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# Crystal structure and molecular docking study of (*E*)-2-{[(*E*)-2-hydroxy-5-methylbenzylidene]-hydrazinylidene}-1,2-diphenylethan-1-one

Sevgi Kansiz,[a]* Digdem Tatlidil,[b] Necmi Dege,[c] Feyzi Alkim Aktas,[d] Samir Osman Mohammed Al-Asbahy[e,f]* and Aysen Alaman Agar[b]

[a]Samsun University, Faculty of Engineering, Department of Fundamental Sciences, 55420, Samsun, Turkey, [b]Ondokuz Mayıs University, Faculty of Arts and Sciences, Department of Chemistry, 55139, Samsun, Turkey, [c]Ondokuz Mayıs University, Faculty of Arts and Sciences, Department of Physics, 55139, Samsun, Turkey, [d]Samsun University, Faculty of Engineering, Biomedical Engineering, 55420, Samsun, Turkey, [e]Ibb University, Science College, Department of Physics, Ibb, Yemen, and [f]Aljanad University for Science & Technology, Engineering College, Taiz, Yemen. *Correspondence e-mail: sevgi.kansiz@samsun.edu.tr, samirbas@gmail.com

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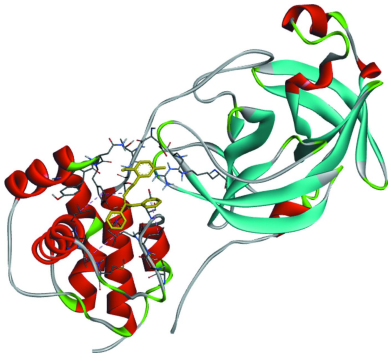
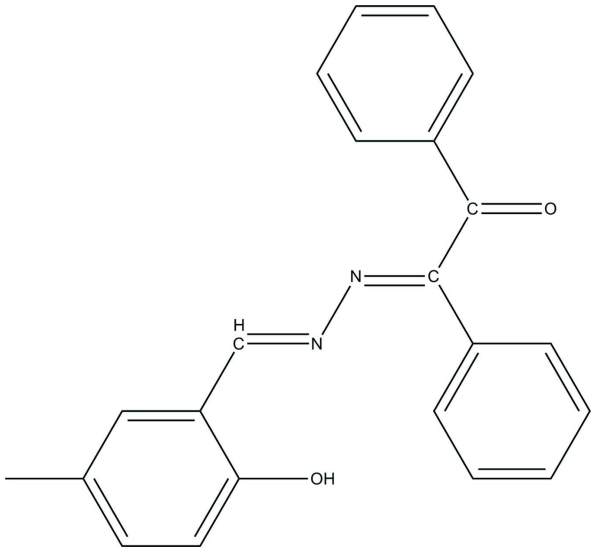
The title compound, $C_{22}H_{18}N_2O_2$, is a Schiff base that exists in the phenol–imine tautomeric form and adopts an *E* configuration with respect to the C=N bond. The molecular structure is stabilized by an O—H···N hydrogen bond, forming an *S*(6) ring motif. In the crystal, pairs of C—H···O hydrogen bonds link the molecules to form inversion dimers. Weak $\pi$–$\pi$ stacking interactions along the *a*-axis direction provide additional stabilization of the crystal structure. The molecule is non-planar, the aromatic ring of the benzaldehyde residue being nearly perpendicular to the phenyl and 4-methylphenol rings with dihedral angles of 88.78 (13) and 82.26 (14)°, respectively. A molecular docking study between the title molecule and the COVID-19 main protease (PDB ID: 6LU7) was performed, showing that it is a potential agent because of its affinity and ability to adhere to the active sites of the protein.

## 1. Chemical context

Schiff bases have wide applications interests as corrosion inhibitors (Antonijevic & Petrovic, 2008), biologically active materials (Al Zoubi, 2013) and thermostable systems (Destri *et al.*, 1998). The optical and semiconducting phenomena of the azomethine linkage group have been also widely investigated as a result of their photo-efficiency, with wavelengths depending on the chemical architecture of the Schiff-base molecules (Iwan & Sek, 2008). Schiff bases have significant importance in the development of metal complexes, because Schiff base ligands are potentially capable of forming stable complexes by coordination of metal ions *via* their oxygen and nitrogen donors (Ebrahimipour *et al.*, 2012). Hydrazine, hydrazone and hydrazide derivatives are relatively scarce in nature and have been isolated from plants, marine organisms and microorganisms. These compounds exhibit remarkable structural diversity and relevant biological activities (Le Goff & Ouazzani, 2014). Salicylaldehyde complexes with transition metals have worked as antimalarial and antileukemic agents (Scovill *et al.*, 1982). In this study, a new Schiff base with potential biological character, (*E*)-2-{[(*E*)-2-hydroxy-5-methylbenzylidene]hydrazineylidene}-1,2-diphenylethan-1-one, was obtained in crystalline form from the reaction of 2-hydroxy-5-methylbenzaldehyde with (*E*)-2-hydrazineylidene-



1,2-diphenylethan-1-one. We report here the synthesis, crystal and molecular structure of the title compound. We have also performed a molecular docking study to determine possible intermolecular interactions between the COVID-19 main protease (PDB ID: 6LU7) and the title compound.




