



IUCrData

ISSN 2414-3146

## 2-Cyano-*N'*-[(1*E*)-1-(3,4-dimethoxyphenyl)ethylidene]acetohydrazide

Meiyazhagan Manvizhi,<sup>a</sup> Srinivasan Senthilkumar<sup>a\*</sup> and Sivashanmugam Selvanayagam<sup>b‡</sup>

<sup>a</sup>Department of Chemistry, Annamalai University, Annamalaiagar, Chidambaram 608 002, India, and <sup>b</sup>PG & Research Department of Physics, Government Arts College, Melur 625 106, India. \*Correspondence e-mail: senraj05@gmail.com

Received 18 December 2025

Accepted 28 January 2026

Edited by M. Weil, Vienna University of Technology, Austria

‡ Additional correspondence author, e-mail: sselvanayagam@gmail.com.

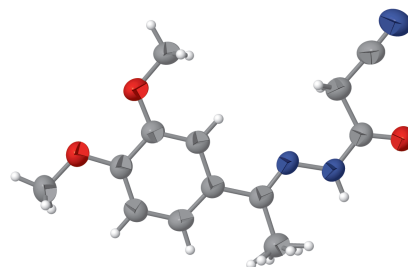
**Keywords:** benzohydrazine; intermolecular hydrogen bonds; Hirshfeld surface analysis; crystal structure.

**CCDC reference:** 2501701

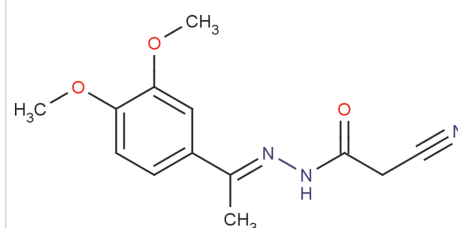
**Structural data:** full structural data are available from iucrdata.iucr.org

The non-H part of the molecule of the title compound, C<sub>13</sub>H<sub>15</sub>N<sub>3</sub>O<sub>3</sub>, is nearly planar, with the 2-cyano-*N'*-[(1*E*)-ethylidene]acetohydrazide moiety and the dimethoxy phenol ring forming a dihedral angle of 2.5 (1)°. Intermolecular N–H···O, C–H···O and C–H···π interactions are mainly responsible for the cohesion within the crystal structure. The intermolecular interactions were quantified and analysed using Hirshfeld surface analysis, revealing that H···H interactions contribute most to the crystal packing (36.9%). The volume of the crystal voids was calculated to be 167.8 Å<sup>3</sup> (13% of the unit-cell volume).

