

2-Methoxybenzaldehyde 2,4-dinitrophenylhydrazone

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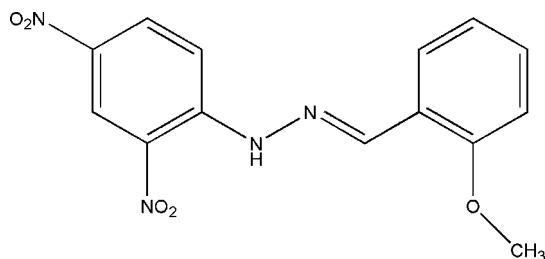
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.045; wR factor = 0.126; data-to-parameter ratio = 23.6.

In the title compound, $\text{C}_{14}\text{H}_{12}\text{N}_4\text{O}_5$, an intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond generates an $S(6)$ ring motif. The dihedral angle between the two benzene rings is $3.91(3)^\circ$, which shows the molecule is almost planar. The *para*-nitro group is twisted from the benzene ring to which it is attached, making a dihedral angle of $8.50(9)^\circ$. In the crystal structure, molecules are linked together by intermolecular $\text{C}-\text{H}\cdots\text{O}$ and intermolecular three-centred $\text{O}\cdots\text{O}$ [$2.8646(12)-2.9213(11)$ Å] and $\text{O}\cdots\text{N}$ [$3.0518(11)$ Å] interactions. The crystal structure is further stabilized by intermolecular $\pi-\pi$ interactions [centroid-to-centroid distances $3.5708(6)-3.9728(12)$ Å].

Related literature

For general background, see: Lambertson *et al.* (1974); Zegota (1999); Cordis *et al.* (1998); Zlotorzynska & Lai (1999); Niknam *et al.* (2005); Guillaumont & Nakamura (2000); Raj & Kurup (2006). For biological applications, see: Okabe *et al.* (1993). Standard bond-length data are given in: Allen *et al.* (1987). For details of the classification of ring motifs, see: Bernstein *et al.* (1995).



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Experimental

Crystal data

$\text{C}_{14}\text{H}_{12}\text{N}_4\text{O}_5$
 $M_r = 316.28$
 Triclinic, $P\bar{1}$
 $a = 7.0315(1)$ Å
 $b = 7.6205(2)$ Å
 $c = 14.1896(4)$ Å
 $\alpha = 98.048(1)^\circ$
 $\beta = 97.064(1)^\circ$
 $\gamma = 109.467(1)^\circ$
 $V = 697.99(3)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹
 $T = 100.0(1)$ K
 $0.57 \times 0.23 \times 0.10$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.936$, $T_{\max} = 0.988$
 14423 measured reflections
 5023 independent reflections
 4442 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.126$
 $S = 1.06$
 5023 reflections
 213 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.52$ e Å⁻³
 $\Delta\rho_{\min} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{N2}-\text{H1N2}\cdots\text{O2}$ | 0.864 (15) | 2.029 (15) | 2.6253 (11) | 125.4 (13) |
| $\text{N2}-\text{H1N2}\cdots\text{O2}^i$ | 0.864 (15) | 2.599 (15) | 3.3475 (11) | 145.6 (13) |
| $\text{C2}-\text{H2A}\cdots\text{O4}^{ii}$ | 0.93 | 2.44 | 3.3113 (12) | 155 |
| $\text{C5}-\text{H5A}\cdots\text{O2}^{iii}$ | 0.93 | 2.60 | 3.3184 (11) | 135 |

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

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); 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, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2139).

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supplementary materials

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2-Methoxybenzaldehyde 2,4-dinitrophenylhydrazone

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Comment

2,4-Dinitrophenylhydrazones play a more important role as stabilizers for the detection, characterization and protection of carbonyl groups than phenylhydrazones (Niknam *et al.*, 2005). 2,4-Dinitrophenylhydrazone derivatives are widely used in various forms of analytical chemistry (Lamberton *et al.*, 1974; Zegota, 1999; Cordis *et al.*, 1998; Zlotorzynska & Lai, 1999) and are also used as dyes (Guillaumont & Nakamura, 2000). They are also found to have versatile coordinating abilities towards different metal ions (Raj & Kurup, 2006). In addition, some phenylhydrazone derivatives have been shown to be potentially DNA-damaging and mutagenic agents (Okabe *et al.*, 1993). For these reasons, the structure of the title compound is reported here.

