Crystal structure of racemic \((R/S,E)-2-(4-hydroxyphenyl)-4-(2-phenylhydrazin-1-ylidene)chromane-5,7-diol ethanol monosolvate\)

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The crystal structure of racemic \((R/S,E)-2-(4-hydroxyphenyl)-4-(2-phenylhydrazin-1-ylidene)chromane-5,7-diol ethanol monosolvate, \(\text{C}_{21}\text{H}_{18}\text{N}_{2}\text{O}_{4}/\text{C}_{1}\) - \(\text{C}_{2}\) \(\text{H}_{6}\)\(\text{O}\)\), in a centrosymmetric lattice is reported. The two racemates occupy the same position in the asymmetric unit – a disordered mixed enantiomeric structure. Hydrogen bonds of the type O—H•••C(1) in addition to typical C—H•••O, O—H•••O and O—H•••N are identified. A positional disorder is seen in the solvent molecule (ethanol) as well. The phenylhydrazone group is nearly coplanar with the chromane ring system [dihedral angle = 15.5 (1)\(^\circ\)], while the 4-hydroxyphenyl ring is perpendicular [dihedral angle = 87.2 (1)\(^\circ\)] to the chromane. The pyran ring has an envelope pucker \([Q = 0.363 (3) \text{ Å}, \theta = 57.6 (3)\] ; and for the enantiomer: \(Q = 0.364 (3) \text{ Å}, \theta = 127.4 (4)\]\)\).

1. Chemical context

Naringenin is a naturally occurring flavanone compound found in citrus fruits, bergamot and tomatoes (Cai \textit{et al.}, 2004). It has been reported to have a wide range of biological activities, including anti-viral, anti-inflammatory and anti-aging properties (Heim \textit{et al.}, 2002). Due to its inherent medicinal properties, derivatives of naringenin have also been synthesized and studied as potential treatments for disease. The title compound, \((R/S,E)-2-(4-hydroxyphenyl)-4-(2-phenylhydrazin-1-ylidene)chromane-5,7-diol\), is a hydrazone naringenin derivative that has been reported to induce apoptosis in human cervical cancer cells (Kim \textit{et al.}, 2012). Its close structural analog, 5-hydroxy-7,4'-diacetoxyflavanone-N-phenylhydrazone, exhibits cytotoxicity against non-small-cell lung cancer cells (Bak \textit{et al.}, 2011). Despite their biological value, crystal structures have not been reported to date of any hydrazone derivatives of naringenin. Herein, we report the first crystal structure of a hydrazone derivative of naringenin.
1.1. Structural commentary

The title compound along with the solvent (ethanol) molecule in a 1:1 ratio, yielded a disordered mixed enantiomeric crystal in a centrosymmetric lattice ($P1$, Fig. 1). The structure was solved and refined in $P1$, and a distorted structure was found. The asymmetric unit has two racemates occupying the same position in a ratio of 0.562 (6):0.438 (6). Enantiomeric structures in centrosymmetric lattices have been discussed by Flack (2003). The title molecule has three phenyl rings, one of which is fused with a pyran ring. The molecule in the asymmetric unit is a superposition of the two enantiomers in the ratio of 0.562 (6):0.438 (6). The phenylhydrazone group is nearly coplanar with the chromane ring system (dihedral angle = 15.5 (1)$^\circ$), while the the 4-hydroxyphenyl ring is perpendicular (dihedral angle = 87.2 (1)$^\circ$) to the chromane. The pyran ring has an envelope pucker ($Q = 0.363 (3)$ Å, $\theta = 57.6 (3)^\circ$); and for the enantiomer: $Q = 0.364 (3)$ Å, $\theta = 127.4 (4)^\circ$). An intramolecular O—H···N hydrogen bond exists between one of the hydroxy groups on the chromane ring and the nitrogen of the hydrazone group (Table 1). The carbon–nitrogen double bond [$N1C7 = 1.295 (2)$ Å] exists as the $E$ isomer.

1.2. Supramolecular features

In the crystal, O—H···C(π) type hydrogen-bond interactions between the solvent ethanol and phenyl ring are observed (Table 1, Fig. 2). The phenyl ring is expected to have a partial negative charge because of the two nitrogen atoms (known electron-releasing groups) just before the phenyl ring (Stewart, 1985). A database analysis of such interactions was

![Figure 1](image1.png)

**Figure 1**
Displacement ellipsoid drawing at 50% probability level of the asymmetric unit showing the superposition of two enantiomers in the asymmetric unit. The disorder in the solvent (ethanol) molecule is resolved here, shown in two partial-occupancy locations.

![Figure 2](image2.png)

**Figure 2**
Crystal packing diagram showing intramolecular O—H···N and intermolecular O—H···O, C—H···O and (O—H···C(π)) hydrogen bonds, as well as extensive π···π stacking interactions.

