

(2*RS*,4'*RS*)-3'-(3-Chloro-4-methoxyphenyl)-4'-phenyl-4'*H*-spiro[indene-2,5'-isoxazol]-1(3*H*)-one ethanol monosolvate

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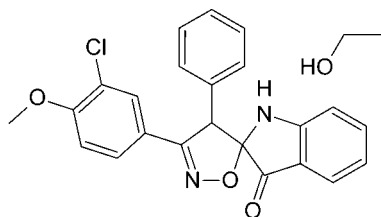
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.039; wR factor = 0.096; data-to-parameter ratio = 16.0.

The title compound, $\text{C}_{23}\text{H}_{17}\text{ClN}_2\text{O}_3 \cdot \text{C}_2\text{H}_6\text{O}$, is the stoichiometric 1:1 ethanol solvate of a racemic reaction product, which forms a conglomerate. The refined Flack parameter of 0.36 (3) indicates racemic twinning. In the structure, molecules are linked into zigzag chains by a series of intermolecular N—H···O and O—H···O hydrogen bonds.

Related literature

For general background to dipolar 1,3-cycloaddition reactions, see: Al Houari *et al.* (2010); Toth *et al.* (1999); El yazidi *et al.* (1994).



Experimental

Crystal data

$\text{C}_{23}\text{H}_{17}\text{ClN}_2\text{O}_3 \cdot \text{C}_2\text{H}_6\text{O}$
 $M_r = 450.90$

Orthorhombic, $P2_12_12_1$
 $a = 8.7112$ (2) Å

$b = 11.3051$ (2) Å
 $c = 22.5913$ (5) Å
 $V = 2224.81$ (8) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.21$ mm⁻¹
 $T = 296$ K
 $0.24 \times 0.17 \times 0.16$ mm

Data collection

Bruker APEXII CCD
 diffractometer
 13160 measured reflections

4757 independent reflections
 3815 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.096$
 $S = 1.06$
 4757 reflections
 297 parameters
 H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³
 Absolute structure: Flack (1983);
 1986 Friedel pairs
 Flack parameter: 0.36 (3)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{N1}-\text{H1}\cdots\text{O4}$ | 0.88 (3) | 2.12 (3) | 2.906 (2) | 149 (2) |
| $\text{O4}-\text{H4A}\cdots\text{O2}^i$ | 0.82 | 2.02 | 2.813 (2) | 164 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2017).

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supplementary materials

Acta Cryst. (2011). E67, o2434 [doi:10.1107/S1600536811032636]

(2*RS*,4'*RS*)-3'-(3-Chloro-4-methoxyphenyl)-4'-phenyl-4'*H*-spiro[indene-2,5'-isoxazol]-1(3*H*)-one ethanol monosolvate

A. Boughaleb, H. Zouihri, S. Gmouh, A. Kerbal and M. El yazidi

Comment

In general, the 1,3-dipolar cycloaddition of aryl nitrile oxides with ethylenic dipolarophiles leads to the regioselective formation of isoxazolines so that the electron withdrawing substituent of the dipolarophile is connected to position 5 of the isoxazoline ring (Al Houari *et al.*, 2010; Toth *et al.*, 1999; El yazidi *et al.*, 1994).

The crystal structure shows that the carbonyl group is bonded to position 5 of the isoxazoline ring, and that the hydrogen atom in position 4 is situated on the same side of the ring as the carbonyl group. The structural study by X-rays is in perfect agreement with the results of IR, ¹H and ¹³C NMR spectroscopic analysis.

The 1,3-dipolar cycloaddition gave the title compound as a racemic product. However, the crystallization process from ethanol resulted in the formation of a conglomerate in space group *P*2₁2₁2₁.

In the crystal molecules are linked into zigzag chains by a series of intermolecular N—H···O and O—H···O hydrogen-bonding interactions, where both the main and the solvent molecules take part (Table 1).