Bond lengths in the title compound (Fig. 1) are normal (Allen *et al.*, 1987). An intramolecular N—H \cdots O hydrogen bond generates an *S*(6) ring motif (Bernstein *et al.*, 1995). The molecule is nearly planar, with a maximum deviation from the mean plane of -0.3464 (8) Å for atom O4 which is due to the intermolecular three-centered O \cdots O and O \cdots N interactions. The dihedral angle between the two benzene rings is 4.63 (1)°. Interesting features of the crystal structure include intermolecular three-centered O2 \cdots O2ⁱ [2.8646 (12) Å; (i) 1 - *x*, 1 - *y*, -*z*], O4 \cdots O4^{iv} [2.8646 (12) Å; (iv) code: -*x*, -*y*, -1 - *z*], and O4 \cdots N4ⁱⁱ [3.0518 (11) Å] interactions. The molecules are also linked by C—H \cdots O hydrogen bonds (Table 1), and by intermolecular π – π interactions giving centroid–centroid distances for rings C1–C6 (Cg1) and C8–C13 (Cg2) of 3.5708 (6) Å and 3.9728 (12) Å [Cg1 \cdots Cg2^v; (v) -*x*, -*y*, -*z* and Cg1 \cdots Cg2^{vi}; (vi) 1 - *x*, -*y*, -*z*] (interplanar spacings are 3.3080 (4) and 3.3691 (4) Å respectively (Fig. 2).

Experimental

The title compound was synthesized based on the reported procedure (Okabe *et al.* 1993) except that 3-methoxybenzaldehyde (1 mmol, 136 mg) was used instead. Single crystals suitable for X-ray diffraction analysis were grown by slow evaporation of a saturated solution of the resulted compound in ethanol.

Refinement

N-bound H atom was located from the difference Fourier map and refined freely. The remaining H atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{C})$. A rotating group model was applied to the methyl group.

Figures

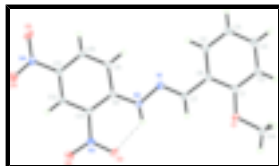


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering scheme. Hydrogen bond is shown as a dashed line.

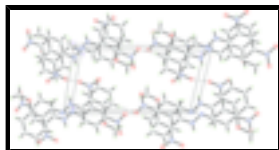


Fig. 2. The crystal packing of the title compound, viewed down the *a* axis, showing the molecules are linked through intermolecular C—H...O and O...O interactions and also are stacked along the *a* axis. Intermolecular interactions are shown as dashed lines.

2-Methoxybenzaldehyde 2,4-dinitrophenylhydrazone

Crystal data

$C_{14}H_{12}N_4O_5$

$M_r = 316.28$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.0315$ (1) Å

$b = 7.6205$ (2) Å

$c = 14.1896$ (4) Å

$\alpha = 98.048$ (1)°

$\beta = 97.064$ (1)°

$\gamma = 109.467$ (1)°

$V = 697.99$ (3) Å³

$Z = 2$

$F_{000} = 328$

$D_x = 1.505$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 6651 reflections

$\theta = 2.9$ – 40.3 °

$\mu = 0.12$ mm⁻¹

$T = 100.0$ (1) K

Block, orange

$0.57 \times 0.23 \times 0.10$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100.0$ (1) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.936$, $T_{\max} = 0.988$

14423 measured reflections

5023 independent reflections

4442 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 32.5$ °

$\theta_{\min} = 1.5$ °

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 11$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$$R[F^2 > 2\sigma(F^2)] = 0.045$$

$$wR(F^2) = 0.126$$

$$S = 1.06$$

5023 reflections

213 parameters

Primary atom site location: structure-invariant direct methods

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2 + 0.1626P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.52 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.24 \text{ e } \text{Å}^{-3}$$

Extinction correction: none

Special details

Experimental. The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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.