### Table 1
Hydrogen-bond geometry (Å, $^\circ$).

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O5A—H5A···C17 | 0.82 | 2.56 | 3.363 (15) | 166 |
| O5A—H5A···C18 | 0.82 | 2.47 | 3.263 (16) | 162 |
| O5B—H5B···C19 | 0.82 | 2.59 | 3.405 (11) | 173 |
| O1—H1···O5A$^a$ | 0.82 | 1.79 | 2.590 (12) | 166 |
| O1—H1···O5B$^b$ | 0.82 | 1.90 | 2.709 (8) | 170 |
| C8—H8B···O4B$^c$ | 0.97 | 2.49 | 3.440 (17) | 168 |
| O4A—H4A···O1$^d$ | 0.82 | 1.89 | 2.677 (13) | 160 |
| C9B—H9B···O2$^e$ | 0.98 | 2.39 | 3.347 (5) | 165 |
| C2—H2···N1 | 0.82 | 1.87 | 2.5975 (18) | 147 |

Symmetry codes: (i) $x, y+1, z+1$; (ii) $x-x+1, y+1, z+2$; (iii) $x, y+1, z+1$.
reported by Viswamitra et al. (1993). The structure also has the not-so-rare C—H⋯O, O—H⋯O and O—H⋯N type hydrogen bonds. Extensive π–π stacking interactions [centroid–centroid distances in the range 4.223 (7) to 4.599 (5) Å] along the [1\bar{1}] direction between the planar cores of neighboring molecules further stabilize the lattice (Fig. 2).

2. Database survey
A structure search was performed in Scifinder and Reaxys. A text search (‘flavanone’ and ‘chroman-4-ylidene’ and ‘di-hydroxochromen-4-phenylhydrazone’) was performed in the Cambridge Structural Database (Groom et al., 2016; accessed January 2022). To date, no crystal structures have been reported for a hydrazone derivative of naringenin, including the two flavonones mentioned in the Chemical context section. The most similar structures for which crystal data have been reported include acyl hydrazone derivatives of 2-phenyl-

3. Synthesis and crystallization
The title compound was synthesized according to a previously reported procedure (Bak et al., 2011).

4. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 2. The superposition of two enantiomers in the asymmetric unit, and the disorder in the solvent (ethanol molecule) necessitated 183 constraints. The hydrogen atoms were placed in their geometrically calculated positions and their coordinates refined using the riding model. Idealized Me of the ethanol molecule were refined as rotating group(s): C22 and C22B (H22A through F) and its idealized tetrahedral OH refined as a rotating group: O5A and O5B

| Crystal data | Chemical formula | C2\textsubscript{1}H\textsubscript{18}N\textsubscript{2}O\textsubscript{4}C\textsubscript{2}H\textsubscript{6}O |
|-------------|-----------------|-------------------------------------------------|
| System, space group | Triclinic, P\textbar{\bar{T}} |  |
| Temperature (K) | 293 |  |
| Parameters | a, b, c (Å) | 9.4329 (3), 10.9974 (4), 11.9310 (3) |
| α, β, γ (°) | 115.244 (3), 93.939 (2), 104.180 (3) |
| Z | 2 |  |
| Radiation type | Cu Kα | 0.74 |
| μ (mm\textsuperscript{-1}) | 0.2 × 0.19 × 0.13 |  |
| Crystal size (mm) | 0.2 |  |
| Data collection | Rigaku Oxford Diffraction, Synergy Custom system, HyPix-Arc 150 |
| Absorption correction | Gaussian (CrysAlis PRO; Rigaku OD, 2021) |
| T\textsubscript{min}, T\textsubscript{max} | 0.638, 1.000 |  |
| No. of measured, independent and observed [F > 2σ(F)] reflections | 14477, 4067, 3077 |  |
| R\textsubscript{int} | 0.020 |  |
| (sinθ/λ)\textsubscript{max} (Å\textsuperscript{-1}) | 0.624 |  |
| Refinement | R[F\textsuperscript{2} > 2σ(F\textsuperscript{2})], wR(F\textsuperscript{2}) | 0.049, 0.161, 1.07 |
| No. of reflections | 4067 |  |
| No. of parameters | 381 |  |
| No. of restraints | 183 |  |
| H-atom treatment | H-atom parameters constrained |  |
| Δρ\textsubscript{max}, Δρ\textsubscript{min} (e Å\textsuperscript{-3}) | 0.24, −0.17 |  |

Computer programs: CrysAlis PRO (Rigaku OD, 2021), SHELXT2018/2 (Sheldrick, 2015a), SHELXL2018/5 (Sheldrick, 2015b), and OLEX2 (Dolomanov et al., 2009).

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2021); cell refinement: *CrysAlis PRO* (Rigaku OD, 2021); data reduction: *CrysAlis PRO* (Rigaku OD, 2021); program(s) used to solve structure: *SHELXT2018/2* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov et al., 2009); software used to prepare material for publication: *OLEX2* (Dolomanov et al., 2009).

