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Crystal structure of 2,3-dimethoxy-*N*-(4-nitrophenyl)benzamide

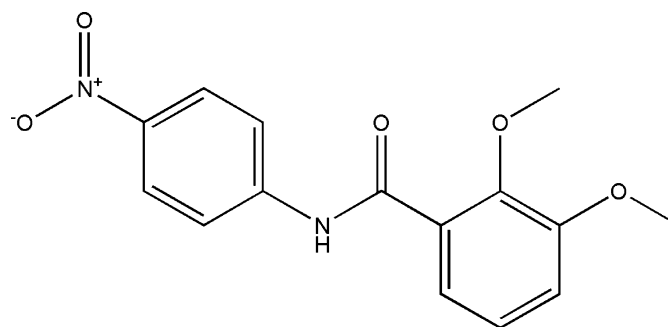
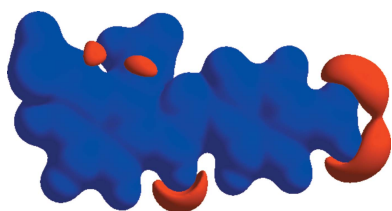
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In the title compound, C₁₅H₁₄N₂O₅, the benzene rings are nearly coplanar, making a dihedral angle of 4.89 (8)°. An intramolecular N—H···O hydrogen bond occurs between the imino and methoxy groups. In the crystal, weak C—H···O hydrogen bonds link the molecules into supramolecular chains propagating along the *a*-axis direction. π – π stacking is observed between parallel benzene rings of neighbouring chains, the centroid-to-centroid distance being 3.6491 (10) Å. Three-dimensional Hirshfeld surface analyses and two-dimensional fingerprint plots have been used to analyse the intermolecular interactions present in the crystal.

1. Chemical context

Amides have a very important place in both organic and biological chemistry. They are used as building blocks for natural products such as proteins and peptides. However, amides are not restricted to biological systems, but also have a wide range of uses in pharmaceutical chemistry (Khalafi-Nezhad *et al.*, 2005; Valeur & Bradley, 2009). Many amide derivatives have been found to possess antitumor, antimicrobial, anti-HIV, anti-inflammatory, anticonvulsant, antibacterial, antifungal, analgesic and anticancer properties (Kushwaha *et al.*, 2011; Fu *et al.*, 2010; Carbone *et al.*, 2005; Siddiqui *et al.*, 2008). Benzamides and their derivatives are compounds of biological and pharmaceutical importance. A variety of benzamide derivatives have been synthesized by the interaction of aniline derivatives that carry electron-donating groups (anisidines, toluidines) and acyl chlorides (2,3-dimethoxybenzoyl chloride and 3-acetoxy-2-methylbenzoyl chloride) in a slightly basic medium (Çakmak *et al.*, 2016; Demir *et al.*, 2015).



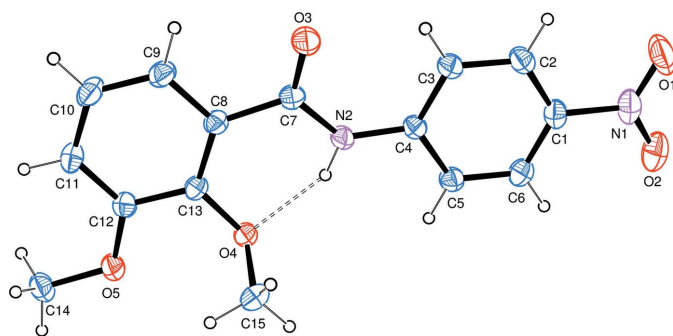


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. The intramolecular N—H \cdots O (Table 1) hydrogen bond is shown as a double dashed line.

2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The bond distances and angles are found to be in good agreement with those in analogous structures (Demir *et al.*, 2015; Tahir *et al.*, 2011). In the molecule, the benzene rings are nearly coplanar, with a dihedral angle of 4.89 (8)°. An intramolecular N—H \cdots O hydrogen bond (Table 1) occurs between the imino and methoxy groups.

3. Supramolecular features

In the crystal, adjacent molecules are linked by weak C—H \cdots O hydrogen bonds, forming supramolecular chains propagating along the *a*-axis direction (Table 1, Fig. 2). π – π stacking is observed between parallel benzene rings of adjacent chains, the centroid-to-centroid distance being 3.6491 (10) Å.