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 $F^2 > 2\sigma(F^2)$ 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 (Å^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|--------------|----------------------------------|
| O1 | 0.52355 (11) | 0.19077 (9) | 0.30740 (5) | 0.01789 (14) |
| O2 | 0.31793 (12) | 0.47602 (10) | -0.06604 (5) | 0.02132 (15) |
| O3 | 0.20422 (12) | 0.51054 (10) | -0.20851 (5) | 0.02350 (16) |
| O4 | -0.18137 (11) | -0.00408 (11) | -0.45869 (5) | 0.02104 (15) |
| O5 | -0.19171 (12) | -0.28928 (10) | -0.44929 (5) | 0.02410 (16) |
| N1 | 0.27092 (11) | 0.00256 (11) | 0.02604 (5) | 0.01410 (14) |
| N2 | 0.26656 (12) | 0.13732 (11) | -0.02830 (5) | 0.01416 (14) |
| N3 | 0.22761 (12) | 0.40930 (10) | -0.15138 (6) | 0.01487 (15) |
| N4 | -0.14093 (12) | -0.11921 (11) | -0.41450 (5) | 0.01611 (15) |
| C1 | 0.46732 (13) | 0.00043 (12) | 0.27560 (6) | 0.01377 (15) |
| C2 | 0.48758 (14) | -0.12628 (13) | 0.33510 (6) | 0.01710 (17) |
| H2A | 0.5476 | -0.0815 | 0.4001 | 0.021* |
| C3 | 0.41767 (15) | -0.31935 (14) | 0.29676 (7) | 0.01947 (18) |
| H3A | 0.4306 | -0.4035 | 0.3365 | 0.023* |
| C4 | 0.32856 (14) | -0.38821 (13) | 0.19954 (7) | 0.01804 (17) |
| H4A | 0.2814 | -0.5177 | 0.1745 | 0.022* |
| C5 | 0.31052 (13) | -0.26234 (12) | 0.14015 (6) | 0.01530 (16) |
| H5A | 0.2514 | -0.3085 | 0.0751 | 0.018* |
| C6 | 0.38009 (12) | -0.06701 (12) | 0.17671 (6) | 0.01284 (15) |
| C7 | 0.36703 (13) | 0.06671 (12) | 0.11394 (6) | 0.01371 (15) |
| H7A | 0.4276 | 0.1967 | 0.1373 | 0.016* |
| C8 | 0.17242 (12) | 0.07996 (12) | -0.12228 (6) | 0.01178 (15) |