Experimental

4 mmol of (*Z*)-2-benzylidene-1,2-dihydro-3*H*-indol-3-one and 4 mmol of 3-chloro-*N*-hydroxy-4-methoxybenzenecarboximidoyl chloride were dissolved in 30 ml of THF in a 100 ml flask equipped with a condenser. The reaction mixture was refluxed until the complete dissolution of the reagents, and then 1 ml of *N,N*-diethylethanamine was added to the reaction. Monitoring the reaction by thin film chromatography revealed the formation of a single cycloaddition product. After filtration, the organic solution was evaporated under reduced pressure. The residue obtained was recrystallized from ethanol.

Refinement

The H atoms bound to C were treated as riding with their parent atoms [C—H distances are 0.93 Å for CH groups with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$, and 0.97 Å for CH₃ groups with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$. The nitrogen- and oxygen-bound H atoms were located in a difference Fourier map. The nitrogen-bound H atom was refined freely, while the H atom of the hydroxyl group was refined as an idealised rotating group and with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

Refinement with TWIN and BASF instructions in *SHELXL* indicated racemic twinning.

Figures

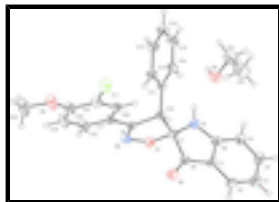


Fig. 1. Molecular view of the title compound showing the atom labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

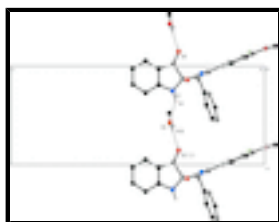


Fig. 2. Partial packing view showing the chain formed by N—H...O and O—H...O hydrogen bondings. H atoms not involved in hydrogen bonds have been omitted for clarity. Symmetry codes: (i) $x+1, y, z$.

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Crystal data

$C_{23}H_{17}ClN_2O_3 \cdot C_2H_6O$

$M_r = 450.90$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 8.7112$ (2) Å

$b = 11.3051$ (2) Å

$c = 22.5913$ (5) Å

$V = 2224.81$ (8) Å³

$Z = 4$

$F(000) = 944$

$D_x = 1.346$ Mg m⁻³

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

Cell parameters from 265 reflections

$\theta = 2.6$ – 26.3°

$\mu = 0.21$ mm⁻¹

$T = 296$ K

Prism, colourless

$0.24 \times 0.17 \times 0.16$ mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