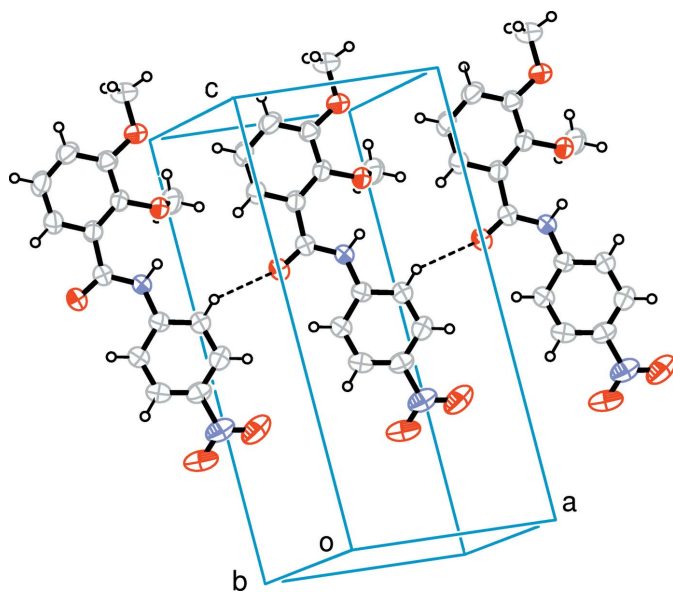


Figure 2
Packing of the title compound in the unit cell. Dashed lines indicate the C—H \cdots O hydrogen bonds (see Table 1).

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

<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—H2 \cdots O4	0.87 (2)	1.924 (19)	2.6805 (16)	144.6 (17)
C5—H5 \cdots O3 ⁱ	0.93	2.48	3.2597 (19)	141

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

4. Hirshfeld surface analysis

Three-dimensional Hirshfeld surfaces (HS) were generated using *Crystal Explorer 3.1* (Wolff *et al.*, 2013) based on the results of the single crystal X-ray diffraction studies. Two-dimensional fingerprint plots (FPs) provide a visual representation of crystal-packing interactions in the structure. The HS is a useful tool for describing the surface characteristics and gaining additional insight into the intermolecular interactions of the molecules.

The molecular Hirshfeld surface, d_{norm} , is depicted in Fig. 3 and mapped over the range -0.1763 to 1.2643 Å. Strong hydrogen-bond interactions, such as C—H \cdots O, are seen as a bright-red area on the Hirshfeld surfaces (Şen *et al.*, 2017). The fingerprint plots over the Hirshfeld surfaces illustrate the significant differences between the intermolecular interaction patterns. In Fig. 4, it is observed $N_{\text{inside}}\cdots H_{\text{outside}} = 2.3\%$, $C_{\text{inside}}\cdots H_{\text{outside}} = 15.7\%$, $O_{\text{inside}}\cdots H_{\text{outside}} = 29.7\%$, $H_{\text{inside}}\cdots H_{\text{outside}} = 38\%$ and all atoms_{inside} \cdots all atoms_{outside} = 100% of the total interactions. Fig. 4 shows that the major contributions are from H \cdots H (38%) and O \cdots H (30%) interactions. Fig. 5 illustrates the distribution of positive and negative potential over the Hirshfeld surfaces. Blue regions correspond to positive electrostatic potential (indicating hydrogen-bond donors) and the red regions to negative electrostatic potential (indicating hydrogen-bond acceptors) (Kumar *et al.*, 2013).