(R/S,E)-2-(4-Hydroxyphenyl)-4-(2-phenylhydrazin-1-ylidene)chromane-5,7-dirol ethanol monosolvate

Crystal data

C21H18N2O4·C2H6O

Mr = 408.44

Triclinic, $P\overline{1}$

$a = 9.4329$ (3) Å

$b = 10.9974$ (4) Å

$c = 11.9310$ (3) Å

$\alpha = 115.244$ (3)°

$\beta = 93.939$ (2)°

$\gamma = 104.180$ (3)°

$V = 1064.01$ (6) Å³

$Z = 2$

$F(000) = 432$

$D_x = 1.275$ Mg m$^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 8481 reflections

$\theta = 4.1$–73.4°

$\mu = 0.74$ mm$^{-1}$

$T = 293$ K

Block, yellow

0.2 × 0.19 × 0.13 mm

Data collection

Rigaku Oxford Diffraction, Synergy Custom system, HyPix-Arc 150 diffractometer

Radiation source: Rotating-anode X-ray tube, Rigaku (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm$^{-1}$

$\omega$ scans

Absorption correction: gaussian

(CrysAlisPro; Rigaku OD, 2021)

$T_{\text{min}} = 0.638, T_{\text{max}} = 1.000$

14477 measured reflections

4067 independent reflections

3077 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 74.0^\circ, \theta_{\text{min}} = 4.2^\circ$

$h = -11\rightarrow11$

$k = -13\rightarrow13$

$l = -12\rightarrow14$

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.161$

$S = 1.07$

4067 reflections

381 parameters

183 restraints

Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F^2) + (0.0996P)^2 + 0.0482P]$

where $P = (F^2 + 2F^2)/3$
(Δ/σ)_{max} < 0.001
Δρ_{max} = 0.24 e Å\(^{-3}\)
Δρ_{min} = −0.17 e Å\(^{-3}\)

Extinction correction: SHELXL2018/3
(Sheldrick 2015b),
Fc^2 = kFc[1+0.001xFc^2\lambda^2/sin(2\theta)]^{1/4}
Extinction coefficient: 0.0070 (13)

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.

Refinement. In light of the crystal structure with two enantiomer molecules sharing the same site in the asymmetric unit of P1, we tried refining the structure non-centrosymmetric P1 space-group, and saw the disorder in the chiral carbon persist even there, in both the independent molecules.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å\(^2\))

|        | x          | y          | z          | U_{iso}*/U_{eq} | Occ. (<1) |
|--------|------------|------------|------------|----------------|-----------|
| O1     | 0.63930 (15) | 0.57227 (12) | 0.15536 (11) | 0.0762 (4) | 0.114*    |
| H1     | 0.702063 | 0.648464 | 0.176530 | 0.0818 (4) | 0.0818 (4) |
| O2     | 0.23838 (15) | 0.35612 (12) | 0.28729 (11) | 0.0708 (4) | 0.0708 (4) |
| H2     | 0.203745 | 0.375438 | 0.350612 | 0.123* | 0.123* |
| O3     | 0.56642 (13) | 0.82028 (11) | 0.57204 (10) | 0.0762 (4) | 0.0762 (4) |
| N1     | 0.20641 (15) | 0.48554 (13) | 0.51936 (12) | 0.0621 (4) | 0.0621 (4) |
| N2     | 0.12754 (15) | 0.49326 (14) | 0.61217 (12) | 0.0602 (4) | 0.0602 (4) |
| H2A    | 0.142115 | 0.572868 | 0.677760 | 0.079* | 0.079* |
| C1     | 0.56086 (19) | 0.58198 (16) | 0.24987 (14) | 0.0661 (4) | 0.0661 (4) |
| C2     | 0.43809 (19) | 0.42783 (16) | 0.22400 (15) | 0.075 (6) | 0.075 (6) |
| H2B    | 0.410135 | 0.389868 | 0.144550 | 0.079* | 0.079* |
| C3     | 0.35788 (19) | 0.42058 (16) | 0.31680 (15) | 0.066 (4) | 0.066 (4) |
| C4     | 0.39633 (17) | 0.58758 (15) | 0.43746 (14) | 0.055 (4) | 0.055 (4) |
| C5     | 0.52020 (17) | 0.70112 (15) | 0.45827 (14) | 0.056 (4) | 0.056 (4) |
| C6     | 0.60281 (18) | 0.69690 (15) | 0.36667 (14) | 0.062 (4) | 0.062 (4) |
| H6     | 0.684922 | 0.776159 | 0.383122 | 0.075* | 0.075* |
| C7     | 0.31398 (16) | 0.59550 (15) | 0.53822 (14) | 0.055 (4) | 0.055 (4) |
| C8     | 0.36188 (19) | 0.73182 (16) | 0.65742 (15) | 0.063 (4) | 0.063 (4) |
| H8A    | 0.273783 | 0.754978 | 0.686079 | 0.077* | 0.077* |
| C16    | 0.02353 (17) | 0.37295 (17) | 0.60136 (15) | 0.062 (4) | 0.062 (4) |
| C17    | −0.0611 (2) | 0.3883 (2) | 0.69450 (18) | 0.074 (5) | 0.074 (5) |
| H17    | −0.046879 | 0.476957 | 0.761543 | 0.090* | 0.090* |
| C18    | −0.1667 (2) | 0.2717 (2) | 0.6877 (2) | 0.087 (5) | 0.087 (5) |
| H18    | −0.223327 | 0.282924 | 0.750162 | 0.105* | 0.105* |
| C19    | −0.1889 (2) | 0.1394 (2) | 0.5899 (2) | 0.091 (6) | 0.091 (6) |
| H19    | −0.259464 | 0.061386 | 0.586085 | 0.110* | 0.110* |
| C20    | −0.1055 (2) | 0.1245 (2) | 0.4981 (2) | 0.085 (6) | 0.085 (6) |
| H20    | −0.120445 | 0.035349 | 0.431597 | 0.102* | 0.102* |
| C21    | 0.00210 (2) | 0.23960 (18) | 0.50212 (17) | 0.072 (5) | 0.072 (5) |
## Supporting Information