3D view

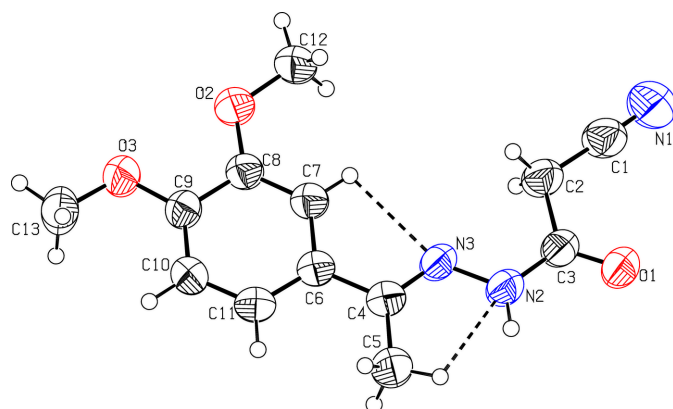


Chemical scheme



### Structure description

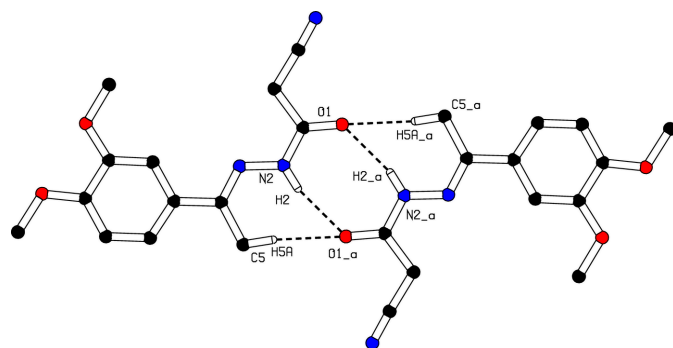
Hydrazone derivatives have long been valued in medicinal and organic chemistry because they are easy to prepare, structurally flexible, and capable of exhibiting a wide range of biological activities. Within this class, acyl hydrazones and heterocycle-linked hydrazones are especially notable, as many of them display antidiabetic, anticancer, antimicrobial, antioxidant, and anti-inflammatory properties (Punitha *et al.*, 2020). In the present work, the title compound, (**I**), was chosen due to the particular combination of functional groups that are known to enhance biological effectiveness. The hydrazone unit (–C=N–NH–) provides an extended-conjugated system that supports strong intermolecular interactions and can promote favourable binding within enzyme active sites. The 3,4-dimethoxyphenyl ring adds lipophilicity, encouraging π–π stacking and improving membrane permeability, which together may enhance pharmacological performance. The cyano (–C≡N) group, being a strong electron-withdrawing substituent, fine-tunes the electronic character of the molecule, increases hydrogen-bond acceptor strength, and is often associated with improved antimicrobial and anticancer effects (Senthilkumar *et al.*, 2021; Maheswari *et al.*, 2025). The coexistence of electron-donating methoxy groups and an electron-withdrawing cyano group creates an internal



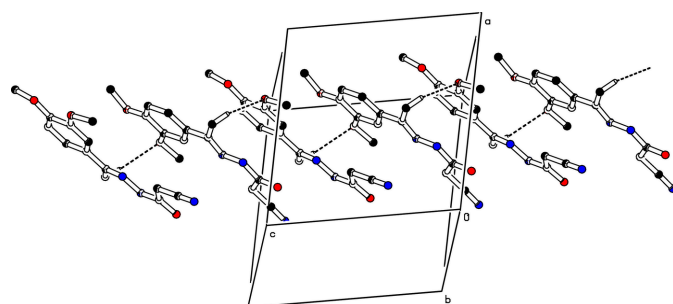
**Figure 1**  
Molecular structure of (**I**) showing the atom-labelling scheme and the intramolecular hydrogen bonds (dashed lines). Displacement ellipsoids are drawn at the 50% probability level. Only one part of the disordered methyl group at C5 is shown.

charge-transfer environment, a feature commonly linked to stronger biological responses in hydrazone frameworks. Because of this combination of structural and electronic attributes, the selected compound offers enhanced biological activity, making it a promising candidate for further pharmacological development (Senthilkumar *et al.*, 2020).

The molecular structure of (**I**) is displayed in Fig. 1. The phenyl ring (C6–C11) is planar with a maximum deviation of 0.010 (3) Å for atom C9, and its attached methoxy atoms O2,



**Figure 2**  
The formation of a centrosymmetric dimer in the crystal structure of (**I**) through N–H···O and C–H···O hydrogen bonds. [Symmetry code: (a)  $-x + 1, -y + 1, -z$ ].



**Figure 3**  
The crystal packing of compound (**I**) viewed along the *b* axis. The C–H···O hydrogen bonds are shown as dashed lines. For clarity, H atoms not involved in hydrogen bonds have been omitted.

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

*C<sub>g</sub>* is the centroid of the benzene ring (C6–C11).

<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>
C5–H5A···N2	0.96	2.38	2.796 (4)	105
C7–H7···N3	0.93	2.44	2.749 (3)	100
N2–H2···O1 <sup>i</sup>	0.86	2.14	2.982 (3)	166
C5–H5A···O1 <sup>i</sup>	0.96	2.33	3.252 (4)	160
C5–H5E···O2 <sup>ii</sup>	0.96	2.59	3.436 (4)	147
C13–H13B··· <i>C<sub>g</sub></i> <sup>iii</sup>	0.97	2.86	3.569 (4)	132