5. IR spectroscopic analyses

The FT-IR spectrum of 2,3-dimethoxy-*N*-(4-nitrophenyl)benzamide, shown in Fig. 6, has several characterization bands. The first characteristic absorption band is at 3311 cm^{-1} and

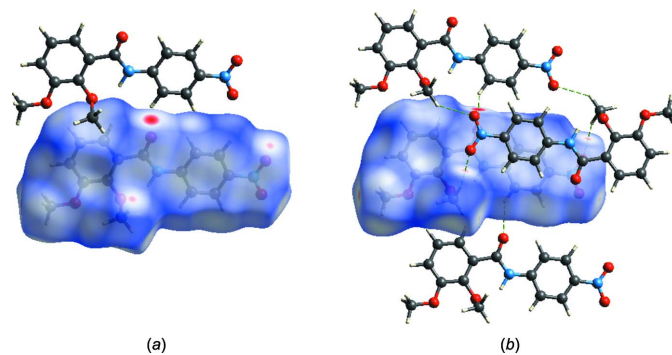


Figure 3
Hirshfeld d_{norm} (a) for 2,3-dimethoxy-*N*-(4-nitrophenyl)benzamide and (b) showing the hydrogen bonding.

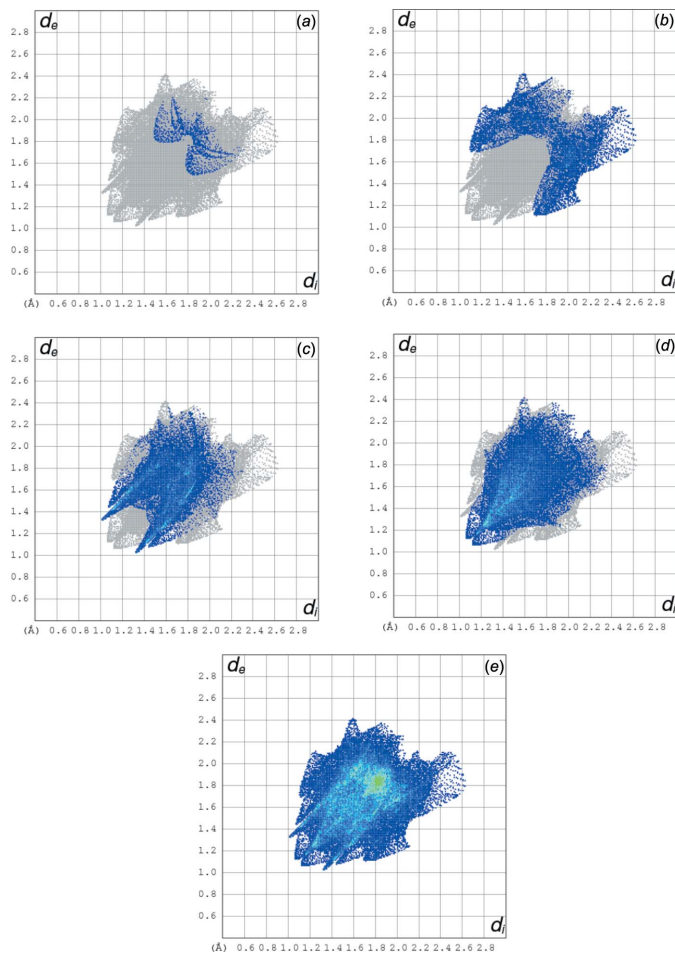


Figure 4
Hirshfeld surface fingerprint of the title compound, (a) $N_{\text{inside}} \cdots H_{\text{outside}}$ (2.3%), (b) $C_{\text{inside}} \cdots H_{\text{outside}}$ (15.7%), (c) $O_{\text{inside}} \cdots H_{\text{outside}}$ (29.7%), (d) $H_{\text{inside}} \cdots H_{\text{outside}}$ (38%), (e) all atoms_{inside} \cdots all atoms_{outside} (100% of total interactions).

was assigned to the N–H stretching vibration. The second remarkable very strong vibrational band is located at 1689 cm^{-1} and can be attributed to the C=O stretching vibration. Another group wavenumber is the C–N stretching vibration that appears at 862 cm^{-1} . This vibration frequency belongs to the nitro group attached to the phenyl ring at the

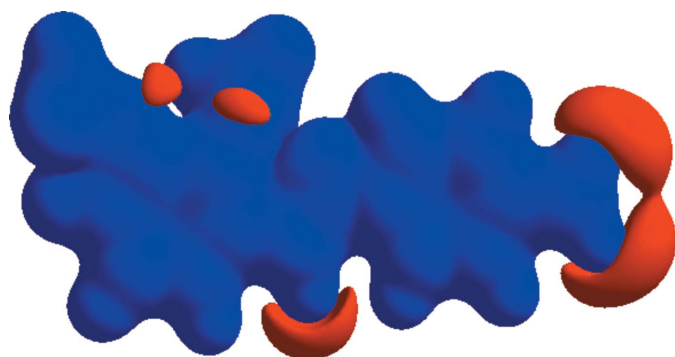


Figure 5
Electrostatic potential mapped on the Hirshfeld surface with $\pm 0.25 \text{ au}$

