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Crystal structure and Hirshfeld surface analysis of 4-allyl-2-methoxy-6-nitrophenol

Yassine El Ghallab,[a]* Sanae Derfoufi,[a] El Mostafa Ketatni,[b] Mohamed Saadi[c] and Lahcen El Ammari[c]

[a]Laboratory of Drugs Sciences, Biomedical Research and Biotechnology, Faculty of Medicine and Pharmacy, Hassan II University, BP 9154, Casablanca 20250, Morocco, [b]Laboratory of Organic and Analytical Chemistry, University Sultan Moulay, Slimane, Faculty of Science and Technology, PO Box 523, Beni-Mellal, Morocco, and [c]Laboratoire de Chimie Appliquée des Matériaux, Centre des Sciences des Matériaux, Faculty of Sciences, Mohammed V University in Rabat, Avenue Ibn Batouta, BP 1014, Rabat, Morocco. *Correspondence e-mail: y_ghallab@yahoo.com

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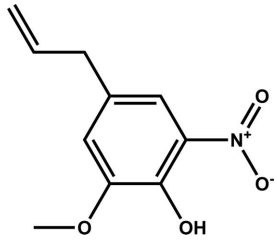
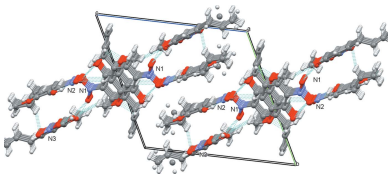
The asymmetric unit of the title compound, $C_{10}H_{11}NO_4$, which was synthesized *via* nitration reaction of eugenol (4-allyl-2-methoxyphenol) with a mixture of nitric acid and sulfuric acid, consists of three independent molecules of similar geometry. Each molecule displays an intramolecular hydrogen bond involving the hydroxide and the nitro group forming an *S*(6) motif. The crystal cohesion is ensured by intermolecular C—H···O hydrogen bonds in addition to π–π stacking interactions between the aromatic rings [centroid–centroid distances = 3.6583 (17)–4.0624 (16) Å]. The Hirshfeld surface analysis and the two-dimensional fingerprint plots show that H···H (39.6%), O···H/H···O (37.7%), C···H/H···C (12.5%) and C···C (4%) are the most important contributors towards the crystal packing.

1. Chemical context

Eugenol, the main constituent of clove essential oil, has many interesting biological properties and participates in the synthesis of bioactive compounds (Kaufman, 2015). The nitroeugenol isomers were tested for their antifungal activity, growth inhibitory activity on human tumor cell lines (Carrasco *et al.*, 2012, 2008), and antioxidant activity (Hidalgo *et al.*, 2009). We report here the synthesis, structure, spectrometric and spectroscopic characterization of the title compound along with an analysis of the calculated Hirshfeld surface and the two-dimensional fingerprint plots.





2. Structural commentary

The asymmetric unit of the title compound (Fig. 1) contains three independent molecules of similar geometry hereafter referred as Mol-N1 (N1/O1–O4/C1–C10), Mol-N2 (N2/O5–O8/C11–C20) and Mol-N3 (N3/O9–O12/C21–C30). The planes through the nitro groups are almost coplanar with those of the attached benzene rings, forming dihedral angles ranging from 2.1 (3)° in Mol-N3 to 6.38 (13)° in Mol-N2. The mean planes though the allyl group C1/C2/C3 (molecule Mol-

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

D—H···A	D—H	H···A	D···A	D—H···A
O10—H10···O11	0.86 (3)	1.81 (3)	2.594 (2)	149 (2)
O6—H6···O7	0.83 (2)	1.83 (2)	2.584 (2)	152 (2)
O2—H2*O*···O3	0.91 (3)	1.78 (3)	2.587 (2)	146 (2)
C12*A*—H12*A*···O12	0.93	2.58	3.382 (4)	145
C12*B*—H12*B*···O3^{i}	0.93	2.56	3.325 (8)	140
C9—H9···O7^{ii}	0.93	2.59	3.394 (2)	145

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

N1) and the disordered allyl groups C11*A*/C11*B*/C12*A*/C12*B*/C13 (molecule Mol-N2) and C21*A*/C21*B*/C22*A*/C22*B*/C23 (molecule Mol-N3) are oriented with dihedral angles of 67.5 (3), 80.8 (3) and 86.1 (4)°, respectively, to the attached benzene rings. The benzene rings of molecules Mol-N2 and Mol-N3 are approximately parallel to each other [dihedral angle 10.60 (7)°], and roughly perpendicular to that of Mol-N1 [dihedral angles of 83.65 (7) and 79.22 (6)°, respectively]. A strong intramolecular O—H···O hydrogen bond involving a nitro O atom and the H atom of the hydroxide group forming an $S(6)$ motif is observed in each molecule (Table 1).

3. Supramolecular features

In the crystal, the molecules are connected by intermolecular C12*A*—H12*A*···O12, C12*B*—H12*B*···O3 and C9—H9···O7 hydrogen bonds (Table 1; Figs. 2 and 3). In addition, centrosymmetrically related pairs of Mol-N1 molecules are connected by π–π interactions to form dimeric units [centroid–centroid distance = 3.7213 (15) Å] (Fig. 2), whereas the Mol-N2 and Mol-N3 molecules are stacked through π–π interactions to form chains running parallel to the b axis [$Cg2\cdots Cg2^{i}$ = 3.6583 (17) Å; $Cg2\cdots Cg3^{ii}$ = 3.6613 (18) Å; $Cg3\cdots Cg3^{iii}$ = 4.0624 (16) Å; symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $1+x, y, z$; (iii) $-x, -y, 1-z$].

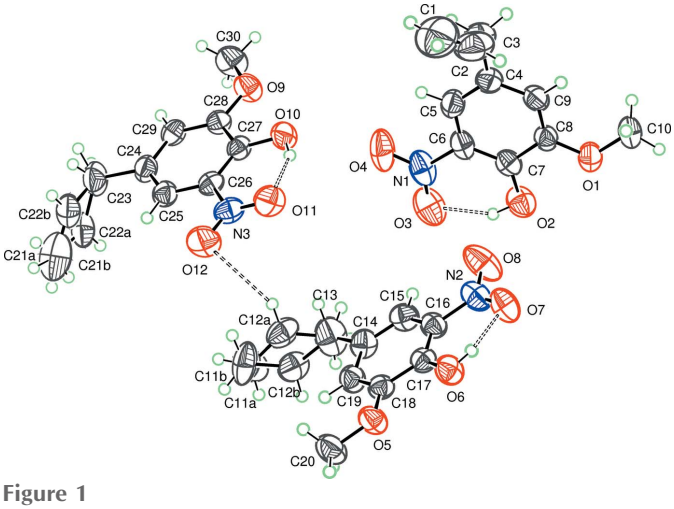


Figure 1
The asymmetric unit of the title compound with the displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small circles. Intra- and intermolecular hydrogen bonds are shown as dashed lines.