supplementary materials

| | | | | |
|------|---------------|---------------|--------------|--------------|
| C9 | 0.09189 (13) | -0.11672 (12) | -0.16426 (6) | 0.01378 (15) |
| H9A | 0.1056 | -0.2038 | -0.1266 | 0.017* |
| C10 | -0.00529 (13) | -0.18102 (12) | -0.25882 (6) | 0.01405 (15) |
| H10A | -0.0547 | -0.3101 | -0.2852 | 0.017* |
| C11 | -0.02975 (12) | -0.05112 (12) | -0.31553 (6) | 0.01291 (15) |
| C12 | 0.04681 (12) | 0.14065 (12) | -0.27956 (6) | 0.01311 (15) |
| H12A | 0.0307 | 0.2253 | -0.3184 | 0.016* |
| C13 | 0.14893 (12) | 0.20609 (11) | -0.18400 (6) | 0.01222 (15) |
| C14 | 0.64623 (15) | 0.27271 (14) | 0.40200 (7) | 0.01993 (18) |
| H14A | 0.6876 | 0.4084 | 0.4120 | 0.030* |
| H14B | 0.7657 | 0.2377 | 0.4074 | 0.030* |
| H14C | 0.5675 | 0.2269 | 0.4500 | 0.030* |
| H1N2 | 0.329 (2) | 0.256 (2) | -0.0027 (11) | 0.035 (4)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|------------|-------------|-------------|
| O1 | 0.0227 (3) | 0.0151 (3) | 0.0117 (3) | 0.0049 (2) | -0.0022 (2) | -0.0014 (2) |
| O2 | 0.0294 (4) | 0.0137 (3) | 0.0151 (3) | 0.0053 (3) | -0.0036 (3) | -0.0028 (2) |
| O3 | 0.0317 (4) | 0.0130 (3) | 0.0239 (4) | 0.0077 (3) | -0.0032 (3) | 0.0055 (3) |
| O4 | 0.0218 (3) | 0.0268 (4) | 0.0147 (3) | 0.0103 (3) | -0.0016 (2) | 0.0048 (3) |
| O5 | 0.0274 (4) | 0.0189 (3) | 0.0185 (3) | 0.0055 (3) | -0.0040 (3) | -0.0063 (3) |
| N1 | 0.0153 (3) | 0.0149 (3) | 0.0115 (3) | 0.0049 (2) | 0.0015 (2) | 0.0030 (2) |
| N2 | 0.0173 (3) | 0.0123 (3) | 0.0109 (3) | 0.0042 (3) | -0.0004 (2) | 0.0014 (2) |
| N3 | 0.0162 (3) | 0.0115 (3) | 0.0158 (3) | 0.0051 (2) | 0.0007 (3) | 0.0007 (2) |
| N4 | 0.0144 (3) | 0.0190 (3) | 0.0126 (3) | 0.0052 (3) | -0.0001 (2) | -0.0002 (3) |
| C1 | 0.0130 (3) | 0.0156 (4) | 0.0116 (3) | 0.0044 (3) | 0.0017 (3) | 0.0018 (3) |
| C2 | 0.0164 (4) | 0.0212 (4) | 0.0130 (4) | 0.0057 (3) | 0.0011 (3) | 0.0049 (3) |
| C3 | 0.0199 (4) | 0.0207 (4) | 0.0195 (4) | 0.0072 (3) | 0.0041 (3) | 0.0091 (3) |
| C4 | 0.0191 (4) | 0.0142 (4) | 0.0199 (4) | 0.0042 (3) | 0.0047 (3) | 0.0040 (3) |
| C5 | 0.0151 (3) | 0.0150 (4) | 0.0136 (4) | 0.0032 (3) | 0.0024 (3) | 0.0014 (3) |
| C6 | 0.0123 (3) | 0.0145 (3) | 0.0108 (3) | 0.0040 (3) | 0.0017 (3) | 0.0018 (3) |
| C7 | 0.0145 (3) | 0.0138 (3) | 0.0118 (3) | 0.0042 (3) | 0.0019 (3) | 0.0016 (3) |
| C8 | 0.0120 (3) | 0.0124 (3) | 0.0103 (3) | 0.0042 (3) | 0.0013 (3) | 0.0010 (3) |
| C9 | 0.0160 (3) | 0.0113 (3) | 0.0128 (4) | 0.0039 (3) | 0.0015 (3) | 0.0020 (3) |
| C10 | 0.0151 (3) | 0.0111 (3) | 0.0136 (4) | 0.0030 (3) | 0.0014 (3) | 0.0001 (3) |
| C11 | 0.0119 (3) | 0.0144 (3) | 0.0103 (3) | 0.0034 (3) | 0.0000 (3) | 0.0007 (3) |
| C12 | 0.0128 (3) | 0.0139 (3) | 0.0124 (3) | 0.0051 (3) | 0.0008 (3) | 0.0022 (3) |
| C13 | 0.0128 (3) | 0.0100 (3) | 0.0129 (3) | 0.0039 (3) | 0.0007 (3) | 0.0007 (3) |
| C14 | 0.0188 (4) | 0.0225 (4) | 0.0131 (4) | 0.0048 (3) | -0.0019 (3) | -0.0032 (3) |

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

| | | | |
|--------|-------------|--------|-------------|
| O1—C1 | 1.3618 (11) | C4—C5 | 1.3883 (13) |
| O1—C14 | 1.4344 (11) | C4—H4A | 0.9300 |
| O2—N3 | 1.2456 (10) | C5—C6 | 1.4001 (12) |
| O3—N3 | 1.2274 (10) | C5—H5A | 0.9300 |
| O4—N4 | 1.2329 (10) | C6—C7 | 1.4617 (12) |
| O5—N4 | 1.2328 (10) | C7—H7A | 0.9300 |