| Atom | U1   | U2   | U3   | U12  | U13  | U23  |
|------|------|------|------|------|------|------|
| H21  | 0.056675 | 0.227415 | 0.438926 | 0.087* | | |
| O4A  | 0.7311 (17) | 1.3456 (9) | 1.1039 (9) | 0.081 (3) | 0.562 (6) | |
| H4A  | 0.703178 | 1.409645 | 1.101761 | 0.121* | 0.562 (6) | |
| C9A  | 0.4577 (4) | 0.8518 (3) | 0.6438 (3) | 0.0590 (10) | 0.562 (6) | |
| H9A  | 0.391142 | 0.878767 | 0.598130 | 0.071* | 0.562 (6) | |
| C10A | 0.5317 (5) | 0.9817 (5) | 0.7668 (4) | 0.0543 (10) | 0.562 (6) | |
| C11A | 0.6354 (6) | 0.9803 (5) | 0.8543 (5) | 0.0658 (12) | 0.562 (6) | |
| H11A | 0.660538 | 0.898387 | 0.837484 | 0.079* | 0.562 (6) | |
| C12A | 0.7007 (13) | 1.1010 (7) | 0.9660 (7) | 0.0732 (17) | 0.562 (6) | |
| H12A | 0.766711 | 1.098188 | 1.025843 | 0.088* | 0.562 (6) | |
| C13A | 0.671 (2) | 1.2248 (10) | 0.9914 (13) | 0.0566 (18) | 0.562 (6) | |
| C14A | 0.5663 (11) | 1.2260 (6) | 0.9061 (7) | 0.0598 (13) | 0.562 (6) | |
| H14A | 0.541170 | 1.308011 | 0.923497 | 0.072* | 0.562 (6) | |
| C15A | 0.4986 (6) | 1.1050 (5) | 0.7947 (5) | 0.0599 (11) | 0.562 (6) | |
| H15A | 0.428862 | 1.107069 | 0.737042 | 0.072* | 0.562 (6) | |
| O4B  | 0.751 (2) | 1.3415 (10) | 1.1041 (12) | 0.0660 (19) | 0.438 (6) | |
| H4B  | 0.811016 | 1.329869 | 1.148770 | 0.099* | 0.438 (6) | |
| C9B  | 0.5225 (5) | 0.8106 (4) | 0.6767 (3) | 0.0556 (12) | 0.438 (6) | |
| H9B  | 0.579037 | 0.755148 | 0.694866 | 0.067* | 0.438 (6) | |
| C10B | 0.5752 (7) | 0.9547 (5) | 0.7898 (5) | 0.0511 (12) | 0.438 (6) | |
| C11B | 0.6689 (9) | 0.9752 (6) | 0.8945 (6) | 0.0663 (15) | 0.438 (6) | |
| H11B | 0.696726 | 0.899741 | 0.894502 | 0.080* | 0.438 (6) | |
| C12B | 0.7214 (16) | 1.1043 (8) | 0.9980 (8) | 0.0658 (18) | 0.438 (6) | |
| H12B | 0.788594 | 1.117299 | 1.065956 | 0.079* | 0.438 (6) | |
| C13B | 0.676 (3) | 1.2142 (13) | 1.0021 (16) | 0.061 (2) | 0.438 (6) | |
| C14B | 0.5818 (16) | 1.1960 (9) | 0.8993 (10) | 0.070 (2) | 0.438 (6) | |
| H14B | 0.552914 | 1.271569 | 0.900630 | 0.084* | 0.438 (6) | |
| C15B | 0.5302 (8) | 1.0657 (7) | 0.7936 (6) | 0.0631 (15) | 0.438 (6) | |
| H15B | 0.464655 | 1.053270 | 0.724979 | 0.076* | 0.438 (6) | |
| O5A  | 0.1303 (15) | 0.2093 (13) | 0.7784 (9) | 0.115 (3) | 0.487 (7) | |
| H5A  | 0.070539 | 0.240431 | 0.754421 | 0.172* | 0.487 (7) | |
| C22A | 0.0763 (14) | 0.0563 (13) | 0.8743 (13) | 0.225 (5) | 0.487 (7) | |
| H22A | 0.138147 | 0.010641 | 0.820731 | 0.338* | 0.487 (7) | |
| H22B | −0.022803 | −0.007894 | 0.850552 | 0.338* | 0.487 (7) | |
| H22C | 0.116684 | 0.084083 | 0.960680 | 0.338* | 0.487 (7) | |
| C23A | 0.0723 (17) | 0.1585 (15) | 0.8628 (12) | 0.169 (4) | 0.487 (7) | |
| H23A | 0.115468 | 0.236841 | 0.946343 | 0.203* | 0.487 (7) | |
| H23B | −0.032950 | 0.150256 | 0.849370 | 0.203* | 0.487 (7) | |
| O5B  | 0.8710 (12) | 0.8064 (8) | 0.2186 (6) | 0.0826 (19) | 0.513 (7) | |
| H5B  | 0.951087 | 0.826239 | 0.263805 | 0.124* | 0.513 (7) | |
| C22B | 0.9159 (9) | 0.7123 (10) | 0.0163 (6) | 0.188 (4) | 0.513 (7) | |
| H22D | 1.006501 | 0.697937 | 0.042231 | 0.282* | 0.513 (7) | |
| H22E | 0.922411 | 0.727388 | −0.056931 | 0.282* | 0.513 (7) | |
| H22F | 0.833254 | 0.630478 | −0.003716 | 0.282* | 0.513 (7) | |
| C23B | 0.8974 (16) | 0.8167 (14) | 0.1032 (9) | 0.170 (5) | 0.513 (7) | |
| H23C | 0.813280 | 0.836918 | 0.071298 | 0.204* | 0.513 (7) | |
| H23D | 0.984834 | 0.896724 | 0.125438 | 0.204* | 0.513 (7) | |
## Atomic displacement parameters (Å²)