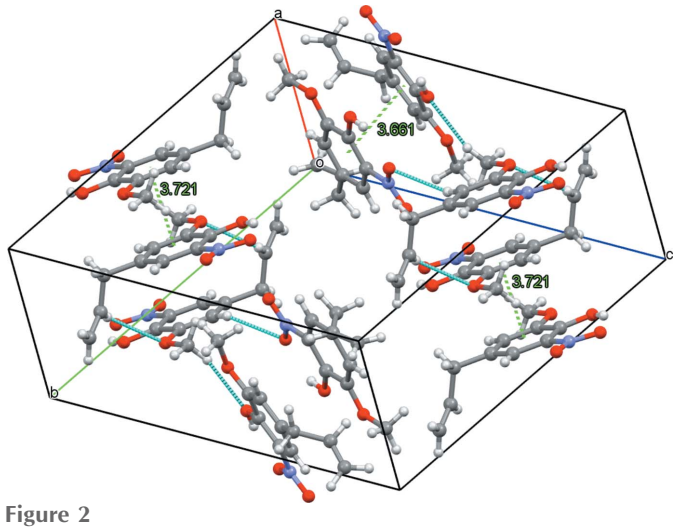


Figure 2
Partial crystal packing of the title compound showing molecules connected by hydrogen bonds (dashed cyan lines) and π–π interactions (dashed green lines).

4. Hirshfeld surface analysis

In order to explore the nature of the intermolecular contacts and their role in the crystal packing, Hirshfeld surfaces (Spackman & Jayatilaka, 2009) and the associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) were calculated using *Crystal Explorer 17.5* (Turner *et al.*, 2017). The three-dimensional molecular Hirshfeld surfaces of the three molecules Mol-N1, Mol-N2 and Mol-N3 and the overall surface were generated using a high standard surface resolution colour-mapped over the normalized contact distance. The red, white and blue regions visible on the d_{norm} surfaces indicate contacts with distances shorter, longer and equal to the van der Waals radii (Fig. 4*a* and 5*a*). The shape-index of the Hirshfeld surface is a tool to visualize the π–π stacking interactions (Fig. 4*b* and 5*b*). The red spots in Fig. 4*a* correspond to the strong C—H···O hydrogen-bond interactions in the crystal structure; in Mol-N1 two of them involve the O atoms of the methoxy (O1) and nitro (O3) groups as acceptors with allyl H atoms (C22*B*– H22*B*···O1 and C12*B*—H12*B*···O3), while the other is due to the interatomic interaction between the aromatic H9 donor atom and the nitro O7 oxygen atom (C9—H9···O7). The longer O—H···O

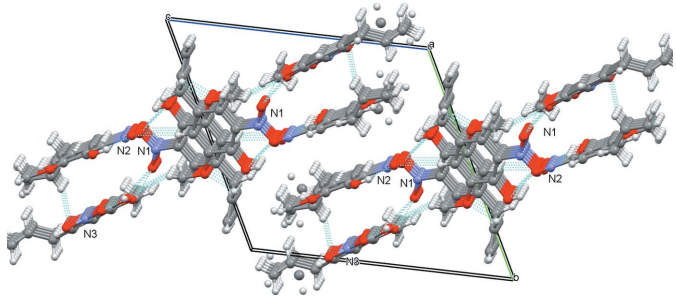


Figure 3
Crystal packing of the title compound viewed along the a axis showing molecules linked by hydrogen bonds (dashed cyan lines).

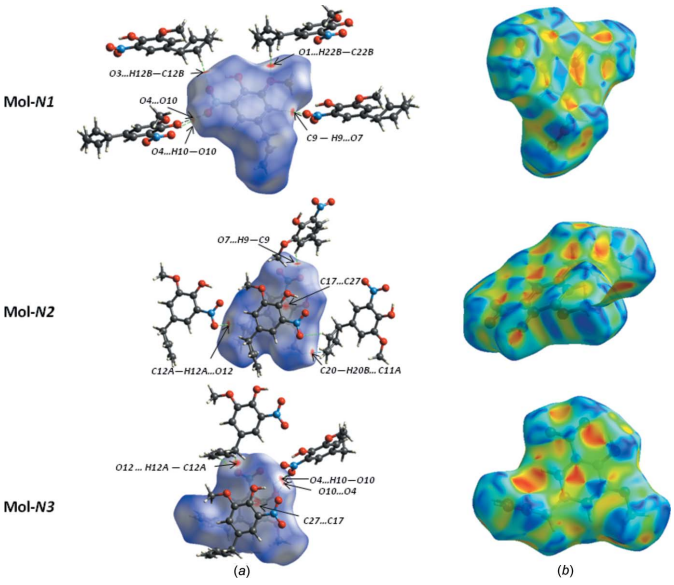


Figure 4
Hirshfeld surface of the title compound (symmetry-independent molecules Mol-N1, Mol-N2 and Mol-N3), with (*a*) d_{norm} with the interaction of neighbouring molecules and (*b*) shape-index.

hydrogen bonds and O···O interactions are characterized by smaller red spots close to each other on the surface, where the faint red spot indicating the O—H···O interactions is associated with the longest O···O contact of 2.96 (3) Å in Mol-N1 and Mol-N3. In Mol-N2, the red spots correspond to C—H···O (C9—H9···O7 and C12*A*—H12*A*···O12) and C—H···C (C20—H20*B*···C11*A*) hydrogen-bond interactions. The corresponding fingerprint plots for each of the independent molecules and for the entire asymmetric unit, showing characteristic pseudo-symmetric wings in the d_e and d_i diagonal axes, and those delineated into H···H, O···H/H···O, C···H/H···C and C···C contacts are illustrated in Fig. 6. The result of the quantitative analysis of all types of intermolecular contacts present in the title compound is summarized in Fig. 7. The most important interaction is H···H, contributing 45.4% to the overall crystal packing (Fig. 6*b*), which is reflected in the widely scattered points of high density due to the large hydrogen-atom content of the molecule. The contribution from the O···H/H···O contacts (31.7%), corresponding to C—H···O and O—H···O interactions, is represented by a pair of sharp spikes characteristic of a strong hydrogen-bond

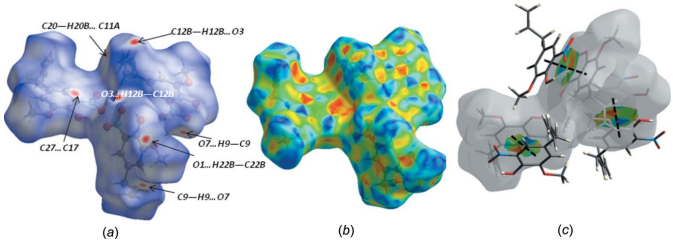


Figure 5
Views of the Hirshfeld surface for a reference molecule of the title compound mapped over (*a*) d_{norm}, (*b*) shape-index and (*c*) the shape-index property highlighting the π–π interactions as black dashed lines.

