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# (*E*)-1-(2,4-Dinitrophenyl)-2-[1-(2-nitrophenyl)ethylidene]hydrazine

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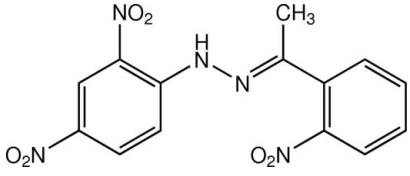
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.041; $wR$ factor = 0.110; data-to-parameter ratio = 18.7.

The title compound, $C_{14}H_{11}N_5O_6$, was obtained from the condensation reaction of 2,4-dinitrophenylhydrazine and 2-nitroacetophenone. The molecule displays an *E* conformation about the C=N double bond and an intramolecular N—H···O hydrogen bond generates an *S*(6) ring motif. The dihedral angle between the benzene rings is 7.84 (6)°. In the crystal, molecules are linked by C—H···O hydrogen bonds and $\pi$–$\pi$ stacking interactions [centroid–centroid distance = 3.6447 (8) Å] into a three-dimensional network.

## Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Fun *et al.* (2011); Shan *et al.* (2003). For background to and the physiological and biological activity of hydrozones, see: Bendre *et al.* (1998); Nakamura & Goto (1996); Rollas & Küçükgüzel (2007); Singh *et al.* (2005); Yacorb (1999). For the stability of the temperature controller used in the data collection, see Cosier & Glazer (1986).




## Experimental

### Crystal data

$C_{14}H_{11}N_5O_6$ $M_r$ = 345.28
Monoclinic, $P2_1/c$
$a$ = 11.9313 (9) Å
$b$ = 8.6700 (7) Å
$c$ = 15.2363 (9) Å
$\beta$ = 112.455 (5)°
$V$ = 1456.61 (19) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.13 mm$^{-1}$
$T$ = 100 K
0.40 × 0.16 × 0.13 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.951, $T_{max}$ = 0.984
16360 measured reflections
4244 independent reflections
3361 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.110
$S$ = 1.04
4244 reflections
227 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O2 | 0.88 | 1.94 | 2.6026 (13) | 131 |
| C10—H10$A$···O4$^{i}$ | 0.93 | 2.42 | 3.2313 (16) | 146 |
| C12—H12$A$···O4$^{ii}$ | 0.93 | 2.55 | 3.4353 (18) | 159 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2643).

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# supporting information

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## (*E*)-1-(2,4-Dinitrophenyl)-2-[1-(2-nitrophenyl)ethylidene]hydrazine

**Boonlerd Nilwanna, Suchada Chantrapromma, Patcharaporn Jansrisewangwong and Hoong-Kun Fun**

### S1. Comment

Hydrazones exhibit physiological and biological activities in the treatment of several diseases with anticonvulsant, antidepressant, analgesic, antiinflammatory, antiplatelet, antimalarial, antimicrobial, antimycobacterial, antitumor, vasodilator, antiviral, antischistosomiasis (Singh *et al.*, 2005; Rollas & Küçükgüzel, 2007) and tyrosinase inhibitory properties (Bendre *et al.*, 1998). Furthermore, they were used in engineering and analytical studies for aldehydes and ketones sampling (Yacorb, 1999) and analysis of protein carbonyls (Nakamura & Goto, 1996). These interesting activities have led us to synthesize the title hydrazone derivative (I). It was screened for antioxidant and antibacterial activities. Our results found that (I) is inactive for these tests. Herein we report the synthesis and crystal structure of (I).