## 2. Structural commentary

The asymmetric unit of the title structure contains one molecule (Fig. 1), which crystallizes in the phenol–imine tautomeric form with an *E* configuration for the imine functionality. The hydroxy H atom is involved in a strong intramolecular O—H···N hydrogen bond, forming an *S*(6) ring motif, which stabilizes the molecular structure. The dibenzylidene hydrazine unit is approximately planar with the dihedral angle formed by the two terminal phenyl rings of 7.62 (15)°. On the other hand, the molecule is non-planar, because the C1–C6 ring is nearly perpendicular to the C9–C14 and C16–C21 rings with dihedral angles of 88.78 (13) and 82.26 (14)°, respectively. The C17—O2, C15—N2 and C15—C16 bond lengths in the molecule are 1.359 (5), 1.287 (5), and 1.452 (5) Å, respectively. These results suggest single-bond character for C17—O2 and C15—C16 and double-bond character for the C15—N2 bond as expected for a phenol–imine structure (Kaştaş *et al.*, 2020). The bond lengths and angles in the title molecule agree reasonably well with those found in closely related structures (Bouchama *et al.*, 2015; Wieland *et al.*, 2011). Based on the refinement parameters, the tautomeric form of the compound is the phenol–imine form in which the tautomeric proton (H2) is located on the phenolic oxygen atom (O2). The distance of 2.650 (5) Å between the nitrogen and the oxygen atoms show that the molecule has a strong O—H···N intramolecular hydrogen bond, forming an *S*(6) ring motif.

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···N2 | 0.82 | 1.94 | 2.650 (5) | 145 |
| C3—H3···O2$^{i}$ | 0.93 | 2.54 | 3.434 (8) | 162 |

Symmetry code: (i) $-x+1, -y+2, -z+1$.

## 3. Supramolecular features

In the crystal, molecules are linked by pairs of C3—H3···O2 hydrogen bonds, forming inversion dimers with an $R_2^2(11)$ ring motif (Table 1 and Fig. 2). There are also weak $\pi$–$\pi$ interactions [$Cg2\cdots Cg3(-x, -y, -z)$ = 3.909 (2) Å; *Cg*2 and *Cg*3 are the centroids of the C9–C14 and C16–C21 rings, respectively] that stabilize the crystal structure, forming a three-dimensional network.

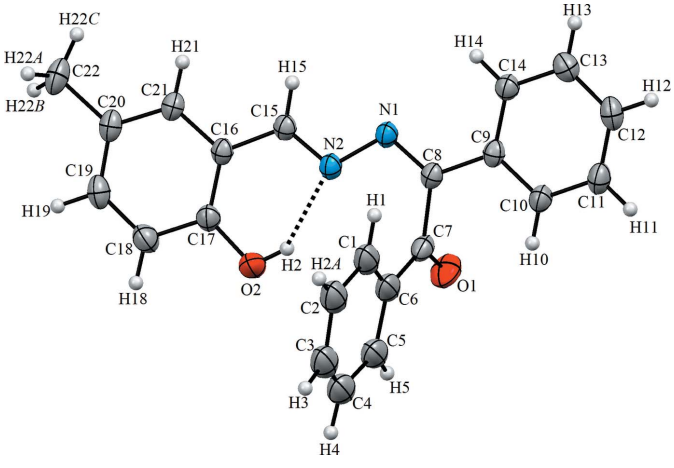



**Figure 1**
The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level. Dashed lines denote the intramolecular O—H···N hydrogen bonds forming an *S*(6) ring motif.

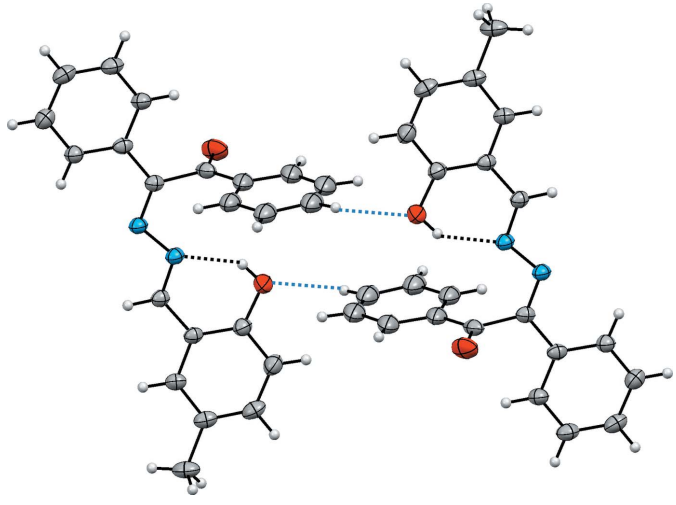


**Figure 2**
A view of the crystal packing of the title compound. Blue dashed lines denote the intermolecular C3—H3···O2 hydrogen bonds forming an inversion dimer (Table 1).