|    | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|----|-----------|-----------|-----------|-----------|-----------|-----------|
| O1 | 0.0905 (9)| 0.0628 (7)| 0.0673 (7)| 0.0139 (6)| 0.0337 (6)| 0.0250 (5)|
| O2 | 0.0838 (8)| 0.0538 (6)| 0.0756 (7)| −0.0089 (6)| 0.0211 (6)| 0.0162 (5)|
| O3 | 0.0715 (7)| 0.0538 (6)| 0.0615 (6)| −0.0053 (5)| 0.0229 (5)| 0.0151 (5)|
| N1 | 0.0566 (7)| 0.0533 (7)| 0.0646 (7)| 0.0045 (5)| 0.0143 (6)| 0.0278 (6)|
| N2 | 0.0672 (8)| 0.0553 (7)| 0.0673 (8)| −0.0019 (6)| 0.0197 (6)| 0.0255 (6)|
| C1 | 0.0715 (10)| 0.0543 (8)| 0.0617 (9)| 0.0184 (7)| 0.0221 (7)| 0.0270 (7)|
| C2 | 0.0749 (10)| 0.0510 (8)| 0.0595 (9)| 0.0105 (7)| 0.0164 (7)| 0.0180 (7)|
| C3 | 0.0643 (9)| 0.0473 (7)| 0.0662 (9)| 0.0058 (6)| 0.0128 (7)| 0.0236 (7)|
| C4 | 0.0552 (8)| 0.0470 (7)| 0.0594 (8)| 0.0096 (6)| 0.0120 (6)| 0.0246 (6)|
| C5 | 0.0579 (8)| 0.0468 (7)| 0.0584 (8)| 0.0077 (6)| 0.0128 (6)| 0.0224 (6)|
| C6 | 0.0648 (9)| 0.0510 (8)| 0.0658 (9)| 0.0066 (7)| 0.0203 (7)| 0.0264 (7)|
| C7 | 0.0537 (8)| 0.0492 (7)| 0.0621 (8)| 0.0076 (6)| 0.0110 (6)| 0.0282 (6)|
| C8 | 0.0634 (9)| 0.0544 (8)| 0.0630 (9)| 0.0046 (7)| 0.0191 (7)| 0.0235 (7)|
| C16| 0.0520 (8)| 0.0596 (9)| 0.0731 (10)| 0.0040 (7)| 0.0110 (7)| 0.0357 (8)|
| C17| 0.0671 (10)| 0.0711 (10)| 0.0861 (11)| 0.0114 (8)| 0.0254 (9)| 0.0396 (9)|
| C18| 0.0708 (11)| 0.0944 (14)| 0.1048 (14)| 0.0091 (10)| 0.0332 (10)| 0.0577 (12)|
| C19| 0.0731 (12)| 0.0769 (12)| 0.1180 (16)| −0.0086 (10)| 0.0166 (11)| 0.0561 (12)|
| C20| 0.0793 (12)| 0.0613 (10)| 0.0965 (13)| −0.0043 (9)| 0.0089 (10)| 0.0351 (9)|
| C21| 0.0682 (10)| 0.0605 (9)| 0.0770 (10)| 0.0025 (8)| 0.0145 (8)| 0.0310 (8)|
| O4A| 0.089 (5)| 0.074 (4)| 0.057 (3)| 0.015 (2)| 0.014 (2)| 0.015 (2)|
| C9A| 0.0606 (17)| 0.0528 (15)| 0.0582 (15)| 0.0097 (13)| 0.0137 (12)| 0.0244 (12)|
| C10A| 0.057 (2)| 0.047 (2)| 0.0600 (18)| 0.0111 (16)| 0.0153 (15)| 0.0274 (15)|
| C11A| 0.076 (3)| 0.0520 (18)| 0.068 (3)| 0.0192 (18)| 0.007 (2)| 0.027 (2)|
| C12A| 0.075 (3)| 0.069 (3)| 0.068 (3)| 0.020 (2)| 0.000 (3)| 0.028 (2)|
| C13A| 0.064 (3)| 0.046 (2)| 0.056 (3)| 0.010 (2)| 0.020 (3)| 0.023 (2)|
| C14A| 0.072 (3)| 0.045 (2)| 0.061 (2)| 0.016 (2)| 0.0172 (17)| 0.0237 (17)|
| C15A| 0.069 (2)| 0.050 (2)| 0.0616 (18)| 0.0167 (16)| 0.0118 (16)| 0.0281 (17)|
| O4B| 0.071 (3)| 0.043 (3)| 0.065 (4)| 0.010 (2)| 0.008 (2)| 0.012 (2)|
| C9B| 0.060 (2)| 0.0465 (17)| 0.0572 (19)| 0.0071 (15)| 0.0107 (15)| 0.0258 (14)|
| C10B| 0.056 (3)| 0.044 (2)| 0.057 (3)| 0.0135 (18)| 0.015 (2)| 0.0265 (18)|
| C11B| 0.080 (4)| 0.053 (2)| 0.065 (3)| 0.019 (2)| 0.008 (2)| 0.027 (2)|
| C12B| 0.074 (4)| 0.054 (2)| 0.058 (4)| 0.016 (2)| 0.001 (3)| 0.020 (2)|