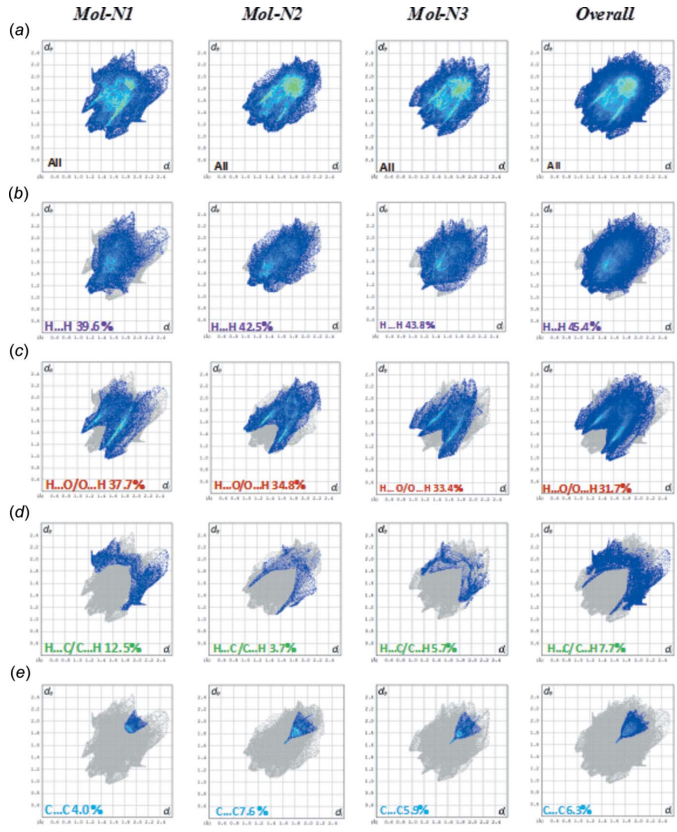


Figure 6
Fingerprint plots representative of specific interatomic contacts in the title compound (symmetry-independent molecules Mol-N1, Mol-N2, Mol-N3 and overall), delineated into H···H, O···H/H···O, C···H/H···C and C···C interactions.

interaction with $d_e + d_i \simeq 2.5$Å (Fig. 6*c*). In the absence of weak C—H···π interactions in the crystal, the pair of characteristic wings in the fingerprint plot delineated into H···C/C···H contacts (7.7% contribution) have a symmetrical distribution of points (Fig. 6*d*), with the tips at $d_e + d_i \simeq$ 2.65 Å. The distribution of points in the $d_e = d_i \simeq 1.6$ Å range in the fingerprint plot delineated into C···C contacts (Fig. 6*e*) indicates the existence of weak π–π stacking interactions between the phenyl rings, which are indicated by adjacent red and blue triangles in the shape-index map (Fig. 4*b* and Fig. 5*c*). The small contribution of the other weak intermolecular O···O, N···H/H···N, C···O/O···C, C···N/N···C and N···O/O···N contacts has a negligible effect on the packing.

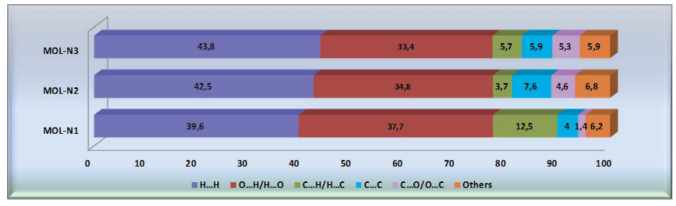


Figure 7
Percentage contribution of various intermolecular interactions in the title compound obtained from decomposed fingerprint plots.

5. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.40, May 2019; Groom *et al.*, 2016) of eugenol derivatives revealed two compounds with very similar structures but with a different position of the nitro group or with the hydroxide group substituted by an acetate group, *viz.* 4-allyl-2-methoxy-5-nitrophenyl acetate (refcode: TEJREG; Carrasco-Altamirano *et al.*, 2006) and 4-hydroxy-3-methoxy-5-nitroacetophenone (5-nitroapocynin) (MUCDOE; Babu *et al.*, 2009). A third related compound, 4-hydroxy-3-methoxy-5-nitrobenzaldehyde, has recently been reported (Vusak *et al.*, 2020). All of these compounds exhibit intramolecular hydrogen bonds involving the nitro O atoms with the H atoms of the hydroxide group, and other intermolecular hydrogen bonds, in addition to π–π interactions, which assure the crystal cohesion.

6. Synthesis and crystallization

In a 250 mL flat-bottom flask containing a stirred solution of eugenol (2.12 g, 12.9 mmol) and dichloromethane (60 mL), a mixture of concentrated sulfuric acid (0.78 mL) and concentrated nitric acid (0.80 mL) was added dropwise for 30 min at 273 K. The complete disappearance of the starting product was confirmed by means of thin layer chromatography using *n*-hexane/AcOEt (9:1 *v*/*v*) as eluent. The reaction mixture was diluted with dichloromethane, washed with brine (3 × 10 mL), dried over anhydrous Na_2SO_4 and concentrated under vacuum. The crude product was subjected to chromatography on a silica-gel column with *n*-hexane/AcOEt (9:1 *v*/*v*) as eluent to afford the title compound as a reddish-orange liquid. Reddish-orange crystals formed spontaneously with a yield of 56%. Good quality crystals suitable for single crystal X-ray diffraction analysis were obtained by slow evaporation of an *n*-hexane:AcOEt solution, m.p. = 317–319 K.

IR (cm^{-1}): 3235, 3080, 3016, 2971, 2910, 1638, 1537, 1392, 1331, 1262, 1128, 1059, 909, 763. The FT–IR spectrum (Fig. 8) illustrates several bands characteristic of 4-allyl-2-methoxy-6-nitrophenol. The absorption band at 3235 cm^{-1} was assigned to the O—H stretching vibration. The bands located at 3080 and 3016 cm^{-1} correspond to the C═CH bond of the aromatic ring and CH═CH_2 bond of the allyl group, respectively. The remarkably strong band at 1537 cm^{-1} was attributed to the stretching vibration of the nitro group. Other C═C stretching vibrations are at 2971, 2910 and 1638 cm^{-1}. The FT–IR spectrum peaks are in agreement with the reported data for similar compounds (Carrasco *et al.*, 2008; Egorov *et al.*, 2014; Heredia *et al.*, 2016).