ω and φ scans

13160 measured reflections

4757 independent reflections

3815 reflections with $I > 2\sigma(I)$

$R_{int} = 0.027$

$\theta_{max} = 27.0^\circ$, $\theta_{min} = 1.8^\circ$

$h = -10 \rightarrow 11$

$k = -14 \rightarrow 14$

$l = -28 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

| | |
|--|---|
| $wR(F^2) = 0.096$ | $w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.1284P]$ |
| $S = 1.06$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 4757 reflections | $(\Delta/\sigma)_{\max} < 0.001$ |
| 297 parameters | $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$ |
| 0 restraints | $\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983); 1986 Friedel pairs Flack parameter: 0.36 (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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R- factors based on ALL data will be even larger.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|---------------|----------------------------------|
| C1 | 0.0305 (2) | 0.33701 (16) | 0.41709 (9) | 0.0378 (4) |
| C10 | 0.1211 (2) | 0.35087 (15) | 0.26128 (8) | 0.0333 (4) |
| C11 | 0.2714 (2) | 0.39793 (16) | 0.23651 (8) | 0.0340 (4) |
| C12 | 0.3965 (2) | 0.32367 (18) | 0.22940 (10) | 0.0470 (5) |
| C13 | 0.5324 (3) | 0.3680 (2) | 0.20606 (11) | 0.0590 (6) |
| C14 | 0.5434 (3) | 0.4828 (2) | 0.18859 (11) | 0.0629 (7) |
| C15 | 0.4190 (3) | 0.5564 (2) | 0.19482 (11) | 0.0585 (6) |
| C16 | 0.2839 (2) | 0.51488 (18) | 0.21932 (10) | 0.0471 (5) |
| C17 | -0.0022 (2) | 0.26234 (16) | 0.16411 (8) | 0.0359 (4) |
| C18 | -0.0316 (3) | 0.16313 (18) | 0.12947 (10) | 0.0533 (6) |
| C19 | -0.0905 (3) | 0.17326 (19) | 0.07365 (11) | 0.0613 (7) |
| C2 | -0.0518 (3) | 0.36087 (19) | 0.46878 (10) | 0.0495 (5) |
| C20 | -0.1223 (3) | 0.28381 (19) | 0.04958 (10) | 0.0526 (5) |
| C21 | -0.0928 (2) | 0.38300 (17) | 0.08457 (9) | 0.0423 (5) |
| C22 | -0.0338 (2) | 0.37289 (17) | 0.14030 (9) | 0.0379 (4) |
| C23 | -0.2016 (4) | 0.2016 (3) | -0.04206 (13) | 0.0908 (10) |
| C24 | 0.6087 (4) | 0.5038 (3) | 0.36539 (16) | 0.0920 (9) |
| C25 | 0.4781 (4) | 0.5779 (3) | 0.37505 (16) | 0.1008 (11) |
| C3 | 0.0272 (3) | 0.3874 (2) | 0.51910 (10) | 0.0587 (6) |
| C4 | 0.1875 (3) | 0.3900 (2) | 0.51872 (10) | 0.0569 (6) |
| C5 | 0.2713 (3) | 0.36897 (19) | 0.46834 (9) | 0.0466 (5) |
| C6 | 0.1917 (2) | 0.34114 (16) | 0.41674 (9) | 0.0378 (4) |
| C7 | 0.1305 (2) | 0.28684 (16) | 0.32140 (8) | 0.0362 (4) |
| C8 | -0.0172 (2) | 0.30244 (18) | 0.35872 (9) | 0.0389 (5) |