4-position. The asymmetrical and symmetrical stretching vibrations of the nitro group are observed at 1549 and 1327 cm^{-1} , respectively. In the IR spectrum, peaks corresponding to $\text{C}=\text{O}$ stretching and $\text{N}-\text{H}$ stretching indicate the presence of an amide linkage. These values are in agreement with those previously reported for similar compounds (Cakmak *et al.*, 2016; Demir *et al.*, 2015).

6. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.38, last update May 2017; Groom *et al.*, 2016) for the 2,3-dimethyl-*N*-(phenyl)benzamide skeleton gave 17 hits. One of these compounds, *viz.* 2,3-dimethoxy-*N*-(4-methylphenyl)benzamide, also named as 2,3-dimethoxy-*N*-(*p*-tolyl)benzamide (UYALEN; Cakmak *et al.*, 2016) is similar to the title compound. However, here the two aryl rings are inclined to one another by *ca* 34.16° , despite the presence of an intramolecular $\text{N}-\text{H} \cdots \text{O}_{\text{methoxy}}$ hydrogen bond. A search for the 4-nitrophenylbenzamide skeleton gave 16 hits. They include 4-nitrophenylbenzamide itself, also called benz-*p*-nitroanilide (BUTDID; Du Plessis *et al.*, 1983) and two polymorphs (orthorhombic and monoclinic) of 4'-nitrosalicylanilide (respectively, KADZEU and KADZIY; Etter *et al.*, 1988). Here, the aryl rings are inclined to one another by *ca* 62.30° in BUTDID, 11.24 (10°) in KADZEU, and 3.02 (12°) and 2.69 (12°) in the two independent molecules of the monoclinic polymorph of 4'-nitrosalicylanilide, *i.e.* KADZIY. The same dihedral angle in the title compound is 4.89 (9°). Only in BUTDID, with a dihedral angle of *ca* 62.30° , is there no intramolecular $\text{N}-\text{H} \cdots \text{O}$ hydrogen present.

7. Synthesis and crystallization

To a solution of 4-nitroaniline (10 mmol) and triethylamine (10 mmol) in THF (10 ml) was added dropwise a THF (10 ml) solution of 2,3-dimethoxybenzoyl chloride (11 mmol) at room temperature. The reaction mixture was stirred at room temperature for 15 h and then the resulting white salt precipitate was filtered off and then 150 ml water was added dropwise to the filtrate. The precipitate was filtered off and washed several times with water to remove excessive aniline

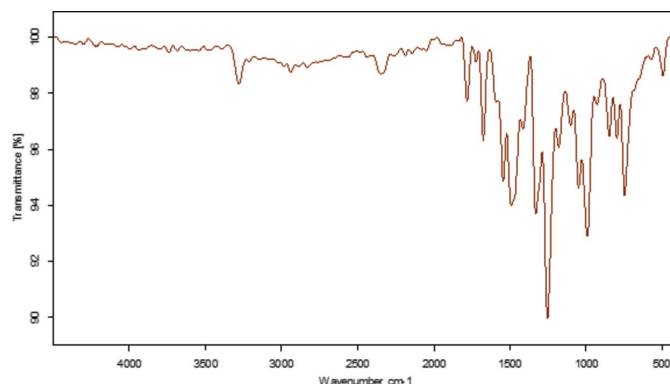


Figure 6
The FT-IR spectrum of the title compound.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₅ H ₁₄ N ₂ O ₅
<i>M_r</i>	302.28
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.9293 (5), 7.3270 (5), 15.7411 (11)
α , β , γ (°)	94.198 (6), 96.189 (6), 116.053 (5)
<i>V</i> (Å ³)	707.27 (9)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.11
Crystal size (mm)	0.74 × 0.49 × 0.28
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (<i>X-RED32</i> ; Stoe & Cie, 2002)
<i>T_{min}</i> , <i>T_{max}</i>	0.947, 0.972
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	10204, 2776, 2011
<i>R_{int}</i>	0.109
(sin θ / λ) _{max} (Å ⁻¹)	0.617
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.039, 0.114, 1.09
No. of reflections	2776
No. of parameters	203
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.16, -0.15

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2002), *SHELXT* (Sheldrick, 2015a), *SHELXL2017* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012), *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

derivative and trimethylamine hydrochloride salt. The crude product was crystallized from acetonitrile (yield 2.09 g 63%; m.p. 448–451 K; Demir *et al.*, 2015; Cakmak *et al.*, 2016).