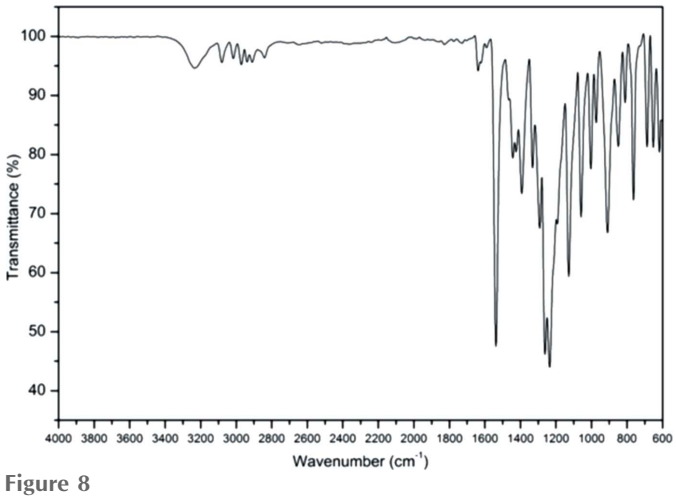


Figure 8
The FT–IR spectrum of the title compound.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{11}NO_4$
M_r	209.20
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	8.706 (3), 13.753 (5), 14.683 (5)
α, β, γ (°)	116.142 (11), 93.871 (12), 96.985 (12)
V (Å^3)	1552.0 (9)
Z	6
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.31 × 0.28 × 0.26
Data collection	
Diffractometer	Bruker D8 VENTURE Super DUO
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.707, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	54955, 6334, 4687
R_{int}	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.134, 1.02
No. of reflections	6334
No. of parameters	460
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.41, −0.20

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2016/6* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012); *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 10.7 (*s*, 1H, OH conjugated), 7.53 (*s*, 1H, Ar-H), 6.99 (*s*, 1H, Ar-H), 6.01–5.88 (*m*, 1H), 5.19–5.13 (*m*, 2H), 3.96 (*s*, 3H), 3.39–3.37 (*d*, 2H). ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 149.87, 144.90, 135.95, 133.66, 131.24, 118.63, 117.16, 115.11, 114.29, 56.72, 39.41. FTMS–ESI, *m*/*z*: 208.04616 (100%) [$C_{10}H_{11}NO_4$].

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C-bound H atoms were located in a difference-Fourier map and refined as riding with C—H = 0.93–0.97 Å, and U_{iso}(H) = 1.2 U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms. A rotating model was used for the methyl groups. The hydroxyl H atoms were located in a difference-Fourier map and refined freely. The two allyl groups of Mol-

N2 and Mol-N3 are disordered over two sets of sites with refined occupancy ratios of 0.648 (8):0.352 (8) and 0.668 (9):0.332 (9) respectively. One outlier (100) was omitted in the cycles of refinement.

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Crystal structure and Hirshfeld surface analysis of 4-allyl-2-methoxy-6-nitrophenol

Yassine El Ghallab, Sanae Derfoufi, El Mostafa Ketatni, Mohamed Saadi and Lahcen El Ammari

Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016/6* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *publCIF* (Westrip, 2010).

4-Allyl-2-methoxy-6-nitrophenol

Crystal data

$C_{10}H_{11}NO_4$
$M_r = 209.20$
Triclinic, $P\overline{1}$
$a = 8.706$ (3) Å
$b = 13.753$ (5) Å
$c = 14.683$ (5) Å
$\alpha = 116.142$ (11)°
$\beta = 93.871$ (12)°
$\gamma = 96.985$ (12)°
$V = 1552.0$ (9) Å^3
$Z = 6$
$F(000) = 660$
$D_x = 1.343$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6334 reflections
$\theta = 2.7$–26.4°
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Block, orange
0.31 × 0.28 × 0.26 mm

Data collection

Bruker D8 VENTURE Super DUO diffractometer
Radiation source: INCOATEC IμS micro-focus source
HELIOS mirror optics monochromator
Detector resolution: 10.4167 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min} = 0.707$, $T_{max} = 0.746$
54955 measured reflections
6334 independent reflections
4687 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.7°$
$h = -10\rightarrow10$
$k = -17\rightarrow17$
$l = -18\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.134$
$S = 1.02$
6334 reflections
460 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.4096P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Extinction correction: SHELXL-2018/3 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.021 (2)

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}	Occ. (<1)
C1	0.7703 (5)	0.1282 (3)	0.9575 (3)	0.1442 (14)	
H1A	0.669741	0.087821	0.935152	0.173*	
H1B	0.856141	0.092334	0.943266	0.173*	
C2	0.7907 (4)	0.2334 (3)	1.0082 (2)	0.1086 (9)	
H2	0.893725	0.269409	1.028622	0.130*	
C3	0.6708 (3)	0.30301 (19)	1.03745 (18)	0.0862 (7)	
H3A	0.681015	0.341956	1.111572	0.103*	
H3B	0.568772	0.256699	1.013201	0.103*	
C4	0.6796 (2)	0.38578 (15)	0.99543 (14)	0.0619 (5)	
C5	0.5910 (2)	0.36410 (16)	0.90633 (14)	0.0628 (5)	
H5	0.523012	0.297687	0.870481	0.075*	
C6	0.60267 (19)	0.44162 (15)	0.86912 (13)	0.0564 (4)	
C7	0.7021 (2)	0.54237 (14)	0.92020 (13)	0.0533 (4)	
C8	0.79192 (19)	0.56398 (14)	1.01284 (12)	0.0516 (4)	
C9	0.7813 (2)	0.48670 (15)	1.04822 (13)	0.0576 (4)	
H9	0.842850	0.501427	1.108578	0.069*	
C10	0.9739 (3)	0.69329 (18)	1.15663 (15)	0.0763 (6)	
H10A	0.904755	0.692601	1.204647	0.115*	
H10B	1.034076	0.765533	1.181861	0.115*	
H10C	1.042918	0.641401	1.148116	0.115*	
N1	0.50602 (19)	0.41416 (17)	0.77400 (13)	0.0741 (5)	
O1	0.88445 (16)	0.66397 (10)	1.06010 (10)	0.0687 (4)	
O2	0.71821 (19)	0.62197 (12)	0.89023 (11)	0.0745 (4)	
O3	0.5206 (2)	0.47908 (17)	0.73564 (13)	0.1008 (6)	
O4	0.41380 (18)	0.32741 (15)	0.73302 (13)	0.0988 (6)	
C11A	0.4789 (14)	0.2126 (11)	0.2212 (9)	0.107 (4)	0.648 (8)
H11A	0.504547	0.263438	0.196542	0.128*	0.648 (8)
H11B	0.429013	0.141711	0.176573	0.128*	0.648 (8)
C12A	0.5127 (4)	0.2406 (3)	0.3175 (3)	0.0775 (16)	0.648 (8)
H12A	0.484720	0.186924	0.338427	0.093*	0.648 (8)
C11B	0.472 (2)	0.1982 (15)	0.2141 (18)	0.094 (7)	0.352 (8)
H11C	0.441218	0.141392	0.230507	0.113*	0.352 (8)
H11D	0.453749	0.186675	0.146615	0.113*	0.352 (8)
C12B	0.5447 (7)	0.3007 (8)	0.2904 (7)	0.075 (3)	0.352 (8)
H12B	0.570414	0.351468	0.265767	0.090*	0.352 (8)