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|------|---------------|--------------|--------------|--------------|
| C9 | 0.0618 (2) | 0.24995 (15) | 0.22427 (8) | 0.0330 (4) |
| C11 | -0.12367 (8) | 0.52300 (5) | 0.05494 (3) | 0.06402 (19) |
| H1 | 0.346 (3) | 0.324 (2) | 0.3510 (10) | 0.060 (7)* |
| H10 | 0.0447 | 0.4144 | 0.2630 | 0.040* |
| H12 | 0.3895 | 0.2445 | 0.2402 | 0.056* |
| H13 | 0.6170 | 0.3184 | 0.2023 | 0.071* |
| H14 | 0.6345 | 0.5113 | 0.1726 | 0.075* |
| H15 | 0.4257 | 0.6348 | 0.1825 | 0.070* |
| H16 | 0.2011 | 0.5659 | 0.2243 | 0.056* |
| H18 | -0.0107 | 0.0884 | 0.1446 | 0.064* |
| H19 | -0.1096 | 0.1055 | 0.0515 | 0.074* |
| H2 | -0.1585 | 0.3587 | 0.4688 | 0.059* |
| H22 | -0.0145 | 0.4406 | 0.1625 | 0.045* |
| H23A | -0.1057 | 0.1606 | -0.0465 | 0.136* |
| H23B | -0.2379 | 0.2263 | -0.0802 | 0.136* |
| H23C | -0.2756 | 0.1497 | -0.0242 | 0.136* |
| H24A | 0.6892 | 0.5250 | 0.3931 | 0.110* |
| H24B | 0.6475 | 0.5166 | 0.3257 | 0.110* |
| H25A | 0.3977 | 0.5561 | 0.3481 | 0.151* |
| H25B | 0.5062 | 0.6590 | 0.3685 | 0.151* |
| H25C | 0.4427 | 0.5684 | 0.4150 | 0.151* |
| H3 | -0.0258 | 0.4038 | 0.5539 | 0.070* |
| H4 | 0.2392 | 0.4066 | 0.5538 | 0.068* |
| H4A | 0.6442 | 0.3400 | 0.3634 | 0.105* |
| H5 | 0.3779 | 0.3732 | 0.4687 | 0.056* |
| N1 | 0.2497 (2) | 0.31383 (17) | 0.36182 (8) | 0.0444 (4) |
| N2 | 0.07647 (17) | 0.14773 (13) | 0.24799 (7) | 0.0391 (4) |
| O1 | 0.13828 (17) | 0.16082 (11) | 0.30525 (6) | 0.0426 (3) |
| O2 | -0.14510 (17) | 0.28485 (15) | 0.33873 (7) | 0.0583 (4) |
| O3 | -0.1799 (2) | 0.30291 (14) | -0.00516 (8) | 0.0716 (5) |
| O4 | 0.57042 (17) | 0.38095 (16) | 0.37275 (9) | 0.0703 (5) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0382 (11) | 0.0361 (10) | 0.0391 (11) | -0.0006 (8) | 0.0064 (9) | 0.0047 (8) |
| C10 | 0.0287 (9) | 0.0337 (9) | 0.0374 (10) | 0.0030 (8) | 0.0024 (8) | 0.0008 (7) |
| C11 | 0.0332 (11) | 0.0352 (9) | 0.0336 (10) | -0.0031 (8) | 0.0001 (8) | -0.0006 (8) |
| C12 | 0.0396 (12) | 0.0441 (11) | 0.0571 (13) | -0.0002 (9) | 0.0087 (10) | -0.0001 (9) |
| C13 | 0.0373 (12) | 0.0655 (15) | 0.0741 (17) | -0.0016 (11) | 0.0164 (12) | -0.0106 (13) |
| C14 | 0.0480 (14) | 0.0782 (18) | 0.0625 (16) | -0.0275 (13) | 0.0121 (12) | 0.0003 (13) |
| C15 | 0.0599 (16) | 0.0500 (13) | 0.0657 (16) | -0.0216 (11) | 0.0001 (13) | 0.0119 (11) |
| C16 | 0.0464 (12) | 0.0387 (10) | 0.0561 (13) | -0.0035 (9) | -0.0030 (11) | 0.0045 (10) |
| C17 | 0.0311 (10) | 0.0377 (10) | 0.0390 (11) | -0.0025 (8) | -0.0014 (8) | -0.0013 (8) |
| C18 | 0.0685 (15) | 0.0365 (11) | 0.0549 (14) | -0.0078 (10) | -0.0150 (12) | 0.0033 (10) |
| C19 | 0.0843 (19) | 0.0429 (12) | 0.0566 (15) | -0.0146 (11) | -0.0225 (13) | -0.0047 (10) |
| C2 | 0.0509 (13) | 0.0523 (13) | 0.0454 (12) | -0.0015 (10) | 0.0150 (11) | 0.0028 (10) |
| C20 | 0.0555 (13) | 0.0545 (13) | 0.0476 (13) | -0.0071 (11) | -0.0158 (12) | 0.0031 (10) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C21 | 0.0380 (12) | 0.0417 (10) | 0.0472 (12) | 0.0030 (8) | -0.0033 (10) | 0.0048 (9) |
| C22 | 0.0344 (10) | 0.0375 (10) | 0.0417 (11) | 0.0010 (8) | -0.0001 (9) | -0.0022 (8) |
| C23 | 0.129 (3) | 0.0773 (18) | 0.0661 (19) | -0.0205 (18) | -0.0483 (19) | -0.0026 (15) |
| C24 | 0.078 (2) | 0.090 (2) | 0.108 (3) | -0.0139 (19) | 0.0090 (19) | -0.0018 (18) |
| C25 | 0.106 (3) | 0.074 (2) | 0.122 (3) | -0.0016 (19) | -0.005 (2) | 0.0111 (19) |
| C3 | 0.0709 (18) | 0.0657 (15) | 0.0396 (13) | 0.0004 (13) | 0.0141 (12) | 0.0017 (11) |
| C4 | 0.0744 (18) | 0.0605 (14) | 0.0357 (12) | 0.0038 (12) | -0.0051 (11) | 0.0001 (10) |
| C5 | 0.0484 (13) | 0.0502 (12) | 0.0413 (12) | 0.0023 (10) | -0.0078 (10) | 0.0075 (9) |
| C6 | 0.0410 (11) | 0.0333 (9) | 0.0392 (11) | 0.0020 (8) | 0.0030 (9) | 0.0054 (8) |
| C7 | 0.0304 (10) | 0.0396 (10) | 0.0387 (10) | 0.0008 (8) | 0.0041 (9) | -0.0017 (8) |
| C8 | 0.0334 (12) | 0.0391 (10) | 0.0442 (12) | -0.0022 (8) | 0.0066 (9) | 0.0003 (9) |
| C9 | 0.0265 (9) | 0.0351 (9) | 0.0374 (10) | -0.0009 (7) | 0.0052 (8) | 0.0013 (8) |
| C11 | 0.0852 (4) | 0.0498 (3) | 0.0571 (3) | 0.0136 (3) | -0.0127 (4) | 0.0081 (3) |
| N1 | 0.0296 (10) | 0.0642 (11) | 0.0393 (10) | -0.0021 (8) | 0.0047 (8) | -0.0020 (8) |
| N2 | 0.0414 (10) | 0.0371 (9) | 0.0389 (9) | -0.0005 (7) | 0.0019 (7) | 0.0001 (7) |
| O1 | 0.0502 (8) | 0.0370 (7) | 0.0405 (8) | 0.0051 (6) | 0.0008 (7) | 0.0041 (6) |
| O2 | 0.0312 (9) | 0.0843 (11) | 0.0595 (10) | -0.0069 (7) | 0.0045 (7) | -0.0158 (8) |
| O3 | 0.0952 (14) | 0.0641 (10) | 0.0555 (10) | -0.0077 (9) | -0.0353 (10) | -0.0004 (8) |
| O4 | 0.0398 (9) | 0.0696 (12) | 0.1014 (14) | -0.0027 (8) | 0.0102 (9) | -0.0073 (10) |