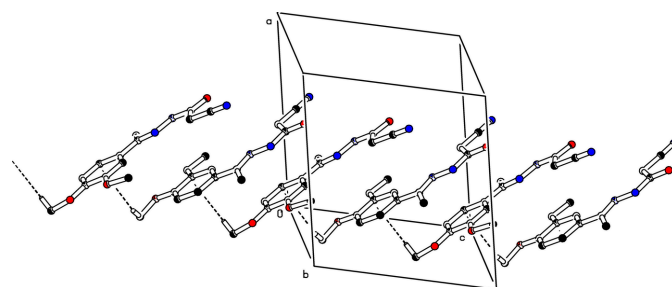
Symmetry codes: (i)  $-x + 1, -y + 1, -z$ ; (ii)  $x, -y + \frac{3}{2}, z - \frac{1}{2}$ ; (iii)  $x, -y + \frac{3}{2}, z + \frac{1}{2}$ .

C12, O3 and C13 deviate by  $-0.015$  (2),  $0.075$  (4),  $0.036$  (2) and  $0.218$  (4) Å, respectively. The 2-cyano-*N'*-(*1E*)-ethylidene]acetohydrazone moiety (N1/C1/C2/C3/O1/N2/N3/C4/C5) is nearly planar with a maximum deviation of 0.120 (4) Å for atom C5. This moiety forms a dihedral angle of  $2.5$  (1)° with the dimethoxy phenyl ring. Weak intramolecular C5–H5A···N2 and C7–H7···N3 contacts, forming two *S*(5) ring motifs (Bernstein *et al.*, 1995) may help to establish the solid-state conformation (Table 1, Fig. 1).

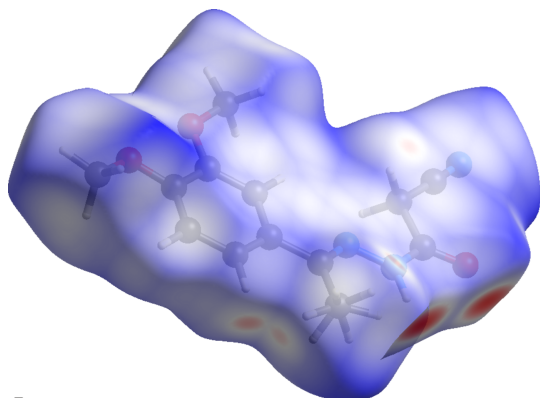
In the crystal, molecules associate pairwise through N2–H2···O1<sup>i</sup> and C5–H5A···O1<sup>i</sup> hydrogen bonds (Table 1) into inversion dimers with  $R_2^2(8)$  and  $R_2^2(14)$  graph-set motifs (Etter *et al.*, 1990; Bernstein *et al.*, 1995), as shown in Fig. 2. The molecules are linked into a *C*(7) chain motif by C5–H5E···O2<sup>ii</sup> hydrogen bonds running parallel to [001] (Table 1, Fig. 3). Moreover, molecules are further linked along the same direction into a *C*(5) chain motif by C–H··· $\pi$  interactions, C13–H13B···*C<sub>g</sub>*, where *C<sub>g</sub>* is the centroid of the symmetry-related C6–C11 benzene ring at  $(x, -y + \frac{3}{2}, z + \frac{1}{2})$  (Table 1, Fig. 4).

In order to further characterize and quantify the intermolecular interactions in the title compound, a Hirshfeld surface (HS) analysis (Spackman & Jayatilaka, 2009) was carried out using *CrystalExplorer* (Spackman *et al.*, 2021). The HS mapped over  $d_{\text{norm}}$  is illustrated in Fig. 5 where the deep-red spots indicative of strong interactions occur at O1, H2 and H5A, and these atoms are responsible for intermolecular N–H···O and C–H···O hydrogen bonds discussed above.

The associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) provide quantitative information about the non-covalent interactions in the crystal packing in



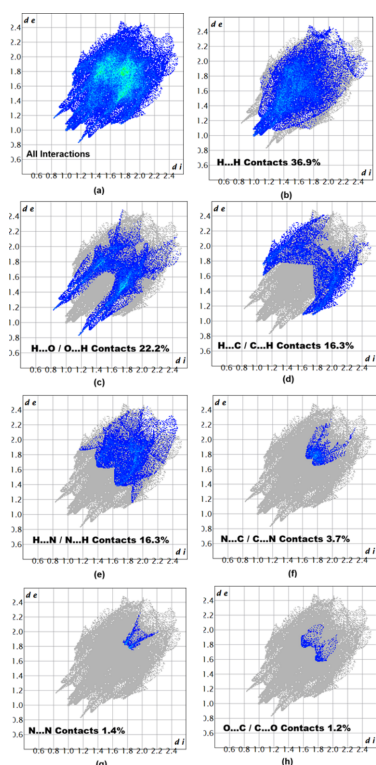
**Figure 4**  
The crystal packing of (**I**). C–H··· $\pi$  interactions are shown as dashed lines. For clarity, H atoms not involved in these interactions have been omitted.



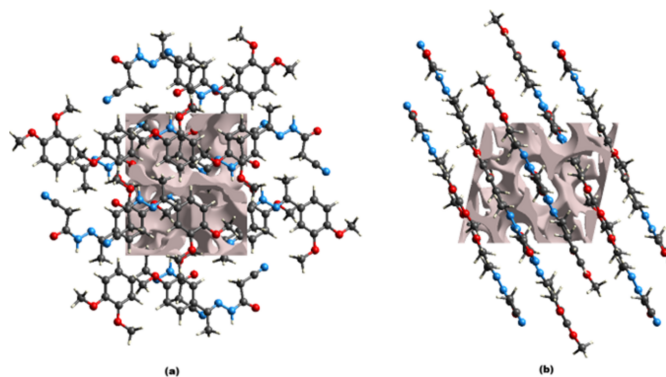
**Figure 5**  
A view of the Hirshfeld surface mapped over  $d_{\text{norm}}$  for compound (**I**).

terms of the percentage contribution of the interatomic contacts (Spackman & McKinnon, 2002). As shown in Fig. 6, the overall two-dimensional fingerprint plot for compound (**I**) is delineated into the different contact types, revealing that  $\text{H}\cdots\text{H}$  (36.9%) and  $\text{H}\cdots\text{O}/\text{O}\cdots\text{H}$  (22.2%) are the main contributors to the crystal packing.

A void analysis was performed by adding up the electron densities of the spherically symmetric atoms contained in the asymmetric unit (Turner *et al.*, 2011). The void surface is defined as an isosurface of the procrystal electron density and is calculated for the whole unit cell where the void surface



**Figure 6**  
Two-dimensional fingerprint plots for compound (**I**), showing (a) all interactions, and delineated into (b)  $\text{H}\cdots\text{H}$ , (c)  $\text{H}\cdots\text{O}/\text{O}\cdots\text{H}$ , (d)  $\text{H}\cdots\text{C}/\text{C}\cdots\text{H}$ , (e)  $\text{H}\cdots\text{N}/\text{N}\cdots\text{H}$ , (f)  $\text{N}\cdots\text{C}/\text{C}\cdots\text{N}$ , (g)  $\text{N}\cdots\text{N}$  and (h)  $\text{O}\cdots\text{C}/\text{C}\cdots\text{O}$  interactions. The  $d_i$  and  $d_e$  values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.



**Figure 7**  
Graphical views of voids in the crystal packing of compound (**I**) viewed down the (a)  $a$ -axis and (b)  $b$ -axis directions.

meets the boundary of the unit cell and capping faces are generated to create an enclosed volume. The volume of the crystal voids (Fig. 7) was calculated to be  $168 \text{ \AA}^3$  (13% of the unit-cell volume). Fig. 7(b) also reveals that individual molecules are arranged in layers parallel to  $(10\bar{1})$ .

### Synthesis and crystallization

Compound (**I**) was prepared through a condensation reaction involving an equimolar ratio of 2-cyanoacetohydrazide (0.05 mol) and 3,4-dimethoxyacetophenone (0.05 mol). The reagents were placed into a clean reaction flask where methanol served as the reaction medium. A few drops of glacial acetic acid were added to promote the formation of the hydrazone bond. The mixture was then heated under reflux for about 6–8 h. Throughout this period, the progress of the reaction was checked at intervals using thin-layer chromatography (TLC) to confirm that the starting materials were being fully consumed. Once the reaction reached completion, the mixture was allowed to cool gradually to room temperature, during which a solid product began to separate out. The resulting precipitate was collected by filtration, thoroughly washed to remove any remaining impurities, and dried under reduced pressure to eliminate traces of solvent. Final purification was achieved by recrystallizing the crude product from warm ethanol solution, affording the desired hydrazone derivative with 75% yield.