C13	0.5863 (3)	0.3424 (3)	0.3925 (2)	0.1000 (8)	
H13A	0.527879	0.363796	0.450327	0.120*	0.648 (8)
H13B	0.581070	0.395340	0.365880	0.120*	0.648 (8)
H13C	0.522486	0.297742	0.416294	0.120*	0.352 (8)
H13D	0.559627	0.415525	0.424614	0.120*	0.352 (8)
C14	0.7560 (2)	0.35083 (16)	0.43148 (15)	0.0619 (5)	
C15	0.8104 (2)	0.39916 (15)	0.53351 (15)	0.0610 (5)	
H15	0.742814	0.427603	0.581481	0.073*	
C16	0.9676 (2)	0.40594 (13)	0.56597 (13)	0.0532 (4)	
C17	1.07440 (19)	0.36536 (13)	0.49776 (13)	0.0493 (4)	
C18	1.01636 (19)	0.31557 (14)	0.39249 (12)	0.0507 (4)	
C19	0.8614 (2)	0.30891 (15)	0.36115 (14)	0.0568 (4)	
H19	0.825339	0.275728	0.291428	0.068*	
C20	1.0751 (3)	0.2270 (2)	0.22242 (15)	0.0808 (6)	
H20A	0.995975	0.163994	0.203937	0.121*	
H20B	1.162250	0.204457	0.186106	0.121*	
H20C	1.033249	0.278869	0.204844	0.121*	
N2	1.0193 (2)	0.45748 (14)	0.67507 (12)	0.0697 (4)	
O5	1.12547 (14)	0.27684 (12)	0.32966 (9)	0.0664 (4)	
O6	1.22701 (14)	0.36805 (12)	0.52235 (11)	0.0641 (4)	
O7	1.1606 (2)	0.47349 (13)	0.70603 (11)	0.0850 (4)	
O8	0.9228 (2)	0.48412 (17)	0.73412 (12)	0.1068 (6)	
C21A	−0.2510 (12)	−0.0879 (7)	0.0621 (5)	0.121 (4)	0.668 (9)
H21A	−0.246926	−0.160899	0.045748	0.145*	0.668 (9)
H21B	−0.232777	−0.062093	0.014302	0.145*	0.668 (9)
C22A	−0.2799 (4)	−0.0266 (4)	0.1454 (3)	0.0764 (14)	0.668 (9)
H22A	−0.281443	0.044748	0.154888	0.092*	0.668 (9)
C21B	−0.233 (2)	−0.0934 (12)	0.0612 (16)	0.119 (8)	0.332 (9)
H21C	−0.233452	−0.028161	0.056564	0.143*	0.332 (9)
H21D	−0.206887	−0.154152	0.007319	0.143*	0.332 (9)
C22B	−0.2705 (8)	−0.0999 (9)	0.1445 (7)	0.078 (3)	0.332 (9)
H22B	−0.264145	−0.170393	0.135978	0.094*	0.332 (9)
C23	−0.3132 (3)	−0.0433 (2)	0.23518 (18)	0.0893 (7)	
H23A	−0.323904	−0.121250	0.215662	0.107*	0.668 (9)
H23B	−0.412887	−0.021186	0.253318	0.107*	0.668 (9)
H23C	−0.385321	−0.093840	0.248213	0.107*	0.332 (9)
H23D	−0.371941	0.009953	0.229832	0.107*	0.332 (9)
C24	−0.1915 (2)	0.01826 (14)	0.32956 (14)	0.0575 (4)	
C25	−0.0350 (2)	0.01984 (14)	0.32198 (13)	0.0547 (4)	
H25	−0.001961	−0.015992	0.258299	0.066*	
C26	0.07431 (18)	0.07529 (13)	0.40997 (13)	0.0483 (4)	
C27	0.03114 (18)	0.13021 (13)	0.50680 (12)	0.0468 (4)	
C28	−0.13073 (18)	0.12718 (13)	0.51315 (13)	0.0494 (4)	
C29	−0.23785 (19)	0.07271 (14)	0.42663 (13)	0.0538 (4)	
H29	−0.343672	0.071824	0.432412	0.065*	
C30	−0.3235 (2)	0.1869 (2)	0.62435 (17)	0.0773 (6)	
H30A	−0.364262	0.226379	0.590885	0.116*	
H30B	−0.331320	0.224162	0.696188	0.116*	