Geometric parameters (Å, °)

| | | | |
|---------|-----------|----------|-----------|
| C1—C2 | 1.397 (3) | C23—H23C | 0.9600 |
| C10—H10 | 0.9800 | C23—H23B | 0.9600 |
| C10—C7 | 1.541 (3) | C23—H23A | 0.9600 |
| C10—C11 | 1.520 (2) | C24—H24B | 0.9700 |
| C10—C9 | 1.506 (2) | C24—H24A | 0.9700 |
| C11—C12 | 1.385 (3) | C24—C25 | 1.429 (5) |
| C11—C16 | 1.382 (3) | C25—H25C | 0.9600 |
| C12—H12 | 0.9300 | C25—H25B | 0.9600 |
| C12—C13 | 1.389 (3) | C25—H25A | 0.9600 |
| C13—H13 | 0.9300 | C3—H3 | 0.9300 |
| C13—C14 | 1.361 (4) | C4—H4 | 0.9300 |
| C14—H14 | 0.9300 | C4—C3 | 1.396 (4) |
| C15—H15 | 0.9300 | C4—C5 | 1.373 (3) |
| C15—C14 | 1.373 (4) | C5—H5 | 0.9300 |
| C16—H16 | 0.9300 | C6—C1 | 1.405 (3) |
| C16—C15 | 1.383 (3) | C6—C5 | 1.392 (3) |
| C17—C9 | 1.476 (3) | C8—C7 | 1.549 (3) |
| C17—C18 | 1.391 (3) | C8—C1 | 1.437 (3) |
| C18—H18 | 0.9300 | N1—H1 | 0.88 (3) |
| C18—C19 | 1.366 (3) | N1—C7 | 1.416 (3) |
| C19—H19 | 0.9300 | N1—C6 | 1.375 (3) |
| C2—H2 | 0.9300 | N2—O1 | 1.409 (2) |
| C2—C3 | 1.362 (3) | N2—C9 | 1.280 (2) |
| C20—C19 | 1.391 (3) | O1—C7 | 1.472 (2) |
| C21—C11 | 1.739 (2) | O2—C8 | 1.218 (2) |
| C21—C20 | 1.396 (3) | O3—C23 | 1.429 (3) |
| C22—H22 | 0.9300 | O3—C20 | 1.352 (3) |