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The imino-H atom was located in a difference-Fourier map. All C-bound H atoms were positioned

geometrically and refined using a riding model with C–H = 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$.

Acknowledgements

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References

- Cakmak, S., Kutuk, H., Odabasoglu, M., Yakan, H. & Buyukgungor, O. (2016). *Lett. Org. Chem.* **13**, 181–194.
- Carbannelle, D., Ebstein, F., Rabu, C., Petit, J. Y., Gregoire, M. & Lang, F. (2005). *Eur. J. Immunol.* **35**, 546–556.
- Demir, S., Cakmak, S., Dege, N., Kutuk, H., Odabasoglu, M. & Kepekci, R. A. (2015). *J. Mol. Struct.* **1100**, 582–591.
- Du Plessis, M. P., Modro, T. A. & Nassimbeni, L. R. (1983). *J. Crystallogr. Spectrosc. Res.* **13**, 179–189.
- Etter, M. C., Urbańczyk-Lipkowska, Z., Ameli, T. M. & Panunto, T. W. (1988). *J. Crystallogr. Spectrosc. Res.* **18**, 491–507.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Fu, J., Cheng, K., Zhang, Z.-M., Fang, R.-Q. & Zhu, H.-L. (2010). *Eur. J. Med. Chem.* **45**, 2638–2643.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Khalafi-Nezhad, A., Parhami, A., Soltani Rad, M. N. & Zarea, A. (2005). *Tetrahedron Lett.* **46**, 6879–6882.
- Kumar, S. M., Manjunath, B., Lingaraju, G., Abdoh, M., Sadashiva, M. & Lokanath, N. (2013). *Cryst. Struct. Theory Appl.* **02**, 124–131.
- Kushwaha, N., Saini, R. K. & Kushwaha, S. K. (2011). *Int. J. Chem. Tech. Res. (USA)*, **3**, 203–209.
- Şen, F., Kansiz, S. & Uçar, İ. (2017). *Acta Cryst.* **C73**, 517–524.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Siddiqui, N., Alam, M. S. & Ahsan, W. (2008). *Acta Pharm.* **58**, 445–454.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Stoe & Cie (2002). *Stoe & Cie, Darmstadt, Germany*.
- Valeur, E. & Bradley, M. (2009). *Chem. Soc. Rev.* **38**, 606–631.
- Wolff, S., Grimwood, D., McKinnon, J., Turner, M., Jayatilaka, D. & Spackman, M. (2013). *Crystal Explorer*. University of Western Australia, Perth, Australia.

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Crystal structure of 2,3-dimethoxy-*N*-(4-nitrophenyl)benzamide

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Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2017* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

2,3-Dimethoxy-*N*-(4-nitrophenyl)benzamide

Crystal data

$C_{15}H_{14}N_2O_5$
 $M_r = 302.28$
 Triclinic, $P\bar{1}$
 $a = 6.9293$ (5) Å
 $b = 7.3270$ (5) Å
 $c = 15.7411$ (11) Å
 $\alpha = 94.198$ (6)°
 $\beta = 96.189$ (6)°
 $\gamma = 116.053$ (5)°
 $V = 707.27$ (9) Å³

$Z = 2$
 $F(000) = 316$
 $D_x = 1.419$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 12957 reflections
 $\theta = 2.6$ – 27.5 °
 $\mu = 0.11$ mm⁻¹
 $T = 296$ K
 Prism, colorless
 $0.74 \times 0.49 \times 0.28$ mm