## 4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.42, update November 2020; Groom *et al.*, 2016) for the (benzylidenehydrazono)-1,2-diphenylethanone skeleton revealed 14 similar compounds. In MOZZEH01 (Marcel *et al.*, 2011), the C═N [1.291 (2) Å], N—N [1.414 (2) Å] and C═O bond lengths [1.235 (1) Å] are within the ranges observed for the title compound. The C7—C8 bond distance of 1.523 (2) Å corresponding to the value expected for a $Csp^2$—$Csp^2$ single bond, is slightly longer than observed for the title compound [1.472 (5) Å]. This bond length is shorter than in NOTZIH [1.528 (3) Å; Bouchama *et al.*, 2015]. In HULFAX (Elmacı *et al.*, 2015), the C15—N2 bond length [1.276 (4) Å] is typical of for an azomethine C═N bond and shorter than in the title compound [1.287 (5) Å]. In MOZZEH (Patra *et al.*, 2009) and MUBTUZ (Patra & Ng, 2009), the dimethylene hydrazine (—C═N—N═C—) units are approximately planar, the torsion angles around the N—N bond being 177.82 (12) and 162.2 (6)°, respectively. Although these values are comparable to the title compound, they are slightly smaller than 178.3 (3)°. In LOTKEN (Yahyaoui *et al.*, 2019), N—N (hydrazone) distances are within the range of typical single bond [1.398 (6)–1.4077 (16) Å and the C═N bonds in the hydrazone units are between 1.2893 (19) and 1.3014 (18) Å]. The torsion angles involving the —N═C— vary between −171.02 and −179.90°. All these values are similar to those observed in the title compound.

## 5. Molecular docking study

Molecular docking is a crucial method for investigating the interaction between small molecules and macromolecules. Intermolecular contacts that occur between a ligand and a protein are evaluated by molecular docking. In summary, this method is one of the major approaches to estimate the binding area where the ligand connects with the protein. In this study, *AutoDockVina* (Trott & Olson, 2010) was utilized for predicting binding sites between the title molecule and 6LU7. 6LU7 is a main protease of COVID-19, and can be efficient for drug design to treat ailments (Jin *et al.*, 2020). The three-dimensional structure of 6LU7 was received from the Protein Data Bank (PDB). Before the computation, the protein must be prepared for efficient insertion. Therefore, all water molecules and ligands were removed from protein active sites. LYS102, VAL104, GLN110, THR111, ASN151, ASP153 and SER158 were defined as active areas. According to these active sites, grid box dimensions were determined to be 100 × 100 × 95 Å. In addition, '*x*, *y*, *z*' centers were adjusted to be −20.378, 27.848, 69.124, respectively, and then the 6LU7 protein was saved in PDBQT format for calculations. In the next step of the experiment, rotatable angles for coupling structures were identified and recorded in PDBQT format. *Discovery Studio Visualizer* (Biovia, 2017) was used for observations and preparations. All docking calculations were computed with *AutoDockVina*. Twenty variable adherences were decided by *AutoDockVina* for the ligands connected to the receptor of the protein. The best affinity energy was observed in the first calculation, of −7.2 kcal mol$^{-1}$. The

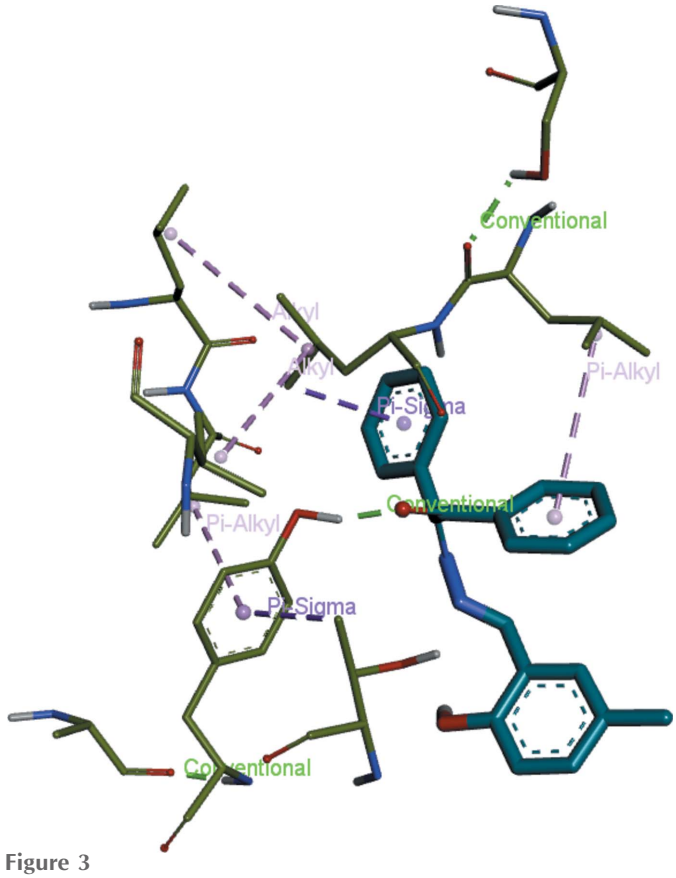



**Figure 3**
Three-dimensional visualization of the intermolecular interactions for the best binding pose of the title compound docking with 6LU7.