The whole molecule of (I) (Fig. 1), $C_{14}H_{11}N_5O_6$, is not planar and exists in an *E* configuration with respect to the ethylidene C═N double bond [1.2905 (15) Å] with the torsion angle N1–N2–C7–C8 = 177.22 (10)°. The dihedral angle between the two benzene rings is 7.84 (6)°. The middle ethylidenehydrazine fragment (C7/C14/N1/N2) is planar with the *r.m.s* deviation of 0.0047 (1) Å. This middle C/C/N/N plane makes the dihedral angles of 11.28 (8) and 9.78 (8)° with the C1–C6 and C8–C13 benzene rings, respectively. The two nitro groups of 2,4-dinitrophenyl are co-planar with the bound benzene ring with the *r.m.s*. deviation of 0.0369 (1) Å for the twelve non H-atoms. However the nitro group of the 2-nitrophenyl tilts away from its bound benzene ring with the dihedral angle of 81.19 (7)° between the C9/N5/O5/O6 plane and C8–C13 benzene ring. This orientation is caused by the steric interaction between the hydrazine and nitro group. An intramolecular N1—H1···O2 hydrogen bond between the hydrazone-NH and the *ortho* nitro group (Fig. 1 and Table 1) generates an S(6) ring motif (Bernstein *et al.*, 1995). The bond distances are within the expected range (Allen *et al.*, 1987) and are comparable with those of related structures (Fun *et al.*, 2011; Shan *et al.*, 2003).

In the crystal structure, the molecules are linked by weak C—H···O hydrogen bonds (Table 1) into a three-dimensional network (Fig. 2) enforced by $\pi\cdots\pi$ stacking interactions ($Cg_1\cdots Cg_2^{\,i}$ = 3.6447 (8) Å; $Cg_1$ and $Cg_2$ are the centroids of C1–C6 and C8–C13 benzenre rings, respectively; symmetry code: (i) 2 - *x*, 2 - *y*, 1 - *z*). Short C···O (2.9999 (15) Å] contacts are also observed.

### S2. Experimental

The title compound was synthesized by dissolving 2,4-dinitrophenylhydrazine (0.40 g, 2 mmol) in ethanol (10.00 ml), and $H_2SO_4$ (98%, 0.50 ml) was slowly added with stirring. 2-Nitroacetophenone (0.27 ml, 2 mmol) was then added to the solution with continuous stirring. The solution was refluxed for 1 h yielding a yellow solid, which was filtered off and washed with methanol. Yellow block-shaped single crystals of the title compound suitable for *X*-ray structure determination were recrystalized from ethanol by slow evaporation of the solvent at room temperature over several days. M.p. 443–444 K.

### S3. Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(N—H) = 0.88 Å, d(C—H) = 0.93 Å for aromatic and 0.96 Å for $CH_3$ atoms. The $U_{iso}$ values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

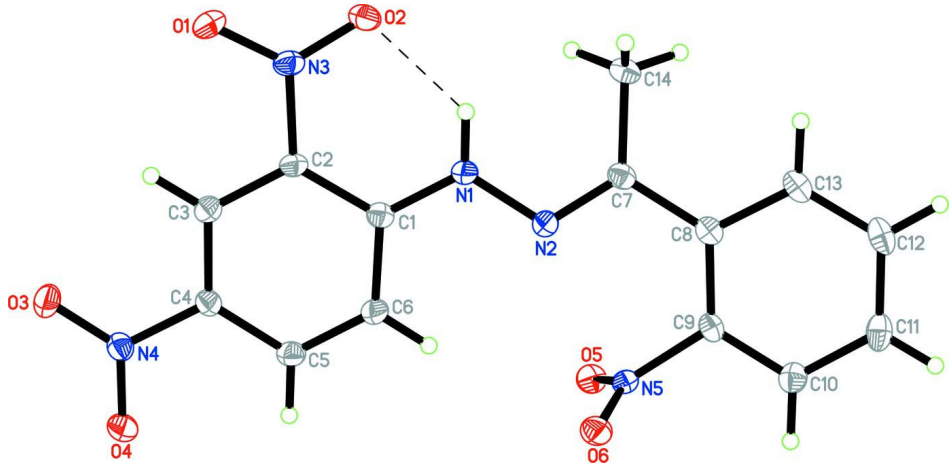


**Figure 1**

The molecular structure of the title compound, showing 50% probability displacement ellipsoids. Hydrogen bond is shown as a dashed line.