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The methyl hydrogen atoms at C5 were refined as equally disordered (using an AFIX 127 instruction with *SHELXL*; Sheldrick, 2015b) with  $\text{C}-\text{H} = 0.98 \text{ \AA}$ .

### Acknowledgements

The authors thank the Single Crystal XRD Facility at VIT, Vellore, Tamil Nadu, India, for providing the instrumentation and support necessary for this study.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2017). *APEX3* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, U. S. A.

Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.

Maheswari, S. U., Senthilkumar, S. & Selvanayagam, S. (2025). *Acta Cryst.* **E81**, 473–475.

McKinnon, J. J., Jayatilaka, D. & Spackman, M. A. (2007). *Chem. Commun.* pp. 3814–3816.

Punitha, P., Senthilkumar, S. & Muthukumaran, G. (2020). *Chem. Data Coll.* **28**, 100373.

Senthilkumar, S., Seralathan, J. & Muthukumaran, G. (2020). *Chem. Data Coll.* **29**, 100514.

Senthilkumar, S., Seralathan, J. & Muthukumaran, G. (2021). *J. Mol. Struct.* **1226**, 129354.

Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.

Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.

Spackman, M. A. & Jayatilaka, D. (2009). *CrystEngComm* **11**, 19–32.

Spackman, M. A. & McKinnon, J. J. (2002). *CrystEngComm* **4**, 378–392.

Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.

Spek, A. L. (2020). *Acta Cryst.* **E76**, 1–11.

Turner, M. J., McKinnon, J. J., Jayatilaka, D. & Spackman, M. A. (2011). *CrystEngComm* **13**, 1804–1813.

Table 2

Experimental details.

Crystal data	
Chemical formula	C <sub>13</sub> H <sub>15</sub> N <sub>3</sub> O <sub>3</sub>
<i>M<sub>r</sub></i>	261.28
Crystal system, space group	Monoclinic, <i>P</i> <sub>2</sub> <sub>1</sub> / <i>c</i>
Temperature (K)	300
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.2308 (13), 11.7792 (13), 10.3668 (12)
$\beta$ (°)	102.692 (4)
<i>V</i> (Å <sup>3</sup> )	1337.9 (3)
<i>Z</i>	4
Radiation type	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.09
Crystal size (mm)	0.46 × 0.12 × 0.06
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.672, 0.746
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	25559, 3317, 1510
<i>R<sub>int</sub></i>	0.064
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.667
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.067, 0.239, 1.04
No. of reflections	3317
No. of parameters	174
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.34, -0.18

Computer programs: *APEX3* and *SAINT* (Bruker, 2017), *SHELXT* (Sheldrick, 2015a), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2020) and *SHELXL* (Sheldrick, 2015b).

## full crystallographic data

*IUCrData* (2026). **11**, x260089 [https://doi.org/10.1107/S2414314626000891]

2-Cyano-*N'*-[(1*E*)-1-(3,4-dimethoxyphenyl)ethylidene]acetohydrazide

Meiyazhagan Manvizhi, Srinivasan Senthilkumar and Sivashanmugam Selvanayagam

2-Cyano-*N'*-[(1*E*)-1-(3,4-dimethoxyphenyl)ethylidene]acetohydrazide*Crystal data*

$C_{13}H_{15}N_3O_3$

$M_r = 261.28$

Monoclinic,  $P2_1/c$

$a = 11.2308$  (13) Å

$b = 11.7792$  (13) Å

$c = 10.3668$  (12) Å

$\beta = 102.692$  (4)°

$V = 1337.9$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 552$

$D_x = 1.297$  Mg m<sup>-3</sup>

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

Cell parameters from 5190 reflections

$\theta = 2.5$ – $24.4$ °

$\mu = 0.09$  mm<sup>-1</sup>

$T = 300$  K

Plate, colourless

$0.46 \times 0.12 \times 0.06$  mm

*Data collection*

Bruker APEXII CCD

diffractometer

Radiation source: i-mu-s microfocus source

$\phi$  and  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.672$ ,  $T_{\max} = 0.746$