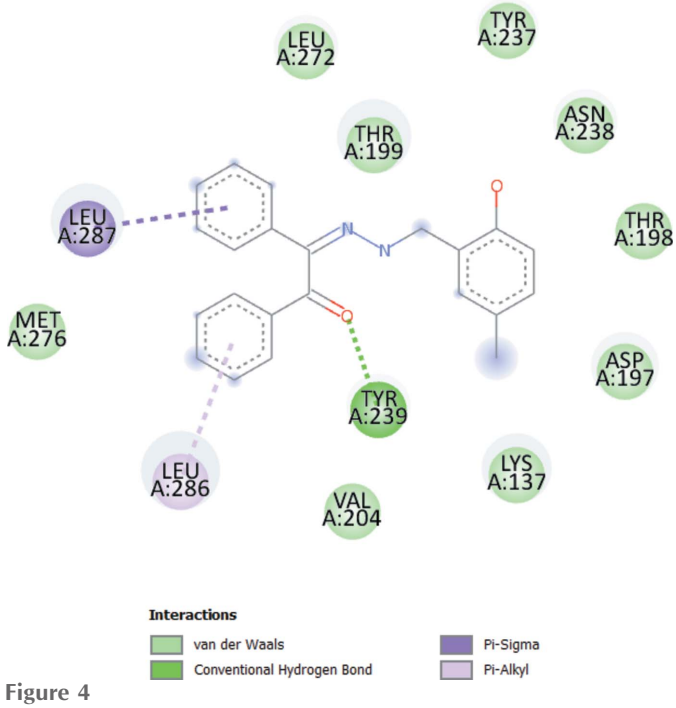



**Figure 4**
Two-dimensional visuals of the intermolecular interactions for the best binding pose of the title compound docking with 6LU7.

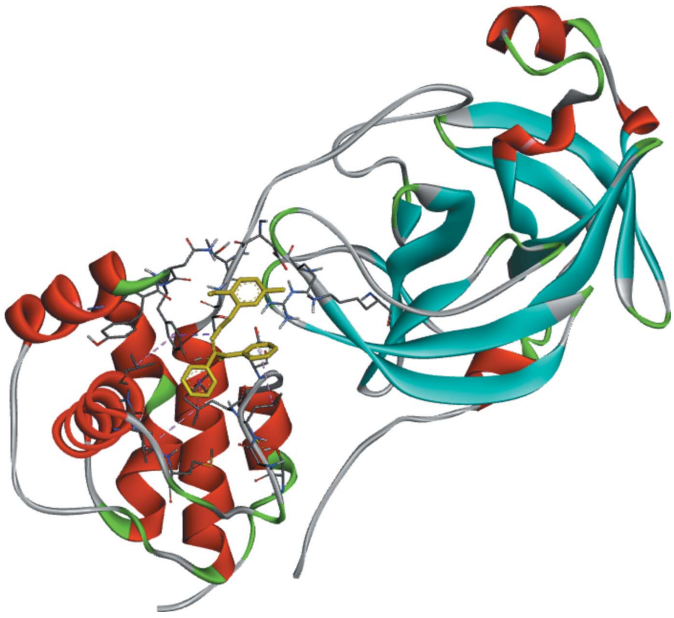


**Figure 5**
Three-dimensional conformation of the complex of the title compound with 6LU7.

bonding type of interaction is demonstrated in Fig. 3. The 2D and 3D visuals of the intermolecular interactions for the best binding pose of the title compound docked into macromolecule 6LU7 can be seen in Fig. 4. In addition, the docking conformation is shown in Fig. 5. As a consequence, the title compound could be a potential molecule for drug design to treat severe acute respiratory syndrome resulting from the novel corona virus SARS CoV2 because of its affinity and ability suitable to adhere to active sites of the protein.

## 6. Synthesis and crystallization

(*E*)-2-{[(*E*)-2-Hydroxy-5-methylbenzylidene]hydrazineylidene}-1,2-diphenylethan-1-one was prepared by refluxing a mixture of a solution containing 2-hydroxy-5-methylbenzaldehyde (0.02 mmol) in ethanol (20 mL) and a solution containing (*E*)-2-hydrazineylidene-1,2-diphenylethan-1-one (0.02 mmol) in ethanol (20 mL). The reaction mixture was stirred for 5 h under reflux. The obtained crystalline material was washed with ethanol and dried at room temperature. Single crystals of the title compound for X-ray analysis were obtained by slow evaporation of an ethanol solution.