H30C	−0.382519	0.113848	0.595437	0.116*
N3	0.23790 (17)	0.07279 (13)	0.39773 (13)	0.0618 (4)
O9	−0.16410 (14)	0.18116 (12)	0.61035 (9)	0.0671 (4)
O10	0.12736 (15)	0.18700 (11)	0.59525 (10)	0.0635 (3)
O11	0.33723 (15)	0.11885 (14)	0.47501 (12)	0.0795 (4)
O12	0.27365 (17)	0.02443 (14)	0.31208 (13)	0.0892 (5)
H2O	0.658 (3)	0.593 (2)	0.829 (2)	0.110 (9)*
H6	1.236 (3)	0.3989 (19)	0.5856 (19)	0.083 (8)*
H10	0.218 (3)	0.180 (2)	0.5758 (19)	0.095 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.187 (4)	0.103 (2)	0.156 (3)	0.034 (2)	0.036 (3)	0.066 (2)
C2	0.104 (2)	0.135 (3)	0.124 (2)	0.0102 (19)	0.0140 (18)	0.094 (2)
C3	0.1077 (18)	0.0779 (14)	0.0734 (14)	−0.0027 (13)	0.0214 (13)	0.0380 (12)
C4	0.0659 (11)	0.0605 (11)	0.0546 (10)	0.0049 (9)	0.0180 (9)	0.0220 (9)
C5	0.0520 (10)	0.0601 (11)	0.0576 (11)	−0.0001 (8)	0.0113 (8)	0.0118 (9)
C6	0.0435 (9)	0.0666 (11)	0.0460 (9)	0.0157 (8)	0.0047 (7)	0.0124 (8)
C7	0.0523 (9)	0.0584 (10)	0.0486 (9)	0.0199 (8)	0.0120 (7)	0.0203 (8)
C8	0.0486 (9)	0.0534 (9)	0.0450 (9)	0.0077 (7)	0.0091 (7)	0.0151 (7)
C9	0.0595 (10)	0.0661 (11)	0.0434 (9)	0.0078 (8)	0.0075 (7)	0.0220 (8)
C10	0.0808 (14)	0.0703 (13)	0.0558 (11)	−0.0058 (10)	−0.0092 (10)	0.0160 (10)
N1	0.0526 (9)	0.0891 (13)	0.0605 (10)	0.0260 (9)	−0.0019 (8)	0.0139 (10)
O1	0.0777 (9)	0.0581 (7)	0.0579 (7)	−0.0041 (6)	−0.0057 (6)	0.0210 (6)
O2	0.0933 (11)	0.0682 (9)	0.0649 (9)	0.0219 (7)	0.0013 (8)	0.0320 (7)
O3	0.0982 (12)	0.1215 (14)	0.0802 (11)	0.0284 (10)	−0.0179 (9)	0.0452 (11)
O4	0.0648 (9)	0.1008 (12)	0.0829 (11)	0.0057 (9)	−0.0214 (8)	0.0052 (9)
C11A	0.071 (7)	0.169 (11)	0.086 (6)	0.015 (6)	0.001 (5)	0.066 (7)
C12A	0.0454 (17)	0.089 (3)	0.109 (4)	0.0035 (17)	0.0061 (18)	0.058 (3)
C11B	0.047 (8)	0.072 (6)	0.116 (13)	0.002 (5)	−0.011 (7)	0.005 (7)
C12B	0.048 (3)	0.098 (6)	0.095 (6)	0.006 (3)	−0.009 (3)	0.063 (5)
C13	0.0515 (12)	0.129 (2)	0.0964 (19)	0.0272 (13)	0.0084 (12)	0.0283 (16)
C14	0.0487 (10)	0.0655 (11)	0.0684 (12)	0.0144 (8)	0.0089 (8)	0.0262 (9)
C15	0.0617 (11)	0.0596 (10)	0.0645 (11)	0.0196 (8)	0.0200 (9)	0.0267 (9)
C16	0.0638 (11)	0.0497 (9)	0.0492 (9)	0.0134 (8)	0.0077 (8)	0.0243 (8)
C17	0.0489 (9)	0.0491 (9)	0.0547 (9)	0.0089 (7)	0.0046 (7)	0.0281 (8)
C18	0.0457 (9)	0.0558 (9)	0.0537 (10)	0.0080 (7)	0.0093 (7)	0.0274 (8)
C19	0.0479 (9)	0.0638 (11)	0.0542 (10)	0.0076 (8)	0.0029 (7)	0.0237 (8)
C20	0.0663 (13)	0.1128 (18)	0.0546 (11)	0.0144 (12)	0.0145 (9)	0.0295 (12)
N2	0.0902 (13)	0.0670 (10)	0.0547 (9)	0.0267 (9)	0.0126 (9)	0.0265 (8)
O5	0.0479 (7)	0.0950 (10)	0.0525 (7)	0.0144 (6)	0.0107 (5)	0.0287 (7)
O6	0.0518 (7)	0.0819 (9)	0.0592 (8)	0.0140 (6)	0.0006 (6)	0.0327 (7)
O7	0.0913 (11)	0.0988 (11)	0.0571 (8)	0.0192 (9)	−0.0071 (8)	0.0300 (8)
O8	0.1188 (14)	0.1410 (16)	0.0609 (9)	0.0613 (12)	0.0319 (9)	0.0334 (10)
C21A	0.134 (6)	0.142 (7)	0.042 (3)	−0.033 (5)	−0.013 (3)	0.021 (4)
C22A	0.075 (2)	0.061 (3)	0.072 (3)	0.0025 (17)	−0.0183 (16)	0.0174 (19)
C21B	0.112 (11)	0.054 (7)	0.160 (19)	0.039 (8)	−0.008 (9)	0.019 (8)

C22B	0.070 (4)	0.069 (6)	0.071 (5)	−0.001 (4)	−0.005 (3)	0.014 (4)
C23	0.0632 (13)	0.1015 (17)	0.0703 (14)	−0.0118 (12)	−0.0097 (11)	0.0184 (13)
C24	0.0488 (9)	0.0548 (10)	0.0595 (10)	−0.0009 (7)	0.0012 (8)	0.0207 (8)
C25	0.0538 (10)	0.0506 (9)	0.0537 (10)	0.0057 (7)	0.0103 (8)	0.0188 (8)
C26	0.0398 (8)	0.0496 (9)	0.0610 (10)	0.0078 (7)	0.0099 (7)	0.0297 (8)
C27	0.0411 (8)	0.0489 (9)	0.0534 (9)	0.0048 (7)	0.0012 (7)	0.0274 (7)
C28	0.0450 (9)	0.0517 (9)	0.0540 (9)	0.0097 (7)	0.0087 (7)	0.0256 (8)
C29	0.0385 (8)	0.0576 (10)	0.0636 (11)	0.0048 (7)	0.0055 (7)	0.0270 (8)
C30	0.0586 (12)	0.0989 (16)	0.0749 (13)	0.0302 (11)	0.0247 (10)	0.0333 (12)
N3	0.0459 (8)	0.0723 (10)	0.0775 (11)	0.0128 (7)	0.0157 (8)	0.0415 (9)
O9	0.0515 (7)	0.0899 (9)	0.0549 (7)	0.0189 (6)	0.0122 (6)	0.0260 (7)
O10	0.0462 (7)	0.0841 (9)	0.0559 (7)	0.0035 (6)	−0.0022 (6)	0.0309 (7)
O11	0.0399 (7)	0.1124 (12)	0.0902 (10)	0.0106 (7)	0.0049 (7)	0.0505 (9)
O12	0.0636 (9)	0.1155 (13)	0.0860 (11)	0.0232 (8)	0.0337 (8)	0.0380 (9)

Geometric parameters (Å, º)