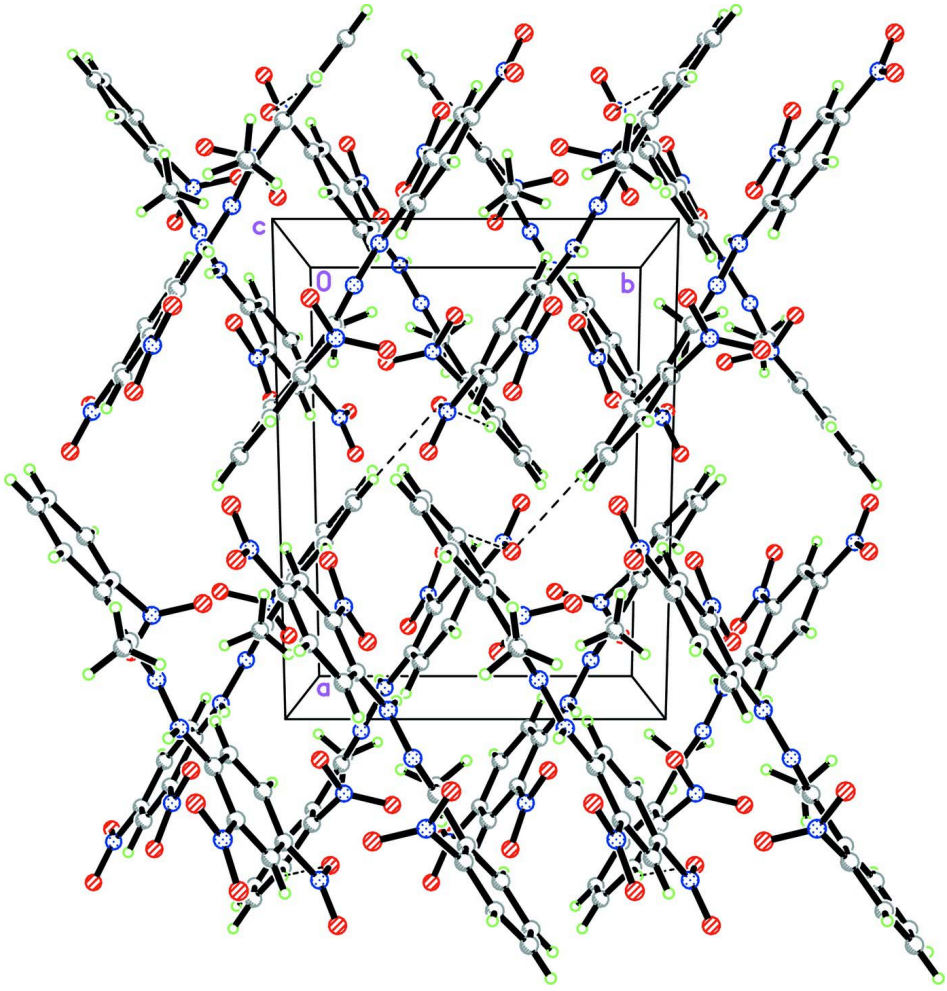


**Figure 2**

The crystal packing of the title compound viewed along the *c* axis, showing molecular stacking along the *b* axis. Hydrogen bonds are shown as dashed lines.

### (*E*)-1-(2,4-Dinitrophenyl)-2-[1-(2-nitrophenyl)ethylidene]hydrazine

*Crystal data*

$C_{14}H_{11}N_5O_6$
$M_r = 345.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.9313\ (9)$ Å
$b = 8.6700\ (7)$ Å
$c = 15.2363\ (9)$ Å
$\beta = 112.455\ (5)°$
$V = 1456.61\ (19)$ Å$^3$
$Z = 4$

$F(000) = 712$
$D_x = 1.574$ Mg m$^{-3}$
Melting point = 443–444 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4244 reflections
$\theta = 1.9–30.0°$
$\mu = 0.13$ mm$^{-1}$
$T = 100$ K
Block, yellow
0.40 × 0.16 × 0.13 mm