## 7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The O-bound H atom was located in a difference-Fourier map and refined with O—H = 0.82 Å and $U_{iso}$(H) = 1.5$U_{eq}$(O). The C-bound H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 and $U_{iso}$(H) = 1.2$U_{eq}$(C) for aromatic H atoms, and with C—H = 0.96 Å and $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl H atoms.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{22}H_{18}N_2O_2$ |
| $M_r$ | 342.38 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 296 |
| $a$, $b$, $c$ (Å) | 7.4089 (6), 11.4544 (14), 21.9491 (17) |
| $\beta$ (°) | 97.814 (6) |
| $V$ (Å$^3$) | 1845.4 (3) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.08 |
| Crystal size (mm) | 0.71 × 0.49 × 0.21 |
| | |
| Data collection | |
| Diffractometer | Stoe *IPDS* 2 |
| Absorption correction | Integration (*X-RED32*; Stoe & Cie, 2002) |
| $T_{min}$, $T_{max}$ | 0.952, 0.987 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 8376, 3262, 1801 |
| $R_{int}$ | 0.037 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.596 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.085, 0.287, 1.11 |
| No. of reflections | 3262 |
| No. of parameters | 237 |
| No. of restraints | 84 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.50, −0.45 |

Computer programs: *X-AREA* and *X-RED* (Stoe & Cie, 2002), *SHELXT2018/3* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020) and *WinGX* (Farrugia, 2012).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Author contributions are as follows. Conceptualization, SK, ND and SOMA; synthesis, DT and AAA; writing (review and editing of the manuscript) SK, FAA and DT; formal analysis, SK and ND; crystal-structure determination, ND; validation, SK, ND and AAA, project administration, SK and SOMA.

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# supporting information

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## Crystal structure and molecular docking study of (*E*)-2-{[(*E*)-2-hydroxy-5-methylbenzylidene]hydrazinylidene}-1,2-diphenylethan-1-one

**Sevgi Kansiz, Digdem Tatlidil, Necmi Dege, Feyzi Alkim Aktas, Samir Osman Mohammed Al-Asbahy and Aysen Alaman Agar**

### Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXT2018/3* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

**(*E*)-2-{[(*E*)-2-Hydroxy-5-methylbenzylidene]hydrazinylidene}-1,2-diphenylethan-1-one**

*Crystal data*

$C_{22}H_{18}N_2O_2$
$M_r$ = 342.38
Monoclinic, $P2_1/c$
$a$ = 7.4089 (6) Å
$b$ = 11.4544 (14) Å
$c$ = 21.9491 (17) Å
$\beta$ = 97.814 (6)°
$V$ = 1845.4 (3) Å$^3$
$Z$ = 4
$F(000)$ = 720
$D_x$ = 1.232 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 7807 reflections
$\theta$ = 1.8–29.1°
$\mu$ = 0.08 mm$^{-1}$
$T$ = 296 K
Prism, orange
0.71 × 0.49 × 0.21 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)
$T_{min}$ = 0.952, $T_{max}$ = 0.987
8376 measured reflections
3262 independent reflections
1801 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.037
$\theta_{max}$ = 25.1°, $\theta_{min}$ = 1.9°
$h$ = −7→8
$k$ = −13→12
$l$ = −26→26