supplementary materials

| | | | |
|-------------|-------------|---------------|-------------|
| C22—C17 | 1.388 (3) | O4—H4A | 0.8200 |
| C22—C21 | 1.365 (3) | O4—C24 | 1.438 (4) |
| C6—C1—C8 | 107.05 (17) | H23A—C23—H23C | 109.5 |
| C2—C1—C8 | 132.17 (19) | O3—C23—H23C | 109.5 |
| C2—C1—C6 | 120.7 (2) | H23A—C23—H23B | 109.5 |
| C7—C10—H10 | 110.1 | O3—C23—H23B | 109.5 |
| C11—C10—H10 | 110.1 | O3—C23—H23A | 109.5 |
| C9—C10—H10 | 110.1 | H24A—C24—H24B | 108.0 |
| C11—C10—C7 | 116.32 (16) | O4—C24—H24B | 109.4 |
| C9—C10—C7 | 98.72 (13) | C25—C24—H24B | 109.4 |
| C9—C10—C11 | 110.86 (15) | O4—C24—H24A | 109.4 |
| C12—C11—C10 | 120.54 (16) | C25—C24—H24A | 109.4 |
| C16—C11—C10 | 120.42 (17) | C25—C24—O4 | 111.3 (3) |
| C16—C11—C12 | 119.01 (17) | H25B—C25—H25C | 109.5 |
| C13—C12—H12 | 120.1 | H25A—C25—H25C | 109.5 |
| C11—C12—H12 | 120.1 | C24—C25—H25C | 109.5 |
| C11—C12—C13 | 119.73 (19) | H25A—C25—H25B | 109.5 |
| C12—C13—H13 | 119.5 | C24—C25—H25B | 109.5 |
| C14—C13—H13 | 119.5 | C24—C25—H25A | 109.5 |
| C14—C13—C12 | 120.9 (2) | C4—C3—H3 | 119.8 |
| C15—C14—H14 | 120.2 | C2—C3—H3 | 119.8 |
| C13—C14—H14 | 120.2 | C2—C3—C4 | 120.3 (2) |
| C13—C14—C15 | 119.5 (2) | C3—C4—H4 | 118.9 |
| C16—C15—H15 | 119.8 | C5—C4—H4 | 118.9 |
| C14—C15—H15 | 119.8 | C5—C4—C3 | 122.2 (2) |
| C14—C15—C16 | 120.4 (2) | C6—C5—H5 | 121.0 |
| C15—C16—H16 | 119.8 | C4—C5—H5 | 121.0 |
| C11—C16—H16 | 119.8 | C4—C5—C6 | 117.9 (2) |
| C11—C16—C15 | 120.3 (2) | C5—C6—C1 | 120.04 (19) |
| C18—C17—C9 | 120.73 (16) | N1—C6—C1 | 111.39 (18) |
| C22—C17—C9 | 121.14 (16) | N1—C6—C5 | 128.57 (18) |
| C22—C17—C18 | 118.13 (17) | C10—C7—C8 | 112.47 (16) |
| C17—C18—H18 | 119.3 | O1—C7—C8 | 106.46 (14) |
| C19—C18—H18 | 119.3 | N1—C7—C8 | 103.50 (15) |
| C19—C18—C17 | 121.38 (19) | O1—C7—C10 | 103.80 (14) |
| C20—C19—H19 | 119.6 | N1—C7—C10 | 120.40 (16) |
| C18—C19—H19 | 119.6 | N1—C7—O1 | 109.55 (15) |
| C18—C19—C20 | 120.74 (19) | C1—C8—C7 | 106.86 (16) |
| C1—C2—H2 | 120.6 | O2—C8—C7 | 122.64 (18) |
| C3—C2—H2 | 120.6 | O2—C8—C1 | 130.48 (19) |
| C3—C2—C1 | 118.7 (2) | C17—C9—C10 | 124.70 (15) |
| C19—C20—C21 | 117.63 (19) | N2—C9—C10 | 114.66 (16) |
| O3—C20—C21 | 117.26 (19) | N2—C9—C17 | 120.58 (16) |
| O3—C20—C19 | 125.11 (19) | C7—N1—H1 | 123.1 (15) |
| C20—C21—C11 | 118.99 (16) | C6—N1—H1 | 124.9 (15) |
| C22—C21—C11 | 119.30 (15) | C6—N1—C7 | 111.18 (17) |
| C22—C21—C20 | 121.64 (18) | C9—N2—O1 | 109.12 (14) |
| C17—C22—H22 | 119.8 | N2—O1—C7 | 108.16 (12) |
| C21—C22—H22 | 119.8 | C20—O3—C23 | 117.03 (19) |

