N1	0.83491 (16)	0.19259 (5)	0.49004 (9)	0.0257 (2)
N2	0.72928 (17)	0.18401 (5)	0.59711 (9)	0.0269 (2)
C1	0.94810 (19)	0.13124 (6)	0.43775 (10)	0.0229 (3)
H1A	0.8895	0.0901	0.4795	0.027*
C2	0.8906 (2)	0.12227 (6)	0.30137 (11)	0.0272 (3)
H2A	0.9519	0.1609	0.2556	0.033*
H2B	0.7277	0.1218	0.2877	0.033*
C3	0.9913 (2)	0.05376 (6)	0.25735 (12)	0.0332 (3)
H3A	0.9191	0.0151	0.2982	0.040*
H3B	0.9610	0.0489	0.1697	0.040*
C4	1.2452 (2)	0.05060 (7)	0.28348 (12)	0.0334 (3)
H4A	1.3010	0.0050	0.2606	0.040*
H4B	1.3194	0.0852	0.2337	0.040*
C5	1.3044 (2)	0.06395 (6)	0.41772 (12)	0.0324 (3)
H5A	1.4677	0.0657	0.4293	0.039*
H5B	1.2488	0.0257	0.4665	0.039*
C6	1.20285 (19)	0.13213 (6)	0.46228 (11)	0.0267 (3)
H6A	1.2356	0.1377	0.5495	0.032*
H6B	1.2693	0.1711	0.4197	0.032*
C7	0.87648 (19)	0.26368 (6)	0.44305 (11)	0.0238 (3)
H7A	0.9683	0.2572	0.3706	0.029*
C8	0.6611 (2)	0.30047 (6)	0.39599 (12)	0.0295 (3)
H8A	0.5820	0.2711	0.3361	0.035*
H8B	0.5617	0.3084	0.4637	0.035*
C9	0.7201 (2)	0.36990 (6)	0.33712 (12)	0.0316 (3)
H9A	0.8051	0.3614	0.2639	0.038*
H9B	0.5817	0.3940	0.3125	0.038*
C10	0.8586 (2)	0.41578 (6)	0.42459 (12)	0.0325 (3)
H10A	0.7687	0.4280	0.4943	0.039*
H10B	0.8988	0.4586	0.3831	0.039*
C11	1.0727 (2)	0.37853 (6)	0.46953 (12)	0.0319 (3)
H11A	1.1548	0.4081	0.5279	0.038*
H11B	1.1691	0.3703	0.4006	0.038*
C12	1.0191 (2)	0.30900 (6)	0.53055 (11)	0.0281 (3)
H12A	1.1588	0.2850	0.5529	0.034*
H12B	0.9374	0.3173	0.6049	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0500 (6)	0.0309 (5)	0.0315 (5)	0.0028 (4)	0.0135 (4)	0.0110 (4)
O2	0.0488 (6)	0.0336 (5)	0.0326 (5)	0.0083 (4)	0.0195 (4)	−0.0017 (4)
N1	0.0321 (6)	0.0234 (5)	0.0221 (5)	0.0019 (4)	0.0107 (4)	0.0028 (4)
N2	0.0291 (5)	0.0294 (6)	0.0224 (5)	0.0017 (4)	0.0055 (4)	0.0030 (4)
C1	0.0263 (6)	0.0199 (6)	0.0227 (6)	0.0002 (5)	0.0038 (5)	0.0010 (5)
C2	0.0249 (6)	0.0288 (7)	0.0277 (6)	−0.0029 (5)	−0.0031 (5)	−0.0019 (5)
C3	0.0415 (8)	0.0301 (7)	0.0283 (7)	−0.0070 (6)	0.0042 (6)	−0.0075 (5)

C4	0.0401 (7)	0.0249 (7)	0.0359 (7)	0.0043 (6)	0.0120 (6)	−0.0001 (5)
C5	0.0280 (7)	0.0325 (7)	0.0370 (7)	0.0061 (5)	0.0040 (5)	0.0077 (6)
C6	0.0277 (7)	0.0289 (6)	0.0232 (6)	0.0001 (5)	−0.0034 (5)	0.0019 (5)
C7	0.0283 (6)	0.0208 (6)	0.0227 (6)	0.0008 (5)	0.0055 (5)	0.0015 (5)
C8	0.0273 (6)	0.0295 (7)	0.0315 (7)	0.0001 (5)	−0.0007 (5)	0.0029 (5)
C9	0.0317 (7)	0.0322 (7)	0.0308 (7)	0.0048 (5)	0.0007 (5)	0.0082 (5)
C10	0.0393 (7)	0.0235 (6)	0.0351 (7)	0.0029 (6)	0.0065 (6)	0.0018 (5)
C11	0.0322 (7)	0.0290 (7)	0.0345 (7)	−0.0045 (5)	−0.0006 (5)	−0.0023 (5)
C12	0.0308 (6)	0.0260 (6)	0.0274 (6)	0.0037 (5)	−0.0016 (5)	−0.0014 (5)

Geometric parameters (Å, º)