C1—C2	1.285 (4)	C16—N2	1.448 (2)
C1—H1A	0.9300	C17—O6	1.345 (2)
C1—H1B	0.9300	C17—C18	1.411 (2)
C2—C3	1.460 (4)	C18—O5	1.359 (2)
C2—H2	0.9300	C18—C19	1.374 (2)
C3—C4	1.512 (3)	C19—H19	0.9300
C3—H3A	0.9700	C20—O5	1.423 (2)
C3—H3B	0.9700	C20—H20A	0.9600
C4—C5	1.363 (3)	C20—H20B	0.9600
C4—C9	1.404 (3)	C20—H20C	0.9600
C5—C6	1.393 (3)	N2—O8	1.219 (2)
C5—H5	0.9300	N2—O7	1.240 (2)
C6—C7	1.392 (3)	O6—H6	0.83 (2)
C6—N1	1.449 (2)	C21A—C22A	1.206 (8)
C7—O2	1.344 (2)	C21A—H21A	0.9300
C7—C8	1.412 (2)	C21A—H21B	0.9300
C8—O1	1.355 (2)	C22A—C23	1.475 (5)
C8—C9	1.370 (2)	C22A—H22A	0.9300
C9—H9	0.9300	C21B—C22B	1.32 (2)
C10—O1	1.432 (2)	C21B—H21C	0.9300
C10—H10A	0.9600	C21B—H21D	0.9300
C10—H10B	0.9600	C22B—C23	1.320 (9)
C10—H10C	0.9600	C22B—H22B	0.9300
N1—O4	1.224 (2)	C23—C24	1.520 (3)
N1—O3	1.247 (2)	C23—H23A	0.9700
O2—H2O	0.91 (3)	C23—H23B	0.9700
C11A—C12A	1.293 (12)	C23—H23C	0.9700
C11A—H11A	0.9300	C23—H23D	0.9700
C11A—H11B	0.9300	C24—C25	1.373 (2)
C12A—C13	1.383 (5)	C24—C29	1.402 (3)
C12A—H12A	0.9300	C25—C26	1.395 (2)

C11B—C12B	1.39 (2)	C25—H25	0.9300
C11B—H11C	0.9300	C26—C27	1.389 (2)
C11B—H11D	0.9300	C26—N3	1.450 (2)
C12B—C13	1.351 (9)	C27—O10	1.341 (2)
C12B—H12B	0.9300	C27—C28	1.415 (2)
C13—C14	1.520 (3)	C28—O9	1.359 (2)
C13—H13A	0.9700	C28—C29	1.370 (2)
C13—H13B	0.9700	C29—H29	0.9300
C13—H13C	0.9700	C30—O9	1.423 (2)
C13—H13D	0.9700	C30—H30A	0.9600
C14—C15	1.364 (3)	C30—H30B	0.9600
C14—C19	1.404 (3)	C30—H30C	0.9600
C15—C16	1.397 (3)	N3—O12	1.219 (2)
C15—H15	0.9300	N3—O11	1.240 (2)
C16—C17	1.392 (2)	O10—H10	0.86 (3)

C2—C1—H1A	120.0	O6—C17—C16	126.40 (16)
C2—C1—H1B	120.0	O6—C17—C18	116.84 (15)
H1A—C1—H1B	120.0	C16—C17—C18	116.75 (15)
C1—C2—C3	127.5 (3)	O5—C18—C19	125.52 (16)
C1—C2—H2	116.2	O5—C18—C17	114.12 (14)
C3—C2—H2	116.2	C19—C18—C17	120.36 (16)
C2—C3—C4	113.5 (2)	C18—C19—C14	121.83 (17)
C2—C3—H3A	108.9	C18—C19—H19	119.1
C4—C3—H3A	108.9	C14—C19—H19	119.1
C2—C3—H3B	108.9	O5—C20—H20A	109.5
C4—C3—H3B	108.9	O5—C20—H20B	109.5
H3A—C3—H3B	107.7	H20A—C20—H20B	109.5
C5—C4—C9	118.99 (18)	O5—C20—H20C	109.5
C5—C4—C3	121.19 (18)	H20A—C20—H20C	109.5
C9—C4—C3	119.81 (18)	H20B—C20—H20C	109.5
C4—C5—C6	119.87 (17)	O8—N2—O7	121.72 (18)
C4—C5—H5	120.1	O8—N2—C16	119.09 (19)
C6—C5—H5	120.1	O7—N2—C16	119.18 (17)
C7—C6—C5	122.23 (16)	C18—O5—C20	117.02 (14)
C7—C6—N1	120.24 (18)	C17—O6—H6	101.5 (17)
C5—C6—N1	117.52 (17)	C22A—C21A—H21A	120.0
O2—C7—C6	125.82 (16)	C22A—C21A—H21B	120.0
O2—C7—C8	116.97 (16)	H21A—C21A—H21B	120.0
C6—C7—C8	117.20 (16)	C21A—C22A—C23	132.2 (6)
O1—C8—C9	125.18 (16)	C21A—C22A—H22A	113.9
O1—C8—C7	114.62 (16)	C23—C22A—H22A	113.9
C9—C8—C7	120.19 (16)	C22B—C21B—H21C	120.0
C8—C9—C4	121.50 (17)	C22B—C21B—H21D	120.0
C8—C9—H9	119.3	H21C—C21B—H21D	120.0
C4—C9—H9	119.3	C23—C22B—C21B	143.2 (12)
O1—C10—H10A	109.5	C23—C22B—H22B	108.4
O1—C10—H10B	109.5	C21B—C22B—H22B	108.4