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.085
$wR(F^2)$ = 0.287
$S$ = 1.11
3262 reflections
237 parameters
84 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1388P)^2 + 0.7696P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.50 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.45 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N2 | 0.8073 (5) | 0.5988 (3) | 0.50905 (13) | 0.0632 (9) |
| N1 | 0.7264 (5) | 0.5098 (3) | 0.47015 (13) | 0.0650 (9) |
| O2 | 0.9055 (6) | 0.8135 (3) | 0.54443 (14) | 0.0968 (12) |
| H2 | 0.866243 | 0.762928 | 0.519659 | 0.145* |
| O1 | 0.8305 (6) | 0.6961 (4) | 0.37321 (16) | 0.1135 (14) |
| C16 | 0.9521 (5) | 0.6438 (4) | 0.61058 (16) | 0.0587 (10) |
| C9 | 0.5718 (5) | 0.4721 (4) | 0.36834 (16) | 0.0604 (10) |
| C15 | 0.8727 (5) | 0.5634 (4) | 0.56320 (16) | 0.0605 (10) |
| H15 | 0.869084 | 0.484218 | 0.572232 | 0.073* |
| C8 | 0.6674 (5) | 0.5488 (3) | 0.41603 (16) | 0.0587 (10) |
| C21 | 1.0127 (6) | 0.6008 (4) | 0.66984 (15) | 0.0647 (11) |
| H21 | 1.008655 | 0.520813 | 0.676726 | 0.078* |
| C14 | 0.5475 (6) | 0.3537 (4) | 0.37911 (18) | 0.0701 (12) |
| H14 | 0.592287 | 0.321938 | 0.417175 | 0.084* |
| C20 | 1.0782 (6) | 0.6731 (5) | 0.71822 (17) | 0.0722 (12) |
| C7 | 0.7011 (7) | 0.6748 (4) | 0.39951 (17) | 0.0729 (12) |
| C17 | 0.9626 (6) | 0.7633 (4) | 0.59982 (19) | 0.0704 (12) |
| C10 | 0.5008 (6) | 0.5172 (4) | 0.31111 (17) | 0.0740 (12) |
| H10 | 0.513452 | 0.596328 | 0.303221 | 0.089* |
| C13 | 0.4574 (7) | 0.2826 (4) | 0.3337 (2) | 0.0806 (13) |
| H13 | 0.442179 | 0.203596 | 0.341196 | 0.097* |
| C12 | 0.3900 (7) | 0.3298 (5) | 0.2771 (2) | 0.0809 (14) |
| H12 | 0.329630 | 0.282317 | 0.246532 | 0.097* |
| C19 | 1.0877 (7) | 0.7906 (5) | 0.7057 (2) | 0.0844 (15) |
| H19 | 1.134050 | 0.840615 | 0.737345 | 0.101* |
| C11 | 0.4120 (7) | 0.4460 (5) | 0.26600 (19) | 0.0832 (14) |
| H11 | 0.366915 | 0.477135 | 0.227836 | 0.100* |
| C18 | 1.0317 (7) | 0.8376 (4) | 0.6481 (2) | 0.0874 (14) |
| H18 | 1.039779 | 0.917581 | 0.641626 | 0.105* |
| C22 | 1.1374 (8) | 0.6258 (6) | 0.78220 (19) | 0.0994 (18) |
| H22A | 1.044825 | 0.641737 | 0.807719 | 0.149* |
| H22B | 1.249168 | 0.662613 | 0.799408 | 0.149* |
| H22C | 1.155645 | 0.543006 | 0.780046 | 0.149* |
| C3 | 0.3300 (11) | 0.9322 (6) | 0.4407 (3) | 0.1213 (13) |
| H3 | 0.245666 | 0.989489 | 0.447121 | 0.146* |
| C2 | 0.2919 (10) | 0.8188 (5) | 0.4544 (3) | 0.1180 (12) |
| H2A | 0.188797 | 0.799054 | 0.472040 | 0.142* |
| C6 | 0.5724 (10) | 0.7645 (5) | 0.4144 (2) | 0.1031 (12) |
| C1 | 0.4177 (10) | 0.7334 (5) | 0.4404 (2) | 0.1081 (12) |

| | | | | |
|---|---|---|---|---|
| H1 | 0.396996 | 0.655268 | 0.448600 | 0.130* |
| C5 | 0.6069 (10) | 0.8824 (5) | 0.4039 (2) | 0.1137 (12) |
| H5 | 0.710653 | 0.905524 | 0.387565 | 0.136* |