| | | | |
|--------------|-------------|----------------|-------------|
| N1—C7 | 1.2858 (11) | C8—C9 | 1.4224 (11) |
| N1—N2 | 1.3736 (10) | C8—C13 | 1.4227 (11) |
| N2—C8 | 1.3545 (10) | C9—C10 | 1.3692 (11) |
| N2—H1N2 | 0.864 (16) | C9—H9A | 0.9300 |
| N3—C13 | 1.4435 (11) | C10—C11 | 1.3998 (12) |
| N4—C11 | 1.4520 (11) | C10—H10A | 0.9300 |
| C1—C2 | 1.3985 (12) | C11—C12 | 1.3730 (12) |
| C1—C6 | 1.4092 (11) | C12—C13 | 1.3912 (11) |
| C2—C3 | 1.3894 (14) | C12—H12A | 0.9300 |
| C2—H2A | 0.9300 | C14—H14A | 0.9600 |
| C3—C4 | 1.3916 (13) | C14—H14B | 0.9600 |
| C3—H3A | 0.9300 | C14—H14C | 0.9600 |
| C1—O1—C14 | 118.36 (7) | C1—C6—C7 | 119.95 (7) |
| C7—N1—N2 | 115.65 (7) | N1—C7—C6 | 119.25 (8) |
| C8—N2—N1 | 118.87 (7) | N1—C7—H7A | 120.4 |
| C8—N2—H1N2 | 121.6 (10) | C6—C7—H7A | 120.4 |
| N1—N2—H1N2 | 119.4 (10) | N2—C8—C9 | 119.67 (7) |
| O3—N3—O2 | 122.20 (7) | N2—C8—C13 | 123.82 (7) |
| O3—N3—C13 | 119.09 (7) | C9—C8—C13 | 116.51 (7) |
| O2—N3—C13 | 118.71 (7) | C10—C9—C8 | 121.61 (8) |
| O5—N4—O4 | 123.66 (8) | C10—C9—H9A | 119.2 |
| O5—N4—C11 | 118.19 (7) | C8—C9—H9A | 119.2 |
| O4—N4—C11 | 118.15 (7) | C9—C10—C11 | 119.52 (8) |
| O1—C1—C2 | 123.98 (8) | C9—C10—H10A | 120.2 |
| O1—C1—C6 | 115.86 (7) | C11—C10—H10A | 120.2 |
| C2—C1—C6 | 120.14 (8) | C12—C11—C10 | 121.62 (8) |
| C3—C2—C1 | 119.74 (8) | C12—C11—N4 | 118.64 (7) |
| C3—C2—H2A | 120.1 | C10—C11—N4 | 119.74 (7) |
| C1—C2—H2A | 120.1 | C11—C12—C13 | 118.80 (8) |
| C2—C3—C4 | 120.73 (8) | C11—C12—H12A | 120.6 |
| C2—C3—H3A | 119.6 | C13—C12—H12A | 120.6 |
| C4—C3—H3A | 119.6 | C12—C13—C8 | 121.90 (7) |
| C5—C4—C3 | 119.58 (8) | C12—C13—N3 | 115.84 (7) |
| C5—C4—H4A | 120.2 | C8—C13—N3 | 122.26 (7) |
| C3—C4—H4A | 120.2 | O1—C14—H14A | 109.5 |
| C4—C5—C6 | 120.96 (8) | O1—C14—H14B | 109.5 |
| C4—C5—H5A | 119.5 | H14A—C14—H14B | 109.5 |
| C6—C5—H5A | 119.5 | O1—C14—H14C | 109.5 |
| C5—C6—C1 | 118.84 (8) | H14A—C14—H14C | 109.5 |
| C5—C6—C7 | 121.21 (7) | H14B—C14—H14C | 109.5 |
| C7—N1—N2—C8 | 178.35 (7) | C13—C8—C9—C10 | -0.77 (12) |
| C14—O1—C1—C2 | 12.61 (13) | C8—C9—C10—C11 | -1.15 (13) |
| C14—O1—C1—C6 | -168.81 (8) | C9—C10—C11—C12 | 1.96 (13) |
| O1—C1—C2—C3 | 177.25 (8) | C9—C10—C11—N4 | -177.18 (8) |
| C6—C1—C2—C3 | -1.27 (13) | O5—N4—C11—C12 | 173.05 (8) |
| C1—C2—C3—C4 | 0.36 (14) | O4—N4—C11—C12 | -7.77 (12) |
| C2—C3—C4—C5 | 0.38 (14) | O5—N4—C11—C10 | -7.78 (12) |
| C3—C4—C5—C6 | -0.21 (14) | O4—N4—C11—C10 | 171.39 (8) |