| C13B| 0.069 (4)| 0.051 (3)| 0.052 (3)| 0.007 (3)| 0.025 (3)| 0.017 (3)|
| C14B| 0.086 (4)| 0.049 (3)| 0.078 (3)| 0.025 (3)| 0.022 (3)| 0.029 (3)|
| C15B| 0.074 (3)| 0.052 (3)| 0.066 (2)| 0.020 (3)| 0.009 (2)| 0.030 (2)|
| O5A| 0.096 (5)| 0.144 (6)| 0.142 (6)| 0.022 (4)| 0.043 (4)| 0.105 (5)|
| C22A| 0.240 (10)| 0.303 (12)| 0.320 (11)| 0.152 (9)| 0.161 (9)| 0.258 (10)|
| C23A| 0.192 (9)| 0.210 (9)| 0.186 (8)| 0.048 (7)| 0.103 (7)| 0.160 (7)|
| O5B| 0.083 (4)| 0.072 (2)| 0.075 (3)| 0.003 (2)| 0.018 (3)| 0.029 (2)|
| C22B| 0.144 (6)| 0.257 (9)| 0.089 (4)| −0.003 (6)| 0.035 (4)| 0.045 (5)|
| C23B| 0.132 (6)| 0.170 (7)| 0.108 (5)| −0.040 (5)| 0.033 (4)| 0.019 (5)|
| Geometric parameters (Å, °) |
|---------------------------|
| O1—H1 0.8200 C9A—C10A 1.505 (4) |
| O1—C1 1.3715 (18) C10A—C11A 1.388 (5) |
| O2—H2 0.8200 C10A—C15A 1.373 (5) |
| O2—C3 1.3595 (18) C11A—H11A 0.9300 |
| O3—C5 1.3680 (17) C11A—C12A 1.377 (5) |
| O3—C9A 1.395 (3) C12A—H12A 0.9300 |
| O3—C9B 1.380 (4) C12A—C13A 1.368 (5) |
| N1—N2 1.3604 (18) C13A—C14A 1.375 (6) |
| N1—C7 1.2953 (19) C14A—H14A 0.9300 |
| N2—H2A 0.8600 C14A—C15A 1.380 (5) |
| N2—C16 1.3896 (18) C15A—H15A 0.9300 |
| C1—C2 1.386 (2) O4B—H4B 0.8200 |
| C1—C6 1.381 (2) O4B—C13B 1.374 (7) |
| C2—H2B 0.9300 C9B—H9B 0.9800 |
| C2—C3 1.378 (2) C9B—C10B 1.509 (5) |
| C3—C4 1.407 (2) C10B—C11B 1.380 (6) |
| C4—C5 1.402 (2) C10B—C15B 1.371 (6) |
| C4—C7 1.459 (2) C11B—H11B 0.9300 |
| C5—C6 1.382 (2) C11B—C12B 1.365 (6) |
| C6—H6 0.9300 C12B—H12B 0.9300 |
| C7—C8 1.495 (2) C12B—C13B 1.363 (7) |
| C8—H8A 0.9700 C13B—C14B 1.372 (7) |
| C8—H8B 0.9700 C14B—H14B 0.9300 |
| C8—H8BC 0.9700 C14B—C15B 1.383 (6) |
| C8—H8BD 0.9700 C15B—H15B 0.9300 |
| C9—C9A 1.484 (3) O5A—H5A 0.8200 |
| C9—C9B 1.496 (4) O5A—C23A 1.425 (7) |
| C16—C17 1.389 (2) C22A—H22A 0.9600 |
| C16—C21 1.389 (2) C22A—H22B 0.9600 |
| C17—H17 0.9300 C22A—H22C 0.9600 |
| C17—C18 1.384 (2) C22A—C23A 1.198 (13) |
| C18—H18 0.9300 C23A—H23A 0.9700 |
| C18—C19 1.375 (3) C23A—H23B 0.9700 |
| C19—H19 0.9300 O5B—H5B 0.8200 |
| C19—C20 1.368 (3) O5B—C23B 1.462 (7) |
| C20—H20 0.9300 C22B—H22D 0.9600 |
| C20—C21 1.390 (2) C22B—H22E 0.9600 |
| C21—H21 0.9300 C22B—H22F 0.9600 |
| O4A—H4A 0.8200 C22B—C23B 1.237 (14) |
| O4A—C13A 1.376 (6) C23B—H23C 0.9700 |
| C9A—H9A 0.9800 C23B—H23D 0.9700 |
| C1—O1—H1 109.5 C15A—C10A—C11A 118.4 (3) |
| C3—O2—H2 109.5 C10A—C11A—H11A 120.2 |
| C5—O3—C9A 116.51 (15) C12A—C11A—C10A 119.5 (4) |
| C5—O3—C9B 117.93 (16) C12A—C11A—H11A 120.2 |
C7—N1—N2 118.79 (13) C11A—C12A—H12A 119.1
N1—N2—H2A 119.9 C13A—C12A—C11A 121.8 (5)
N1—N2—C16 120.22 (13) C13A—C12A—H12A 119.1
C16—N2—H2A 119.9 C12A—C13A—O4A 122.9 (8)