| C4 | 0.4790 (11) | 0.9644 (6) | 0.4189 (2) | 0.1204 (13) |
| H4 | 0.500278 | 1.043459 | 0.413196 | 0.144* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N2 | 0.066 (2) | 0.068 (2) | 0.0531 (17) | 0.0089 (17) | −0.0017 (14) | −0.0065 (15) |
| N1 | 0.073 (2) | 0.069 (2) | 0.0499 (17) | 0.0104 (17) | −0.0016 (15) | −0.0052 (15) |
| O2 | 0.137 (3) | 0.0681 (19) | 0.076 (2) | 0.010 (2) | −0.0158 (19) | 0.0019 (16) |
| O1 | 0.123 (3) | 0.137 (3) | 0.086 (2) | −0.046 (3) | 0.033 (2) | 0.010 (2) |
| C16 | 0.054 (2) | 0.070 (3) | 0.0515 (19) | 0.0107 (19) | 0.0062 (16) | −0.0051 (17) |
| C9 | 0.054 (2) | 0.076 (3) | 0.0511 (19) | 0.007 (2) | 0.0080 (16) | −0.0057 (18) |
| C15 | 0.059 (2) | 0.067 (2) | 0.055 (2) | 0.0119 (19) | 0.0068 (17) | −0.0041 (17) |
| C8 | 0.055 (2) | 0.071 (3) | 0.0500 (19) | 0.0077 (19) | 0.0063 (16) | 0.0002 (17) |
| C21 | 0.061 (3) | 0.083 (3) | 0.050 (2) | 0.001 (2) | 0.0076 (17) | −0.0030 (18) |
| C14 | 0.077 (3) | 0.073 (3) | 0.058 (2) | 0.006 (2) | 0.004 (2) | −0.002 (2) |
| C20 | 0.058 (3) | 0.103 (4) | 0.054 (2) | 0.003 (2) | 0.0051 (18) | −0.011 (2) |
| C7 | 0.084 (3) | 0.085 (3) | 0.047 (2) | −0.020 (3) | 0.001 (2) | 0.0051 (19) |
| C17 | 0.078 (3) | 0.070 (3) | 0.061 (2) | 0.012 (2) | 0.000 (2) | −0.007 (2) |
| C10 | 0.087 (3) | 0.082 (3) | 0.051 (2) | 0.003 (2) | 0.0026 (19) | −0.003 (2) |
| C13 | 0.083 (3) | 0.076 (3) | 0.082 (3) | 0.002 (2) | 0.010 (2) | −0.016 (2) |
| C12 | 0.073 (3) | 0.101 (4) | 0.068 (3) | 0.006 (3) | 0.003 (2) | −0.029 (3) |
| C19 | 0.077 (3) | 0.105 (4) | 0.068 (3) | −0.005 (3) | −0.001 (2) | −0.026 (3) |
| C11 | 0.087 (4) | 0.105 (4) | 0.054 (2) | 0.008 (3) | −0.002 (2) | −0.009 (2) |
| C18 | 0.098 (4) | 0.074 (3) | 0.087 (3) | 0.001 (3) | 0.001 (3) | −0.016 (2) |
| C22 | 0.097 (4) | 0.147 (5) | 0.052 (2) | −0.010 (3) | 0.000 (2) | −0.007 (3) |
| C3 | 0.163 (3) | 0.104 (2) | 0.0846 (19) | 0.038 (2) | −0.0283 (19) | −0.0113 (17) |
| C2 | 0.157 (3) | 0.105 (2) | 0.0817 (18) | 0.036 (2) | −0.0232 (18) | −0.0129 (17) |
| C6 | 0.153 (3) | 0.0836 (19) | 0.0617 (17) | 0.024 (2) | −0.0240 (17) | −0.0021 (16) |
| C1 | 0.150 (3) | 0.0933 (19) | 0.0707 (18) | 0.033 (2) | −0.0220 (18) | −0.0100 (16) |
| C5 | 0.165 (3) | 0.0914 (19) | 0.0731 (18) | 0.022 (2) | −0.0253 (18) | 0.0004 (16) |
| C4 | 0.169 (3) | 0.097 (2) | 0.0819 (19) | 0.028 (2) | −0.0292 (19) | −0.0017 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N2—C15 | 1.287 (5) | C10—H10 | 0.9300 |
| N2—N1 | 1.411 (4) | C13—C12 | 1.384 (6) |
| N1—C8 | 1.289 (4) | C13—H13 | 0.9300 |
| O2—C17 | 1.359 (5) | C12—C11 | 1.366 (7) |
| O2—H2 | 0.8200 | C12—H12 | 0.9300 |
| O1—C7 | 1.209 (5) | C19—C18 | 1.384 (7) |
| C16—C17 | 1.393 (6) | C19—H19 | 0.9300 |
| C16—C21 | 1.406 (5) | C11—H11 | 0.9300 |
| C16—C15 | 1.452 (5) | C18—H18 | 0.9300 |
| C9—C14 | 1.393 (6) | C22—H22A | 0.9600 |