| | | | |
|---------------|-------------|------------|-------|
| C21—C22—C17 | 120.49 (18) | C24—O4—H4A | 109.5 |
| H23B—C23—H23C | 109.5 | | |

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> |
|---------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N1—H1 \cdots O4 | 0.88 (3) | 2.12 (3) | 2.906 (2) | 149 (2) |
| O4—H4A \cdots O2 ⁱ | 0.82 | 2.02 | 2.813 (2) | 164. |

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

Fig. 1

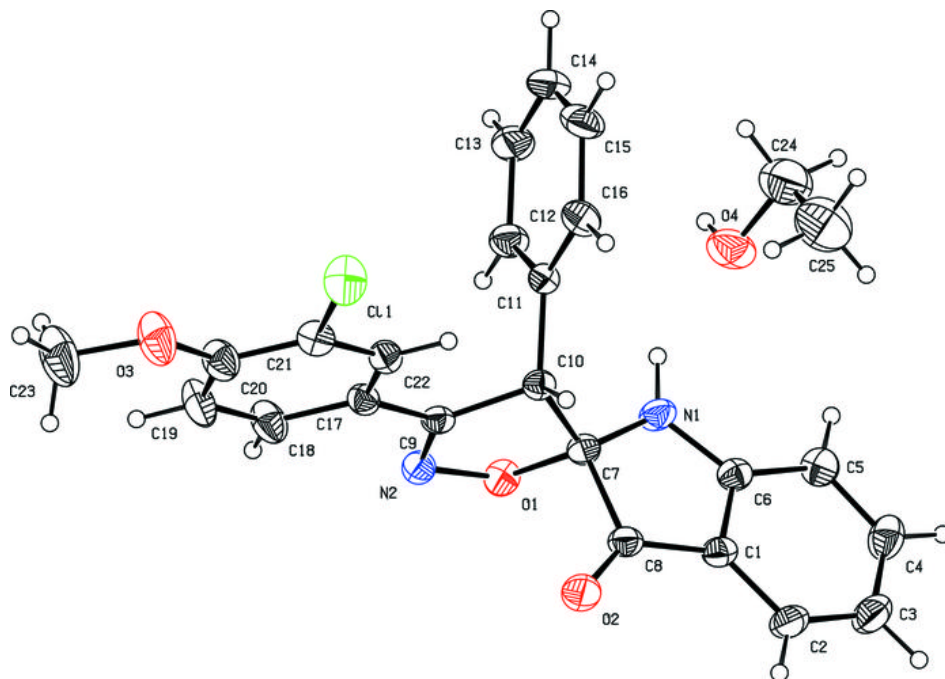


Fig. 2