H10A—C10—H10B	109.5	C22B—C23—C24	120.4 (4)
O1—C10—H10C	109.5	C22A—C23—C24	115.5 (2)
H10A—C10—H10C	109.5	C22A—C23—H23A	108.4
H10B—C10—H10C	109.5	C24—C23—H23A	108.4
O4—N1—O3	121.93 (19)	C22A—C23—H23B	108.4
O4—N1—C6	119.1 (2)	C24—C23—H23B	108.4
O3—N1—C6	118.96 (19)	H23A—C23—H23B	107.5
C8—O1—C10	117.66 (15)	C22B—C23—H23C	107.2
C7—O2—H2O	105.1 (17)	C24—C23—H23C	107.2
C12A—C11A—H11A	120.0	C22B—C23—H23D	107.2
C12A—C11A—H11B	120.0	C24—C23—H23D	107.2
H11A—C11A—H11B	120.0	H23C—C23—H23D	106.9
C11A—C12A—C13	126.5 (7)	C25—C24—C29	118.75 (16)
C11A—C12A—H12A	116.8	C25—C24—C23	120.96 (18)
C13—C12A—H12A	116.8	C29—C24—C23	120.28 (17)
C12B—C11B—H11C	120.0	C24—C25—C26	119.89 (16)
C12B—C11B—H11D	120.0	C24—C25—H25	120.1
H11C—C11B—H11D	120.0	C26—C25—H25	120.1
C13—C12B—C11B	134.0 (11)	C27—C26—C25	122.34 (15)
C13—C12B—H12B	113.0	C27—C26—N3	120.08 (15)
C11B—C12B—H12B	113.0	C25—C26—N3	117.58 (15)
C12B—C13—C14	118.1 (3)	O10—C27—C26	126.64 (15)
C12A—C13—C14	116.4 (2)	O10—C27—C28	116.39 (15)
C12A—C13—H13A	108.2	C26—C27—C28	116.96 (14)
C14—C13—H13A	108.2	O9—C28—C29	125.80 (15)
C12A—C13—H13B	108.2	O9—C28—C27	113.62 (14)
C14—C13—H13B	108.2	C29—C28—C27	120.58 (16)
H13A—C13—H13B	107.3	C28—C29—C24	121.47 (15)
C12B—C13—H13C	107.8	C28—C29—H29	119.3
C14—C13—H13C	107.8	C24—C29—H29	119.3
C12B—C13—H13D	107.8	O9—C30—H30A	109.5
C14—C13—H13D	107.8	O9—C30—H30B	109.5
H13C—C13—H13D	107.1	H30A—C30—H30B	109.5
C15—C14—C19	118.49 (17)	O9—C30—H30C	109.5
C15—C14—C13	121.91 (18)	H30A—C30—H30C	109.5
C19—C14—C13	119.59 (18)	H30B—C30—H30C	109.5
C14—C15—C16	120.02 (17)	O12—N3—O11	121.95 (16)
C14—C15—H15	120.0	O12—N3—C26	119.16 (16)
C16—C15—H15	120.0	O11—N3—C26	118.88 (16)
C17—C16—C15	122.55 (16)	C28—O9—C30	117.87 (14)
C17—C16—N2	119.58 (16)	C27—O10—H10	102.6 (17)
C15—C16—N2	117.87 (16)		

C1—C2—C3—C4	−120.5 (3)	O6—C17—C18—C19	179.56 (15)
C2—C3—C4—C5	95.0 (3)	C16—C17—C18—C19	0.2 (2)
C2—C3—C4—C9	−84.5 (3)	O5—C18—C19—C14	179.73 (17)
C9—C4—C5—C6	0.1 (3)	C17—C18—C19—C14	0.1 (3)
C3—C4—C5—C6	−179.48 (18)	C15—C14—C19—C18	−0.1 (3)

C4—C5—C6—C7	−0.3 (3)	C13—C14—C19—C18	179.3 (2)
C4—C5—C6—N1	179.90 (16)	C17—C16—N2—O8	−173.68 (18)
C5—C6—C7—O2	−178.92 (16)	C15—C16—N2—O8	6.3 (3)
N1—C6—C7—O2	0.8 (3)	C17—C16—N2—O7	6.4 (3)
C5—C6—C7—C8	−0.3 (2)	C15—C16—N2—O7	−173.57 (17)
N1—C6—C7—C8	179.51 (14)	C19—C18—O5—C20	1.2 (3)
O2—C7—C8—O1	−0.1 (2)	C17—C18—O5—C20	−179.13 (16)
C6—C7—C8—O1	−178.89 (14)	C21B—C22B—C23—C24	−92.5 (19)
O2—C7—C8—C9	179.89 (15)	C21A—C22A—C23—C24	113.8 (7)
C6—C7—C8—C9	1.1 (2)	C22B—C23—C24—C25	2.5 (7)
O1—C8—C9—C4	178.59 (16)	C22A—C23—C24—C25	−45.2 (4)
C7—C8—C9—C4	−1.4 (3)	C22B—C23—C24—C29	−176.1 (7)
C5—C4—C9—C8	0.8 (3)	C22A—C23—C24—C29	136.1 (3)
C3—C4—C9—C8	−179.65 (18)	C29—C24—C25—C26	−0.2 (3)
C7—C6—N1—O4	−176.42 (16)	C23—C24—C25—C26	−178.83 (18)
C5—C6—N1—O4	3.4 (2)	C24—C25—C26—C27	−0.1 (3)
C7—C6—N1—O3	4.4 (3)	C24—C25—C26—N3	179.01 (15)
C5—C6—N1—O3	−175.79 (17)	C25—C26—C27—O10	−178.86 (15)
C9—C8—O1—C10	−2.6 (3)	N3—C26—C27—O10	2.0 (2)
C7—C8—O1—C10	177.44 (16)	C25—C26—C27—C28	0.4 (2)
C11B—C12B—C13—C14	100.3 (13)	N3—C26—C27—C28	−178.66 (14)
C11A—C12A—C13—C14	−105.3 (7)	O10—C27—C28—O9	−1.6 (2)
C12B—C13—C14—C15	175.2 (5)	C26—C27—C28—O9	179.02 (14)
C12A—C13—C14—C15	−132.1 (3)	O10—C27—C28—C29	178.92 (15)
C12B—C13—C14—C19	−4.2 (5)	C26—C27—C28—C29	−0.5 (2)
C12A—C13—C14—C19	48.6 (4)	O9—C28—C29—C24	−179.25 (16)
C19—C14—C15—C16	−0.1 (3)	C27—C28—C29—C24	0.1 (3)
C13—C14—C15—C16	−179.5 (2)	C25—C24—C29—C28	0.2 (3)
C14—C15—C16—C17	0.3 (3)	C23—C24—C29—C28	178.83 (19)
C14—C15—C16—N2	−179.68 (17)	C27—C26—N3—O12	−179.78 (16)
C15—C16—C17—O6	−179.69 (16)	C25—C26—N3—O12	1.1 (2)
N2—C16—C17—O6	0.3 (3)	C27—C26—N3—O11	1.1 (2)
C15—C16—C17—C18	−0.4 (2)	C25—C26—N3—O11	−178.04 (16)
N2—C16—C17—C18	179.66 (15)	C29—C28—O9—C30	−3.2 (3)
O6—C17—C18—O5	−0.1 (2)	C27—C28—O9—C30	177.35 (16)
C16—C17—C18—O5	−179.54 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O10—H10···O11	0.86 (3)	1.81 (3)	2.594 (2)	149 (2)
O6—H6···O7	0.83 (2)	1.83 (2)	2.584 (2)	152 (2)
O2—H2*O*···O3	0.91 (3)	1.78 (3)	2.587 (2)	146 (2)
C12*A*—H12*A*···O12	0.93	2.58	3.382 (4)	145
C12*B*—H12*B*···O3^{i}	0.93	2.56	3.325 (8)	140
C9—H9···O7^{ii}	0.93	2.59	3.394 (2)	145

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