25559 measured reflections

3317 independent reflections

1510 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.064$

$\theta_{\max} = 28.3$ °,  $\theta_{\min} = 1.9$ °

$h = -14 \rightarrow 14$

$k = -15 \rightarrow 15$

$l = -13 \rightarrow 13$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.239$

$S = 1.04$

3317 reflections

174 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1011P)^2 + 0.4583P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.34$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.18$  e Å<sup>-3</sup>

Extinction correction: SHELXL (Sheldrick, 2015b),  $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.008 (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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.4532 (2)	0.63384 (18)	-0.0687 (2)	0.0873 (8)	
O2	0.87019 (19)	0.96378 (15)	0.55091 (18)	0.0716 (6)	
O3	1.00787 (18)	0.85504 (16)	0.74705 (17)	0.0680 (6)	
N1	0.3940 (3)	0.9169 (3)	-0.1399 (4)	0.1051 (11)	
N2	0.5860 (2)	0.61306 (19)	0.1263 (2)	0.0626 (7)	
H2	0.585827	0.540243	0.120280	0.075*	
N3	0.65690 (19)	0.66651 (18)	0.2344 (2)	0.0590 (6)	
C1	0.4510 (3)	0.8648 (3)	-0.0567 (3)	0.0750 (9)	
C2	0.5239 (3)	0.8034 (2)	0.0532 (3)	0.0680 (8)	
H2A	0.495556	0.820980	0.132830	0.082*	
H2B	0.608161	0.827932	0.066527	0.082*	
C3	0.5174 (2)	0.6761 (2)	0.0306 (3)	0.0629 (7)	
C4	0.7271 (2)	0.6060 (2)	0.3220 (3)	0.0554 (7)	
C5	0.7404 (3)	0.4793 (2)	0.3148 (3)	0.0791 (9)	
H5A	0.696800	0.452706	0.229983	0.119*	0.5
H5B	0.707704	0.444113	0.383042	0.119*	0.5
H5C	0.825218	0.460173	0.326694	0.119*	0.5
H5D	0.789682	0.451955	0.396497	0.119*	0.5
H5E	0.778777	0.460548	0.243437	0.119*	0.5
H5F	0.661263	0.444489	0.299785	0.119*	0.5
C6	0.8006 (2)	0.6688 (2)	0.4357 (2)	0.0544 (7)	
C7	0.7997 (2)	0.7877 (2)	0.4374 (3)	0.0557 (7)	
H7	0.752355	0.826757	0.366329	0.067*	
C8	0.8673 (2)	0.8482 (2)	0.5422 (2)	0.0544 (7)	
C9	0.9413 (2)	0.7895 (2)	0.6492 (2)	0.0548 (7)	
C10	0.9413 (3)	0.6734 (2)	0.6487 (3)	0.0627 (7)	
H10	0.987959	0.634148	0.720009	0.075*	
C11	0.8724 (3)	0.6137 (2)	0.5426 (3)	0.0646 (8)	
H11	0.874624	0.534744	0.543617	0.078*	
C12	0.8042 (3)	1.0254 (2)	0.4392 (3)	0.0795 (9)	
H12A	0.812865	1.105355	0.456610	0.119*	
H12B	0.836055	1.007186	0.363009	0.119*	
H12C	0.719483	1.005024	0.422915	0.119*	
C13	1.0939 (3)	0.7968 (3)	0.8472 (3)	0.0808 (10)	
H13A	1.135675	0.850615	0.910895	0.121*	
H13B	1.051878	0.742740	0.890291	0.121*	
H13C	1.152076	0.758002	0.807590	0.121*	

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0962 (16)	0.0556 (12)	0.0888 (15)	-0.0086 (11)	-0.0261 (13)	-0.0013 (11)
O2	0.0960 (14)	0.0461 (11)	0.0650 (12)	0.0002 (10)	0.0014 (10)	-0.0039 (9)
O3	0.0821 (13)	0.0586 (11)	0.0568 (11)	-0.0016 (10)	0.0011 (10)	-0.0062 (9)
N1	0.102 (2)	0.081 (2)	0.118 (3)	0.0087 (18)	-0.006 (2)	0.0182 (19)