*Data collection*

Bruker APEXII CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.951, $T_{max}$ = 0.984
16360 measured reflections
4244 independent reflections
3361 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033
$\theta_{max}$ = 30.0°, $\theta_{min}$ = 1.9°
$h$ = −16→16
$k$ = −9→12
$l$ = −21→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.110
$S$ = 1.04
4244 reflections
227 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
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.498P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

**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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 1.32132 (8) | 0.59779 (12) | 0.77977 (6) | 0.0221 (2) |
| O2 | 1.14527 (9) | 0.70362 (12) | 0.74461 (6) | 0.0233 (2) |
| O3 | 1.44309 (8) | 0.36901 (13) | 0.55130 (7) | 0.0265 (2) |
| O4 | 1.33701 (8) | 0.39821 (12) | 0.40099 (6) | 0.0229 (2) |
| O5 | 0.76820 (8) | 0.73691 (11) | 0.30121 (6) | 0.0191 (2) |
| O6 | 0.87386 (8) | 0.93847 (12) | 0.29701 (6) | 0.0200 (2) |
| N1 | 1.01173 (9) | 0.76742 (13) | 0.56858 (7) | 0.0166 (2) |
| H1 | 1.0252 | 0.7860 | 0.6283 | 0.020* |
| N2 | 0.91649 (9) | 0.83451 (13) | 0.49714 (7) | 0.0160 (2) |
| N3 | 1.22384 (9) | 0.64213 (13) | 0.72129 (7) | 0.0168 (2) |
| N4 | 1.35479 (9) | 0.42076 (13) | 0.48519 (7) | 0.0168 (2) |
| N5 | 0.79296 (9) | 0.87393 (13) | 0.31434 (7) | 0.0147 (2) |
| C1 | 1.09536 (10) | 0.68522 (15) | 0.54842 (8) | 0.0145 (2) |
| C2 | 1.19951 (11) | 0.62140 (14) | 0.62097 (8) | 0.0143 (2) |