Data collection

Stoe IPDS 2
 diffractometer
 Radiation source: sealed X-ray tube, 12 x 0.4
 mm long-fine focus
 Plane graphite monochromator
 Detector resolution: 6.67 pixels mm⁻¹
 rotation method scans
 Absorption correction: integration
 (X-RED32; Stoe & Cie, 2002)

$T_{\min} = 0.947$, $T_{\max} = 0.972$
 10204 measured reflections
 2776 independent reflections
 2011 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.109$
 $\theta_{\max} = 26.0$ °, $\theta_{\min} = 2.6$ °
 $h = -8 \rightarrow 8$
 $k = -9 \rightarrow 9$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.114$
 $S = 1.09$
 2776 reflections
 203 parameters
 0 restraints

Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 0.0364P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.16$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O4	0.64070 (15)	0.29058 (17)	0.80665 (6)	0.0545 (3)
O5	0.60933 (17)	0.26544 (19)	0.97197 (7)	0.0616 (3)
N2	0.4382 (2)	0.2366 (2)	0.64515 (8)	0.0511 (3)
O3	0.10391 (19)	0.2209 (2)	0.63299 (8)	0.0808 (4)
C13	0.4424 (2)	0.2492 (2)	0.83248 (9)	0.0471 (3)
C4	0.4797 (2)	0.2459 (2)	0.56018 (9)	0.0459 (3)
C8	0.2622 (2)	0.2150 (2)	0.77272 (10)	0.0488 (4)
O2	0.8200 (3)	0.2760 (3)	0.30063 (10)	0.1064 (5)
N1	0.6417 (3)	0.2623 (2)	0.30988 (11)	0.0768 (5)
C7	0.2601 (2)	0.2247 (2)	0.67740 (10)	0.0517 (4)
C12	0.4247 (2)	0.2346 (2)	0.91990 (10)	0.0507 (4)
C1	0.5861 (3)	0.2588 (2)	0.39713 (10)	0.0570 (4)
C3	0.3267 (3)	0.2228 (3)	0.49000 (10)	0.0549 (4)
H3	0.188086	0.202409	0.498155	0.066*
O1	0.5080 (3)	0.2514 (3)	0.25036 (9)	0.1153 (6)
C5	0.6860 (2)	0.2752 (2)	0.54688 (10)	0.0540 (4)
H5	0.788868	0.291175	0.593672	0.065*
C9	0.0660 (2)	0.1701 (3)	0.80232 (11)	0.0598 (4)
H9	-0.055930	0.146485	0.763333	0.072*
C6	0.7391 (3)	0.2808 (3)	0.46522 (11)	0.0597 (4)
H6	0.876591	0.299130	0.456262	0.072*
C11	0.2278 (3)	0.1919 (3)	0.94704 (10)	0.0610 (4)
H11	0.215193	0.184597	1.005063	0.073*
C2	0.3818 (3)	0.2305 (3)	0.40828 (10)	0.0594 (4)
H2B	0.281017	0.216468	0.361048	0.071*
C10	0.0500 (3)	0.1603 (3)	0.88803 (12)	0.0667 (5)