O1—C1—C2 117.22 (14) C12A—C13A—C14A 118.8 (6)
O1—C1—C6 121.70 (14) C14A—C13A—O4A 118.0 (8)
C6—C1—C2 121.08 (14) C13A—C14A—H14A 120.1
C1—C2—H2B 120.2 C13A—C14A—C15A 119.8 (5)
C3—C2—C1 119.57 (14) C15A—C14A—H14A 120.1
C3—C2—H2B 120.2 C10A—C15A—C14A 121.6 (4)
O2—C3—C2 117.53 (14) C10A—C15A—H15A 119.2
O2—C3—C4 120.95 (14) C14A—C15A—H15A 119.2
C2—C3—C4 121.52 (14) C13B—O4B—H4B 109.5
C3—C4—C7 123.41 (13) O3—C9B—C8 115.2 (3)
C5—C4—C3 116.68 (13) O3—C9B—H9B 105.5
C5—C4—C7 119.91 (13) O3—C9B—C10B 109.5 (3)
O3—C5—C4 121.29 (13) C8—C9B—H9B 105.5
O3—C5—C6 116.16 (13) C8—C9B—C10B 114.6 (3)
C6—C5—C4 122.55 (13) C10B—C9B—C10A 122.3 (5)
C1—C6—C5 118.60 (14) C10B—C9B—H9B 119.1 (5)
C1—C6—H6 120.7 C15B—C10B—C9B 118.6 (5)
C5—C6—H6 120.7 C15B—C10B—C11B 119.4
N1—C7—C4 118.58 (13) C10B—C11B—H11B 121.1 (6)
N1—C7—C8 124.66 (14) C12B—C11B—C10B 119.4
C4—C7—C8 116.75 (12) C12B—C11B—H11B 119.9
C7—C8—H8A 108.8 C11B—C12B—C11B 120.1 (7)
C7—C8—H8B 108.8 C11B—C12B—H12B 119.9
C7—C8—H8BC 109.3 C11B—C12B—H12B 119.9
C7—C8—H8BD 109.3 C12B—C13B—O4B 113.9 (10)
C9A—C8—C7 111.81 (17) C12B—C13B—C14B 119.6 (7)
H8A—C8—H8B 107.7 C14B—C13B—O4B 125.5 (11)
H8BC—C8—H8BD 107.9 C13B—C14B—H14B 119.9
C9A—C8—C7 113.85 (15) C13B—C14B—C15B 120.2 (7)
C9A—C8—H8A 108.8 C15B—C14B—H14B 119.9
C9A—C8—H8B 108.8 C10B—C15B—C14B 120.2 (6)
C9B—C8—H8BC 109.3 C10B—C15B—H15B 119.9
C9B—C8—H8BD 109.3 C14B—C15B—H15B 119.9
C17—C16—N2 117.90 (15) C23A—O5A—H5A 109.5
C17—C16—C21 119.15 (15) H22A—C22A—H22B 109.5
C21—C16—N2 122.94 (15) H22A—C22A—H22C 109.5
C16—C17—H17 120.0 H22B—C22A—H22C 109.5
C18—C17—C16 120.09 (18) C23A—C22A—H22B 109.5
C18—C17—H17 120.0 C23A—C22A—H22B 109.5
C19—C18—H18 119.5 C23A—C22A—H22C 109.5
C19—C18—C17 120.91 (18) O5A—C23A—H23A 104.5
C19—C18—H18 119.5 O5A—C23A—H23B 104.5
C18—C19—H19 120.5 C22A—C23A—O5A 130.9 (14)
C20—C19—C18 118.93 (17) C22A—C23A—H23A 104.5
| Bond         | Angle (°) | Bond         | Angle (°) | Bond         | Angle (°) |
|-------------|-----------|-------------|-----------|-------------|-----------|
| O3—C9A—C8   | 115.0 (2) | C23B—C22B—H22D | 109.5   | C20—C19—H19 | 120.5     |
| O3—C9A—H9A  | 106.1     | O5B—C23B—H23C | 108.1   | C19—C20—H20 | 119.2     |
| O3—C9A—C10A | 108.3 (2) | O5B—C23B—H23D | 108.1   | C19—C20—C21 | 121.52 (19) |
| C8—C9A—H9A  | 106.1     | C22B—C23B—O5B | 116.8 (12) | C19—C20—H20 | 119.2 |
| C8—C9A—C10A | 114.4 (2) | C22B—C23B—H23C | 108.1   | C21—C20—H20 | 119.2 |
| C10A—C9A—H9A | 106.1   | C22B—C23B—H23D | 108.1   | C22A—C23A—H23B | 104.5 |
| C11A—C10A—C9A | 121.1 (4) | H23C—C23B—H23D | 107.3   | C12A—C11A—C10A | 64.1 (5) |
| C15A—C10A—C9A | 120.5 (4) |                         |          | C19—C20—C21 | 120.3     |