| | | | | |
|---|---|---|---|---|
| C3 | 1.28425 (10) | 0.53651 (14) | 0.59994 (8) | 0.0151 (2) |
| H3A | 1.3523 | 0.4966 | 0.6483 | 0.018* |
| C4 | 1.26628 (10) | 0.51219 (15) | 0.50638 (8) | 0.0151 (2) |
| C5 | 1.16558 (11) | 0.57308 (15) | 0.43212 (8) | 0.0170 (2) |
| H5A | 1.1555 | 0.5560 | 0.3693 | 0.020* |
| C6 | 1.08201 (11) | 0.65806 (16) | 0.45296 (8) | 0.0171 (2) |
| H6A | 1.0153 | 0.6987 | 0.4037 | 0.021* |
| C7 | 0.83449 (11) | 0.89627 (15) | 0.52181 (8) | 0.0152 (2) |
| C8 | 0.73396 (10) | 0.97562 (15) | 0.44578 (8) | 0.0156 (2) |
| C9 | 0.71612 (10) | 0.97024 (14) | 0.34927 (8) | 0.0143 (2) |
| C10 | 0.62471 (11) | 1.04965 (15) | 0.27935 (9) | 0.0182 (3) |
| H10A | 0.6165 | 1.0430 | 0.2163 | 0.022* |
| C11 | 0.54516 (11) | 1.13956 (16) | 0.30456 (10) | 0.0207 (3) |
| H11A | 0.4835 | 1.1942 | 0.2585 | 0.025* |
| C12 | 0.55861 (11) | 1.14699 (17) | 0.39886 (10) | 0.0214 (3) |
| H12A | 0.5048 | 1.2056 | 0.4159 | 0.026* |
| C13 | 0.65176 (11) | 1.06765 (16) | 0.46806 (9) | 0.0192 (3) |
| H13A | 0.6600 | 1.0757 | 0.5311 | 0.023* |
| C14 | 0.83892 (12) | 0.89321 (17) | 0.62186 (9) | 0.0212 (3) |
| H14A | 0.8708 | 0.7959 | 0.6507 | 0.032* |
| H14B | 0.7586 | 0.9067 | 0.6209 | 0.032* |
| H14C | 0.8902 | 0.9750 | 0.6578 | 0.032* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0217 (4) | 0.0283 (6) | 0.0134 (4) | 0.0021 (4) | 0.0035 (3) | 0.0030 (4) |
| O2 | 0.0306 (5) | 0.0261 (6) | 0.0165 (4) | 0.0090 (4) | 0.0126 (4) | 0.0021 (4) |
| O3 | 0.0211 (4) | 0.0347 (6) | 0.0202 (5) | 0.0117 (4) | 0.0041 (4) | 0.0004 (4) |
| O4 | 0.0234 (4) | 0.0294 (6) | 0.0170 (4) | 0.0045 (4) | 0.0091 (4) | −0.0032 (4) |
| O5 | 0.0240 (4) | 0.0140 (5) | 0.0189 (4) | −0.0013 (4) | 0.0079 (4) | −0.0031 (3) |
| O6 | 0.0177 (4) | 0.0241 (5) | 0.0204 (4) | −0.0010 (4) | 0.0098 (3) | 0.0028 (4) |
| N1 | 0.0187 (5) | 0.0190 (6) | 0.0121 (4) | 0.0042 (4) | 0.0059 (4) | 0.0001 (4) |
| N2 | 0.0165 (4) | 0.0168 (6) | 0.0141 (4) | 0.0026 (4) | 0.0053 (4) | 0.0002 (4) |
| N3 | 0.0218 (5) | 0.0157 (6) | 0.0134 (4) | 0.0002 (4) | 0.0074 (4) | 0.0019 (4) |
| N4 | 0.0160 (4) | 0.0171 (6) | 0.0177 (5) | 0.0008 (4) | 0.0070 (4) | −0.0014 (4) |
| N5 | 0.0150 (4) | 0.0171 (6) | 0.0113 (4) | 0.0008 (4) | 0.0042 (4) | 0.0006 (4) |
| C1 | 0.0166 (5) | 0.0130 (6) | 0.0147 (5) | −0.0006 (4) | 0.0067 (4) | −0.0006 (4) |
| C2 | 0.0178 (5) | 0.0138 (6) | 0.0116 (5) | −0.0004 (4) | 0.0060 (4) | 0.0009 (4) |
| C3 | 0.0156 (5) | 0.0140 (6) | 0.0151 (5) | −0.0008 (4) | 0.0053 (4) | 0.0013 (4) |
| C4 | 0.0159 (5) | 0.0146 (6) | 0.0160 (5) | 0.0006 (4) | 0.0073 (4) | −0.0005 (4) |
| C5 | 0.0193 (5) | 0.0185 (7) | 0.0133 (5) | 0.0008 (5) | 0.0065 (4) | −0.0008 (5) |
| C6 | 0.0183 (5) | 0.0192 (7) | 0.0128 (5) | 0.0024 (5) | 0.0047 (4) | −0.0003 (5) |