H10	-0.081928	0.131915	0.906628	0.080*
C14	0.6008 (3)	0.2573 (3)	1.06211 (10)	0.0672 (5)
H14A	0.740231	0.281308	1.091532	0.101*
H14B	0.563274	0.360514	1.084938	0.101*
H14C	0.493006	0.124535	1.070342	0.101*
C15	0.8027 (3)	0.5006 (3)	0.83037 (13)	0.0752 (5)
H15A	0.935274	0.518241	0.810544	0.113*
H15B	0.751827	0.589244	0.804407	0.113*
H15C	0.828921	0.534368	0.891963	0.113*
H2	0.540 (3)	0.244 (3)	0.6846 (12)	0.072 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0428 (5)	0.0755 (8)	0.0474 (6)	0.0287 (5)	0.0087 (4)	0.0056 (5)
O5	0.0594 (6)	0.0816 (8)	0.0443 (6)	0.0319 (6)	0.0082 (5)	0.0108 (5)
N2	0.0453 (7)	0.0696 (9)	0.0424 (7)	0.0301 (6)	0.0038 (5)	0.0075 (6)
O3	0.0560 (7)	0.1396 (13)	0.0604 (7)	0.0554 (8)	0.0078 (5)	0.0211 (7)
C13	0.0438 (7)	0.0507 (9)	0.0508 (8)	0.0236 (6)	0.0127 (6)	0.0076 (6)
C4	0.0485 (8)	0.0452 (8)	0.0443 (7)	0.0217 (6)	0.0051 (6)	0.0057 (6)
C8	0.0440 (7)	0.0521 (9)	0.0522 (8)	0.0228 (6)	0.0102 (6)	0.0078 (7)
O2	0.1207 (13)	0.1259 (14)	0.0872 (11)	0.0572 (11)	0.0587 (10)	0.0277 (9)
N1	0.1039 (13)	0.0702 (10)	0.0590 (9)	0.0362 (9)	0.0319 (9)	0.0164 (7)
C7	0.0443 (8)	0.0595 (10)	0.0522 (8)	0.0245 (7)	0.0057 (6)	0.0076 (7)
C12	0.0531 (8)	0.0515 (9)	0.0494 (8)	0.0245 (7)	0.0104 (6)	0.0094 (7)
C1	0.0736 (10)	0.0493 (9)	0.0486 (8)	0.0258 (8)	0.0177 (7)	0.0102 (7)
C3	0.0518 (8)	0.0631 (10)	0.0505 (9)	0.0272 (7)	0.0043 (6)	0.0090 (7)
O1	0.1461 (15)	0.1591 (17)	0.0487 (8)	0.0731 (13)	0.0207 (9)	0.0256 (9)
C5	0.0496 (8)	0.0629 (10)	0.0515 (8)	0.0278 (7)	0.0061 (6)	0.0063 (7)
C9	0.0451 (8)	0.0729 (11)	0.0647 (10)	0.0278 (8)	0.0127 (7)	0.0157 (8)
C6	0.0586 (9)	0.0623 (11)	0.0634 (10)	0.0293 (8)	0.0201 (8)	0.0107 (8)
C11	0.0670 (10)	0.0690 (11)	0.0549 (9)	0.0325 (8)	0.0256 (8)	0.0196 (8)
C2	0.0682 (10)	0.0594 (10)	0.0472 (8)	0.0273 (8)	0.0010 (7)	0.0092 (7)
C10	0.0523 (9)	0.0820 (13)	0.0743 (11)	0.0318 (9)	0.0277 (8)	0.0247 (9)
C14	0.0816 (12)	0.0716 (12)	0.0448 (9)	0.0317 (9)	0.0075 (8)	0.0086 (8)
C15	0.0497 (9)	0.0840 (14)	0.0778 (12)	0.0158 (9)	0.0176 (8)	0.0110 (10)