N2	0.0645 (14)	0.0452 (12)	0.0703 (15)	-0.0035 (10)	-0.0021 (12)	-0.0056 (11)
N3	0.0589 (13)	0.0472 (12)	0.0648 (14)	-0.0062 (10)	0.0006 (11)	-0.0067 (11)
C1	0.0699 (19)	0.0540 (17)	0.095 (2)	0.0030 (15)	0.0049 (18)	0.0030 (17)
C2	0.0673 (17)	0.0479 (15)	0.082 (2)	-0.0003 (13)	0.0028 (15)	-0.0026 (14)
C3	0.0587 (16)	0.0524 (15)	0.0709 (18)	-0.0058 (13)	-0.0004 (14)	-0.0005 (14)
C4	0.0586 (15)	0.0477 (14)	0.0603 (16)	-0.0027 (12)	0.0136 (13)	-0.0004 (12)
C5	0.100 (2)	0.0519 (17)	0.075 (2)	-0.0004 (16)	-0.0025 (17)	-0.0036 (15)
C6	0.0588 (15)	0.0478 (14)	0.0558 (15)	-0.0019 (12)	0.0108 (12)	0.0000 (12)
C7	0.0621 (16)	0.0491 (14)	0.0553 (15)	-0.0007 (12)	0.0113 (13)	0.0010 (12)
C8	0.0614 (15)	0.0480 (14)	0.0534 (15)	-0.0002 (12)	0.0115 (12)	-0.0005 (12)
C9	0.0592 (15)	0.0549 (15)	0.0494 (14)	-0.0010 (12)	0.0103 (12)	-0.0049 (12)
C10	0.0705 (17)	0.0559 (16)	0.0566 (16)	0.0007 (13)	0.0032 (13)	0.0063 (13)
C11	0.0753 (18)	0.0454 (14)	0.0697 (18)	-0.0022 (13)	0.0083 (15)	0.0039 (13)
C12	0.106 (2)	0.0497 (16)	0.073 (2)	-0.0010 (16)	0.0002 (17)	0.0056 (15)
C13	0.085 (2)	0.076 (2)	0.0684 (19)	0.0057 (17)	-0.0101 (16)	-0.0089 (16)

*Geometric parameters (Å, °)*

O1—C3	1.225 (3)	C5—H5D	0.9600
O2—C8	1.364 (3)	C5—H5E	0.9600
O2—C12	1.428 (3)	C5—H5F	0.9600
O3—C9	1.360 (3)	C6—C11	1.382 (4)
O3—C13	1.429 (3)	C6—C7	1.401 (4)
N1—C1	1.135 (4)	C7—C8	1.379 (3)
N2—C3	1.339 (3)	C7—H7	0.9300
N2—N3	1.376 (3)	C8—C9	1.412 (3)
N2—H2	0.8600	C9—C10	1.367 (4)
N3—C4	1.280 (3)	C10—C11	1.390 (4)
C1—C2	1.443 (4)	C10—H10	0.9300
C2—C3	1.517 (4)	C11—H11	0.9300
C2—H2A	0.9700	C12—H12A	0.9600
C2—H2B	0.9700	C12—H12B	0.9600
C4—C6	1.480 (3)	C12—H12C	0.9600
C4—C5	1.504 (4)	C13—H13A	0.9600
C5—H5A	0.9600	C13—H13B	0.9600
C5—H5B	0.9600	C13—H13C	0.9600
C5—H5C	0.9600		
C8—O2—C12	116.9 (2)	H5A—C5—H5F	56.3
C9—O3—C13	116.3 (2)	H5B—C5—H5F	56.3
C3—N2—N3	119.0 (2)	H5C—C5—H5F	141.1
C3—N2—H2	120.5	H5D—C5—H5F	109.5
N3—N2—H2	120.5	H5E—C5—H5F	109.5
C4—N3—N2	118.6 (2)	C11—C6—C7	117.7 (2)
N1—C1—C2	177.2 (4)	C11—C6—C4	121.9 (2)
C1—C2—C3	112.0 (2)	C7—C6—C4	120.4 (2)
C1—C2—H2A	109.2	C8—C7—C6	121.5 (2)
C3—C2—H2A	109.2	C8—C7—H7	119.2