**Notes:**
- All bond angles are given in degrees.
- The angles refer to the typical bond angles found in organic chemistry.
- The superscript numbers refer to the source of the data.
C5—O3—C9B—C8  
C5—O3—C9B—C10B  
C5—C4—C7—N1  
C5—C4—C7—C8  
C6—C1—C2—C3  
C7—N1—N2—C16  
C7—C4—C5—O3  
C7—C4—C5—C6  

\[ \text{Hydrogen-bond geometry (Å, °)} \]

| D—H···A         | D—H | H···A | D···A   | D—H···A |
|-----------------|------|-------|--------|---------|
| O5A—H5A···C17  | 0.82 | 2.56  | 3.363 (15) | 166     |
| O5A—H5A···C18  | 0.82 | 2.47  | 3.263 (16) | 162     |
| O5B—H5B···C19i | 0.82 | 2.59  | 3.405 (11) | 173     |
| O1—H1···O5Ai   | 0.82 | 1.79  | 2.590 (12) | 166     |
| O1—H1···O5B    | 0.82 | 1.90  | 2.709 (8)  | 170     |
| C8—H8BC···O4Bi | 0.97 | 2.49  | 3.440 (17) | 168     |
| O4A—H4A···O1ai | 0.82 | 1.89  | 2.677 (13) | 160     |
| C9B—H9B···O2i  | 0.98 | 2.39  | 3.347 (5)  | 165     |
| O2—H2···N1     | 0.82 | 1.87  | 2.5975 (18) | 147    |

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