Geometric parameters (Å, °)

O4—C13	1.3849 (16)	C1—C6	1.371 (2)
O4—C15	1.441 (2)	C3—C2	1.379 (2)
O5—C12	1.3612 (18)	C3—H3	0.9300
O5—C14	1.4311 (19)	C5—C6	1.373 (2)
N2—C7	1.3547 (19)	C5—H5	0.9300
N2—C4	1.3983 (19)	C9—C10	1.370 (2)
N2—H2	0.870 (19)	C9—H9	0.9300
O3—C7	1.2115 (18)	C6—H6	0.9300
C13—C8	1.394 (2)	C11—C10	1.380 (2)
C13—C12	1.402 (2)	C11—H11	0.9300
C4—C3	1.391 (2)	C2—H2B	0.9300
C4—C5	1.392 (2)	C10—H10	0.9300
C8—C9	1.393 (2)	C14—H14A	0.9600
C8—C7	1.506 (2)	C14—H14B	0.9600
O2—N1	1.221 (2)	C14—H14C	0.9600
N1—O1	1.216 (2)	C15—H15A	0.9600
N1—C1	1.465 (2)	C15—H15B	0.9600
C12—C11	1.383 (2)	C15—H15C	0.9600
C1—C2	1.370 (2)		

C13—O4—C15	113.96 (12)	C6—C5—H5	119.7
C12—O5—C14	117.60 (13)	C4—C5—H5	119.7
C7—N2—C4	129.19 (13)	C10—C9—C8	120.98 (15)
C7—N2—H2	113.0 (12)	C10—C9—H9	119.5
C4—N2—H2	117.7 (12)	C8—C9—H9	119.5
O4—C13—C8	120.97 (12)	C1—C6—C5	118.91 (15)
O4—C13—C12	118.37 (12)	C1—C6—H6	120.5
C8—C13—C12	120.61 (13)	C5—C6—H6	120.5
C3—C4—C5	119.50 (14)	C10—C11—C12	119.99 (14)
C3—C4—N2	123.89 (13)	C10—C11—H11	120.0
C5—C4—N2	116.58 (13)	C12—C11—H11	120.0
C9—C8—C13	118.36 (14)	C1—C2—C3	119.54 (15)
C9—C8—C7	116.01 (13)	C1—C2—H2B	120.2
C13—C8—C7	125.63 (13)	C3—C2—H2B	120.2
O1—N1—O2	123.34 (17)	C9—C10—C11	120.66 (14)
O1—N1—C1	118.39 (18)	C9—C10—H10	119.7
O2—N1—C1	118.27 (18)	C11—C10—H10	119.7
O3—C7—N2	122.79 (14)	O5—C14—H14A	109.5
O3—C7—C8	120.31 (13)	O5—C14—H14B	109.5
N2—C7—C8	116.90 (12)	H14A—C14—H14B	109.5
O5—C12—C11	125.07 (14)	O5—C14—H14C	109.5
O5—C12—C13	115.56 (13)	H14A—C14—H14C	109.5
C11—C12—C13	119.37 (14)	H14B—C14—H14C	109.5
C2—C1—C6	121.87 (15)	O4—C15—H15A	109.5
C2—C1—N1	119.17 (16)	O4—C15—H15B	109.5
C6—C1—N1	118.95 (16)	H15A—C15—H15B	109.5
C2—C3—C4	119.64 (15)	O4—C15—H15C	109.5
C2—C3—H3	120.2	H15A—C15—H15C	109.5
C4—C3—H3	120.2	H15B—C15—H15C	109.5
C6—C5—C4	120.53 (14)		
C15—O4—C13—C8	107.52 (16)	O1—N1—C1—C2	4.1 (3)
C15—O4—C13—C12	-74.91 (17)	O2—N1—C1—C2	-175.90 (16)
C7—N2—C4—C3	-6.9 (3)	O1—N1—C1—C6	-176.93 (17)
C7—N2—C4—C5	174.65 (15)	O2—N1—C1—C6	3.1 (2)
O4—C13—C8—C9	178.74 (14)	C5—C4—C3—C2	-0.4 (2)
C12—C13—C8—C9	1.2 (2)	N2—C4—C3—C2	-178.75 (15)
O4—C13—C8—C7	-2.0 (2)	C3—C4—C5—C6	-0.2 (2)
C12—C13—C8—C7	-179.54 (15)	N2—C4—C5—C6	178.27 (14)
C4—N2—C7—O3	-0.2 (3)	C13—C8—C9—C10	0.2 (2)
C4—N2—C7—C8	179.49 (14)	C7—C8—C9—C10	-179.11 (16)
C9—C8—C7—O3	9.6 (2)	C2—C1—C6—C5	-0.3 (3)
C13—C8—C7—O3	-169.65 (16)	N1—C1—C6—C5	-179.30 (15)
C9—C8—C7—N2	-170.15 (14)	C4—C5—C6—C1	0.6 (2)
C13—C8—C7—N2	10.6 (2)	O5—C12—C11—C10	-178.99 (15)
C14—O5—C12—C11	-1.3 (2)	C13—C12—C11—C10	1.3 (2)
C14—O5—C12—C13	178.43 (14)	C6—C1—C2—C3	-0.2 (3)
O4—C13—C12—O5	0.7 (2)	N1—C1—C2—C3	178.71 (15)

C8—C13—C12—O5	178.26 (14)	C4—C3—C2—C1	0.6 (2)
O4—C13—C12—C11	-179.54 (14)	C8—C9—C10—C11	-0.9 (3)
C8—C13—C12—C11	-2.0 (2)	C12—C11—C10—C9	0.1 (3)

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N2—H2...O4	0.870 (19)	1.924 (19)	2.6805 (16)	144.6 (17)
C5—H5...O3 ⁱ	0.93	2.48	3.2597 (19)	141

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