| C7 | 0.0186 (5) | 0.0128 (6) | 0.0164 (5) | −0.0011 (4) | 0.0091 (4) | −0.0011 (4) |
| C8 | 0.0155 (5) | 0.0143 (6) | 0.0182 (5) | −0.0016 (4) | 0.0078 (4) | −0.0018 (4) |
| C9 | 0.0135 (5) | 0.0119 (6) | 0.0183 (5) | −0.0013 (4) | 0.0070 (4) | −0.0026 (4) |
| C10 | 0.0166 (5) | 0.0175 (7) | 0.0182 (5) | −0.0004 (5) | 0.0040 (4) | −0.0016 (5) |
| C11 | 0.0149 (5) | 0.0167 (7) | 0.0263 (6) | 0.0011 (5) | 0.0030 (5) | −0.0016 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C12 | 0.0159 (5) | 0.0193 (7) | 0.0303 (7) | 0.0011 (5) | 0.0102 (5) | −0.0051 (5) |
| C13 | 0.0190 (5) | 0.0180 (7) | 0.0237 (6) | −0.0003 (5) | 0.0118 (5) | −0.0035 (5) |
| C14 | 0.0239 (6) | 0.0262 (8) | 0.0172 (6) | 0.0030 (5) | 0.0121 (5) | 0.0011 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—N3 | 1.2259 (13) | C5—C6 | 1.3710 (17) |
| O2—N3 | 1.2424 (14) | C5—H5A | 0.9300 |
| O3—N4 | 1.2309 (14) | C6—H6A | 0.9300 |
| O4—N4 | 1.2332 (13) | C7—C8 | 1.4807 (17) |
| O5—N5 | 1.2220 (14) | C7—C14 | 1.5051 (16) |
| O6—N5 | 1.2284 (13) | C8—C13 | 1.4029 (16) |
| N1—C1 | 1.3535 (15) | C8—C9 | 1.4037 (16) |
| N1—N2 | 1.3672 (14) | C9—C10 | 1.3824 (17) |
| N1—H1 | 0.8766 | C10—C11 | 1.3909 (18) |
| N2—C7 | 1.2909 (15) | C10—H10A | 0.9300 |
| N3—C2 | 1.4539 (14) | C11—C12 | 1.3849 (19) |
| N4—C4 | 1.4521 (15) | C11—H11A | 0.9300 |
| N5—C9 | 1.4812 (15) | C12—C13 | 1.3874 (18) |
| C1—C6 | 1.4210 (16) | C12—H12A | 0.9300 |
| C1—C2 | 1.4224 (16) | C13—H13A | 0.9300 |
| C2—C3 | 1.3835 (16) | C14—H14A | 0.9600 |
| C3—C4 | 1.3744 (16) | C14—H14B | 0.9600 |
| C3—H3A | 0.9300 | C14—H14C | 0.9600 |
| C4—C5 | 1.4014 (16) | | |
| | | | |
| C1—N1—N2 | 120.26 (10) | C5—C6—H6A | 119.4 |
| C1—N1—H1 | 118.2 | C1—C6—H6A | 119.4 |
| N2—N1—H1 | 121.0 | N2—C7—C8 | 116.26 (10) |
| C7—N2—N1 | 115.88 (10) | N2—C7—C14 | 123.38 (11) |
| O1—N3—O2 | 122.46 (10) | C8—C7—C14 | 120.34 (10) |
| O1—N3—C2 | 118.58 (10) | C13—C8—C9 | 115.60 (11) |
| O2—N3—C2 | 118.96 (10) | C13—C8—C7 | 120.47 (11) |
| O3—N4—O4 | 123.16 (10) | C9—C8—C7 | 123.89 (11) |
| O3—N4—C4 | 119.00 (10) | C10—C9—C8 | 123.37 (11) |
| O4—N4—C4 | 117.84 (10) | C10—C9—N5 | 114.72 (10) |
| O5—N5—O6 | 124.63 (10) | C8—C9—N5 | 121.88 (10) |
| O5—N5—C9 | 117.57 (10) | C9—C10—C11 | 119.14 (12) |
| O6—N5—C9 | 117.73 (10) | C9—C10—H10A | 120.4 |
| N1—C1—C6 | 121.03 (11) | C11—C10—H10A | 120.4 |
| N1—C1—C2 | 121.97 (10) | C12—C11—C10 | 119.46 (12) |
| C6—C1—C2 | 117.01 (11) | C12—C11—H11A | 120.3 |
| C3—C2—C1 | 121.72 (10) | C10—C11—H11A | 120.3 |
| C3—C2—N3 | 116.05 (10) | C11—C12—C13 | 120.47 (12) |
| C1—C2—N3 | 122.23 (10) | C11—C12—H12A | 119.8 |
| C4—C3—C2 | 118.92 (11) | C13—C12—H12A | 119.8 |
| C4—C3—H3A | 120.5 | C12—C13—C8 | 121.95 (12) |
| C2—C3—H3A | 120.5 | C12—C13—H13A | 119.0 |