supplementary materials

| | | | |
|--------------|-------------|-----------------|-------------|
| C4—C5—C6—C1 | -0.69 (13) | C10—C11—C12—C13 | -0.75 (13) |
| C4—C5—C6—C7 | 178.17 (8) | N4—C11—C12—C13 | 178.40 (7) |
| O1—C1—C6—C5 | -177.21 (7) | C11—C12—C13—C8 | -1.28 (13) |
| C2—C1—C6—C5 | 1.43 (13) | C11—C12—C13—N3 | 179.24 (7) |
| O1—C1—C6—C7 | 3.91 (12) | N2—C8—C13—C12 | -178.46 (8) |
| C2—C1—C6—C7 | -177.45 (8) | C9—C8—C13—C12 | 2.01 (12) |
| N2—N1—C7—C6 | -179.67 (7) | N2—C8—C13—N3 | 0.98 (13) |
| C5—C6—C7—N1 | 7.29 (13) | C9—C8—C13—N3 | -178.55 (7) |
| C1—C6—C7—N1 | -173.86 (8) | O3—N3—C13—C12 | -1.30 (12) |
| N1—N2—C8—C9 | -3.93 (12) | O2—N3—C13—C12 | 178.99 (8) |
| N1—N2—C8—C13 | 176.55 (8) | O3—N3—C13—C8 | 179.23 (8) |
| N2—C8—C9—C10 | 179.68 (8) | O2—N3—C13—C8 | -0.48 (12) |

Hydrogen-bond geometry (\AA , $^\circ$)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N2—H1N2 \cdots O2 | 0.864 (15) | 2.029 (15) | 2.6253 (11) | 125.4 (13) |
| N2—H1N2 \cdots O2 ⁱ | 0.864 (15) | 2.599 (15) | 3.3475 (11) | 145.6 (13) |
| C2—H2A \cdots O4 ⁱⁱ | 0.93 | 2.44 | 3.3113 (12) | 155 |
| C5—H5A \cdots O2 ⁱⁱⁱ | 0.93 | 2.60 | 3.3184 (11) | 135 |

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

Fig. 1

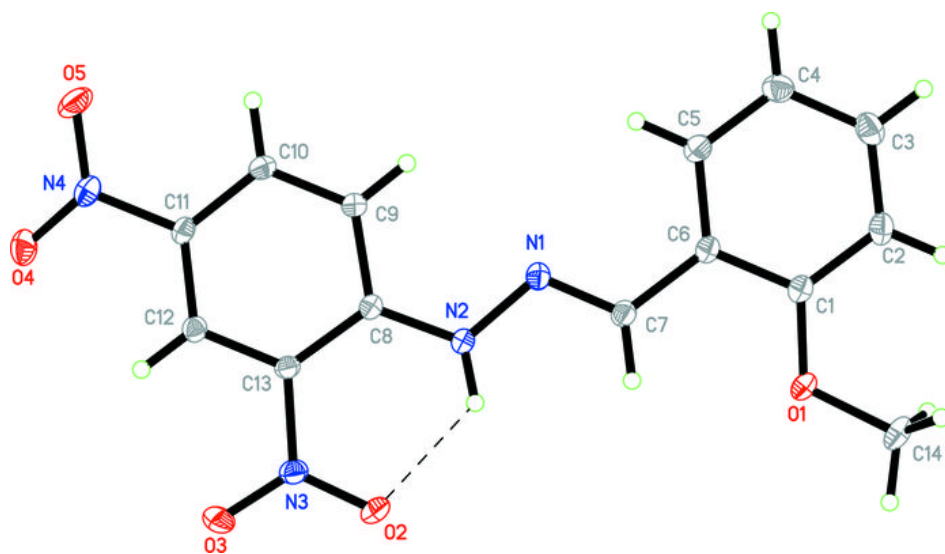


Fig. 2