O1—N2	1.2360 (12)	C6—H6A	0.9700
O2—N2	1.2342 (12)	C6—H6B	0.9700
N1—N2	1.3509 (13)	C7—C12	1.5238 (16)
N1—C1	1.4764 (14)	C7—C8	1.5297 (16)
N1—C7	1.4789 (14)	C7—H7A	0.9800
C1—C6	1.5216 (16)	C8—C9	1.5225 (16)
C1—C2	1.5231 (16)	C8—H8A	0.9700
C1—H1A	0.9800	C8—H8B	0.9700
C2—C3	1.5255 (17)	C9—C10	1.5172 (18)
C2—H2A	0.9700	C9—H9A	0.9700
C2—H2B	0.9700	C9—H9B	0.9700
C3—C4	1.5202 (18)	C10—C11	1.5206 (18)
C3—H3A	0.9700	C10—H10A	0.9700
C3—H3B	0.9700	C10—H10B	0.9700
C4—C5	1.5145 (18)	C11—C12	1.5271 (17)
C4—H4A	0.9700	C11—H11A	0.9700
C4—H4B	0.9700	C11—H11B	0.9700
C5—C6	1.5240 (16)	C12—H12A	0.9700
C5—H5A	0.9700	C12—H12B	0.9700
C5—H5B	0.9700		
N2—N1—C1	117.66 (9)	C1—C6—H6B	109.8
N2—N1—C7	119.84 (9)	C5—C6—H6B	109.8
C1—N1—C7	121.18 (9)	H6A—C6—H6B	108.2
O2—N2—O1	123.29 (9)	N1—C7—C12	113.79 (9)
O2—N2—N1	118.09 (9)	N1—C7—C8	113.25 (9)
O1—N2—N1	118.59 (9)	C12—C7—C8	112.64 (10)
N1—C1—C6	112.45 (9)	N1—C7—H7A	105.4
N1—C1—C2	112.12 (9)	C12—C7—H7A	105.4
C6—C1—C2	111.17 (9)	C8—C7—H7A	105.4
N1—C1—H1A	106.9	C9—C8—C7	110.15 (10)
C6—C1—H1A	106.9	C9—C8—H8A	109.6
C2—C1—H1A	106.9	C7—C8—H8A	109.6
C1—C2—C3	109.07 (10)	C9—C8—H8B	109.6
C1—C2—H2A	109.9	C7—C8—H8B	109.6
C3—C2—H2A	109.9	H8A—C8—H8B	108.1

C1—C2—H2B	109.9	C10—C9—C8	111.61 (10)
C3—C2—H2B	109.9	C10—C9—H9A	109.3
H2A—C2—H2B	108.3	C8—C9—H9A	109.3
C4—C3—C2	111.63 (10)	C10—C9—H9B	109.3
C4—C3—H3A	109.3	C8—C9—H9B	109.3
C2—C3—H3A	109.3	H9A—C9—H9B	108.0
C4—C3—H3B	109.3	C9—C10—C11	110.93 (10)
C2—C3—H3B	109.3	C9—C10—H10A	109.5
H3A—C3—H3B	108.0	C11—C10—H10A	109.5
C5—C4—C3	111.73 (10)	C9—C10—H10B	109.5
C5—C4—H4A	109.3	C11—C10—H10B	109.5
C3—C4—H4A	109.3	H10A—C10—H10B	108.0
C5—C4—H4B	109.3	C10—C11—C12	111.54 (10)
C3—C4—H4B	109.3	C10—C11—H11A	109.3
H4A—C4—H4B	107.9	C12—C11—H11A	109.3
C4—C5—C6	111.79 (10)	C10—C11—H11B	109.3
C4—C5—H5A	109.3	C12—C11—H11B	109.3
C6—C5—H5A	109.3	H11A—C11—H11B	108.0
C4—C5—H5B	109.3	C7—C12—C11	110.10 (10)
C6—C5—H5B	109.3	C7—C12—H12A	109.6
H5A—C5—H5B	107.9	C11—C12—H12A	109.6
C1—C6—C5	109.42 (10)	C7—C12—H12B	109.6
C1—C6—H6A	109.8	C11—C12—H12B	109.6
C5—C6—H6A	109.8	H12A—C12—H12B	108.2

C1—N1—N2—O2	177.38 (10)	C2—C1—C6—C5	−59.24 (12)
C7—N1—N2—O2	10.30 (16)	C4—C5—C6—C1	55.84 (13)
C1—N1—N2—O1	−4.46 (15)	N2—N1—C7—C12	58.93 (14)
C7—N1—N2—O1	−171.54 (10)	C1—N1—C7—C12	−107.69 (12)
N2—N1—C1—C6	−100.93 (12)	N2—N1—C7—C8	−71.41 (13)
C7—N1—C1—C6	65.97 (13)	C1—N1—C7—C8	121.98 (11)
N2—N1—C1—C2	132.93 (11)	N1—C7—C8—C9	−174.08 (9)
C7—N1—C1—C2	−60.17 (13)	C12—C7—C8—C9	55.01 (13)
N1—C1—C2—C3	−173.68 (9)	C7—C8—C9—C10	−55.28 (13)
C6—C1—C2—C3	59.49 (12)	C8—C9—C10—C11	56.61 (14)
C1—C2—C3—C4	−56.37 (13)	C9—C10—C11—C12	−56.53 (14)
C2—C3—C4—C5	54.20 (14)	N1—C7—C12—C11	174.42 (9)
C3—C4—C5—C6	−53.84 (14)	C8—C7—C12—C11	−54.94 (13)
N1—C1—C6—C5	174.10 (9)	C10—C11—C12—C7	55.23 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*A*···O2^{i}	0.97	2.60	3.5684 (14)	176

Symmetry code: (i) *x*+1/2, −*y*+1/2, *z*−1/2.