| | | | |
|---|---|---|---|
| C3—C4—C5 | 121.71 (11) | C8—C13—H13A | 119.0 |
| C3—C4—N4 | 118.41 (10) | C7—C14—H14A | 109.5 |
| C5—C4—N4 | 119.88 (10) | C7—C14—H14B | 109.5 |
| C6—C5—C4 | 119.37 (11) | H14A—C14—H14B | 109.5 |
| C6—C5—H5A | 120.3 | C7—C14—H14C | 109.5 |
| C4—C5—H5A | 120.3 | H14A—C14—H14C | 109.5 |
| C5—C6—C1 | 121.27 (11) | H14B—C14—H14C | 109.5 |
| | | | |
| C1—N1—N2—C7 | 172.81 (11) | C2—C1—C6—C5 | 0.48 (19) |
| N2—N1—C1—C6 | −4.25 (18) | N1—N2—C7—C8 | 177.22 (10) |
| N2—N1—C1—C2 | 176.20 (11) | N1—N2—C7—C14 | −1.52 (18) |
| N1—C1—C2—C3 | 179.52 (11) | N2—C7—C8—C13 | −169.40 (12) |
| C6—C1—C2—C3 | −0.04 (18) | C14—C7—C8—C13 | 9.39 (18) |
| N1—C1—C2—N3 | −0.93 (19) | N2—C7—C8—C9 | 8.14 (18) |
| C6—C1—C2—N3 | 179.51 (11) | C14—C7—C8—C9 | −173.07 (12) |
| O1—N3—C2—C3 | 7.08 (17) | C13—C8—C9—C10 | 0.25 (18) |
| O2—N3—C2—C3 | −172.76 (11) | C7—C8—C9—C10 | −177.40 (12) |
| O1—N3—C2—C1 | −172.51 (12) | C13—C8—C9—N5 | −177.90 (11) |
| O2—N3—C2—C1 | 7.65 (18) | C7—C8—C9—N5 | 4.45 (18) |
| C1—C2—C3—C4 | −0.68 (19) | O5—N5—C9—C10 | −96.72 (13) |
| N3—C2—C3—C4 | 179.74 (11) | O6—N5—C9—C10 | 80.43 (13) |
| C2—C3—C4—C5 | 1.00 (19) | O5—N5—C9—C8 | 81.58 (14) |
| C2—C3—C4—N4 | −178.99 (11) | O6—N5—C9—C8 | −101.27 (13) |
| O3—N4—C4—C3 | −0.47 (18) | C8—C9—C10—C11 | −0.28 (19) |
| O4—N4—C4—C3 | 179.19 (11) | N5—C9—C10—C11 | 177.99 (11) |
| O3—N4—C4—C5 | 179.54 (12) | C9—C10—C11—C12 | −0.39 (19) |
| O4—N4—C4—C5 | −0.81 (17) | C10—C11—C12—C13 | 1.1 (2) |
| C3—C4—C5—C6 | −0.6 (2) | C11—C12—C13—C8 | −1.1 (2) |
| N4—C4—C5—C6 | 179.41 (12) | C9—C8—C13—C12 | 0.45 (18) |
| C4—C5—C6—C1 | −0.2 (2) | C7—C8—C13—C12 | 178.18 (12) |
| N1—C1—C6—C5 | −179.09 (12) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O2 | 0.88 | 1.94 | 2.6026 (13) | 131 |
| C10—H10*A*···O4$^{i}$ | 0.93 | 2.42 | 3.2313 (16) | 146 |
| C12—H12*A*···O4$^{ii}$ | 0.93 | 2.55 | 3.4353 (18) | 159 |

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