C1—C2—H2B	109.2	C6—C7—H7	119.2
C3—C2—H2B	109.2	O2—C8—C7	124.7 (2)
H2A—C2—H2B	107.9	O2—C8—C9	115.8 (2)
O1—C3—N2	122.3 (3)	C7—C8—C9	119.5 (2)
O1—C3—C2	122.0 (2)	O3—C9—C10	124.7 (2)
N2—C3—C2	115.7 (2)	O3—C9—C8	116.1 (2)
N3—C4—C6	115.8 (2)	C10—C9—C8	119.2 (2)
N3—C4—C5	124.7 (2)	C9—C10—C11	120.6 (2)
C6—C4—C5	119.5 (2)	C9—C10—H10	119.7
C4—C5—H5A	109.5	C11—C10—H10	119.7
C4—C5—H5B	109.5	C6—C11—C10	121.5 (2)
H5A—C5—H5B	109.5	C6—C11—H11	119.3
C4—C5—H5C	109.5	C10—C11—H11	119.3
H5A—C5—H5C	109.5	O2—C12—H12A	109.5
H5B—C5—H5C	109.5	O2—C12—H12B	109.5
C4—C5—H5D	109.5	H12A—C12—H12B	109.5
H5A—C5—H5D	141.1	O2—C12—H12C	109.5
H5B—C5—H5D	56.3	H12A—C12—H12C	109.5
H5C—C5—H5D	56.3	H12B—C12—H12C	109.5
C4—C5—H5E	109.5	O3—C13—H13A	109.5
H5A—C5—H5E	56.3	O3—C13—H13B	109.5
H5B—C5—H5E	141.1	H13A—C13—H13B	109.5
H5C—C5—H5E	56.3	O3—C13—H13C	109.5
H5D—C5—H5E	109.5	H13A—C13—H13C	109.5
C4—C5—H5F	109.5	H13B—C13—H13C	109.5
C3—N2—N3—C4	-176.4 (2)	C12—O2—C8—C9	174.9 (2)
N3—N2—C3—O1	179.3 (3)	C6—C7—C8—O2	-179.6 (3)
N3—N2—C3—C2	-0.9 (4)	C6—C7—C8—C9	1.3 (4)
C1—C2—C3—O1	-2.4 (4)	C13—O3—C9—C10	7.8 (4)
C1—C2—C3—N2	177.8 (2)	C13—O3—C9—C8	-172.6 (2)
N2—N3—C4—C6	-179.9 (2)	O2—C8—C9—O3	-0.9 (3)
N2—N3—C4—C5	1.3 (4)	C7—C8—C9—O3	178.2 (2)
N3—C4—C6—C11	174.5 (3)	O2—C8—C9—C10	178.7 (2)
C5—C4—C6—C11	-6.7 (4)	C7—C8—C9—C10	-2.1 (4)
N3—C4—C6—C7	-5.8 (4)	O3—C9—C10—C11	-178.5 (2)
C5—C4—C6—C7	173.0 (3)	C8—C9—C10—C11	1.9 (4)
C11—C6—C7—C8	-0.4 (4)	C7—C6—C11—C10	0.1 (4)
C4—C6—C7—C8	180.0 (2)	C4—C6—C11—C10	179.8 (2)
C12—O2—C8—C7	-4.2 (4)	C9—C10—C11—C6	-0.9 (4)

*Hydrogen-bond geometry (Å, °)*C<sub>g</sub> is the centroid of the benzene ring (C6–C11).

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C5—H5A...N2	0.96	2.38	2.796 (4)	105
C7—H7...N3	0.93	2.44	2.749 (3)	100
N2—H2...O1 <sup>i</sup>	0.86	2.14	2.982 (3)	166

---

C5—H5A···O1 <sup>i</sup>	0.96	2.33	3.252 (4)	160
C5—H5E···O2 <sup>ii</sup>	0.96	2.59	3.436 (4)	147
C13—H13B···Cg <sup>iii</sup>	0.97	2.86	3.569 (4)	132

---

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