| | | | |
|---|---|---|---|
| C9—C10 | 1.393 (5) | C22—H22B | 0.9600 |
| C9—C8 | 1.472 (5) | C22—H22C | 0.9600 |
| C15—H15 | 0.9300 | C3—C4 | 1.314 (9) |
| C8—C7 | 1.518 (6) | C3—C2 | 1.372 (9) |
| C21—C20 | 1.382 (6) | C3—H3 | 0.9300 |
| C21—H21 | 0.9300 | C2—C1 | 1.413 (8) |
| C14—C13 | 1.386 (6) | C2—H2A | 0.9300 |
| C14—H14 | 0.9300 | C6—C1 | 1.393 (9) |
| C20—C19 | 1.377 (7) | C6—C5 | 1.399 (8) |
| C20—C22 | 1.514 (6) | C1—H1 | 0.9300 |
| C7—C6 | 1.469 (7) | C5—C4 | 1.405 (9) |
| C17—C18 | 1.402 (6) | C5—H5 | 0.9300 |
| C10—C11 | 1.379 (6) | C4—H4 | 0.9300 |

| | | | |
|---|---|---|---|
| C15—N2—N1 | 114.0 (3) | C11—C12—C13 | 120.2 (4) |
| C8—N1—N2 | 111.7 (3) | C11—C12—H12 | 119.9 |
| C17—O2—H2 | 109.5 | C13—C12—H12 | 119.9 |
| C17—C16—C21 | 118.9 (4) | C20—C19—C18 | 123.1 (4) |
| C17—C16—C15 | 121.9 (3) | C20—C19—H19 | 118.5 |
| C21—C16—C15 | 119.2 (4) | C18—C19—H19 | 118.5 |
| C14—C9—C10 | 118.2 (4) | C12—C11—C10 | 120.3 (4) |
| C14—C9—C8 | 121.4 (3) | C12—C11—H11 | 119.8 |
| C10—C9—C8 | 120.4 (4) | C10—C11—H11 | 119.8 |
| N2—C15—C16 | 121.8 (4) | C19—C18—C17 | 119.1 (5) |
| N2—C15—H15 | 119.1 | C19—C18—H18 | 120.4 |
| C16—C15—H15 | 119.1 | C17—C18—H18 | 120.4 |
| N1—C8—C9 | 121.2 (4) | C20—C22—H22A | 109.5 |
| N1—C8—C7 | 120.3 (3) | C20—C22—H22B | 109.5 |
| C9—C8—C7 | 118.5 (3) | H22A—C22—H22B | 109.5 |
| C20—C21—C16 | 122.4 (4) | C20—C22—H22C | 109.5 |
| C20—C21—H21 | 118.8 | H22A—C22—H22C | 109.5 |
| C16—C21—H21 | 118.8 | H22B—C22—H22C | 109.5 |
| C13—C14—C9 | 120.7 (4) | C4—C3—C2 | 123.6 (7) |
| C13—C14—H14 | 119.7 | C4—C3—H3 | 118.2 |
| C9—C14—H14 | 119.7 | C2—C3—H3 | 118.2 |
| C19—C20—C21 | 117.0 (4) | C3—C2—C1 | 116.5 (7) |
| C19—C20—C22 | 121.3 (4) | C3—C2—H2A | 121.8 |
| C21—C20—C22 | 121.7 (5) | C1—C2—H2A | 121.8 |
| O1—C7—C6 | 123.2 (5) | C1—C6—C5 | 119.6 (6) |
| O1—C7—C8 | 118.2 (5) | C1—C6—C7 | 120.5 (5) |
| C6—C7—C8 | 118.6 (4) | C5—C6—C7 | 119.9 (7) |
| O2—C17—C16 | 123.4 (4) | C6—C1—C2 | 121.0 (6) |
| O2—C17—C18 | 117.2 (4) | C6—C1—H1 | 119.5 |
| C16—C17—C18 | 119.5 (4) | C2—C1—H1 | 119.5 |
| C11—C10—C9 | 120.9 (5) | C6—C5—C4 | 117.6 (7) |
| C11—C10—H10 | 119.6 | C6—C5—H5 | 121.2 |
| C9—C10—H10 | 119.6 | C4—C5—H5 | 121.2 |
| C12—C13—C14 | 119.7 (5) | C3—C4—C5 | 121.6 (7) |

| | | | |
|---|---|---|---|
| C12—C13—H13 | 120.1 | C3—C4—H4 | 119.2 |
| C14—C13—H13 | 120.1 | C5—C4—H4 | 119.2 |
| | | | |
| C15—N2—N1—C8 | −178.3 (3) | C14—C9—C10—C11 | −1.2 (6) |
| N1—N2—C15—C16 | −176.5 (3) | C8—C9—C10—C11 | 179.6 (4) |
| C17—C16—C15—N2 | −0.4 (6) | C9—C14—C13—C12 | −0.3 (7) |
| C21—C16—C15—N2 | 176.7 (4) | C14—C13—C12—C11 | −0.1 (7) |
| N2—N1—C8—C9 | −177.8 (3) | C21—C20—C19—C18 | 1.5 (7) |
| N2—N1—C8—C7 | 4.3 (5) | C22—C20—C19—C18 | −178.7 (5) |
| C14—C9—C8—N1 | −2.4 (6) | C13—C12—C11—C10 | −0.2 (7) |
| C10—C9—C8—N1 | 176.7 (4) | C9—C10—C11—C12 | 0.9 (7) |
| C14—C9—C8—C7 | 175.5 (4) | C20—C19—C18—C17 | −0.4 (8) |
| C10—C9—C8—C7 | −5.3 (6) | O2—C17—C18—C19 | 178.6 (4) |
| C17—C16—C21—C20 | 1.7 (6) | C16—C17—C18—C19 | −0.1 (7) |
| C15—C16—C21—C20 | −175.5 (4) | C4—C3—C2—C1 | −3.8 (8) |
| C10—C9—C14—C13 | 0.9 (6) | O1—C7—C6—C1 | 174.8 (4) |
| C8—C9—C14—C13 | −179.9 (4) | C8—C7—C6—C1 | −3.2 (6) |
| C16—C21—C20—C19 | −2.1 (6) | O1—C7—C6—C5 | −6.6 (7) |
| C16—C21—C20—C22 | 178.0 (4) | C8—C7—C6—C5 | 175.3 (4) |
| N1—C8—C7—O1 | 96.0 (5) | C5—C6—C1—C2 | 2.1 (7) |
| C9—C8—C7—O1 | −81.9 (5) | C7—C6—C1—C2 | −179.4 (4) |
| N1—C8—C7—C6 | −85.9 (5) | C3—C2—C1—C6 | 0.5 (7) |
| C9—C8—C7—C6 | 96.2 (4) | C1—C6—C5—C4 | −1.6 (7) |
| C21—C16—C17—O2 | −179.2 (4) | C7—C6—C5—C4 | 179.9 (4) |
| C15—C16—C17—O2 | −2.1 (7) | C2—C3—C4—C5 | 4.4 (9) |
| C21—C16—C17—C18 | −0.5 (6) | C6—C5—C4—C3 | −1.5 (8) |
| C15—C16—C17—C18 | 176.6 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···N2 | 0.82 | 1.94 | 2.650 (5) | 145 |
| C3—H3···O2$^{i}$ | 0.93 | 2.54 | 3.434 (8) | 162 |

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.