Crystal structure and Hirshfeld surface analysis of (Z)-4-(4-hydroxybenzylidene)-3-methylisoxazol-5(4H)-one. Corrigendum

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In the paper by Zemamouche et al. [Acta Cryst. (2018), E74, 926–930], there is an error in the name of the first author.

The name of the first author in the paper by Zemamouche et al. (2018) is incorrect and should be ‘Wissame Zemamouche’ as given above.

References

Zemamouche, W., Laroun, R., Hamdouni, N., Brihi, O., Boudjada, A. & Debache, A. (2018). Acta Cryst. E74, 926–930.
Crystal structure and Hirshfeld surface analysis of (Z)-4-(4-hydroxybenzylidene)-3-methylisoaxol-5(4H)-one

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The title compound, C11H9NO3, contains an isoxazole and a hydroxybenzylidene ring, which are inclined to each other by 3.18 (8)°. There is an intramolecular C—H···O contact forming an S(7) ring. In the crystal, molecules stack head-to-tail in columns along the b-axis direction, linked by offset π···π interactions [intercentroid distances of 3.676 (1) and 3.723 (1) Å]. The columns are linked by O—H···O and O—H···N hydrogen bonds, forming layers parallel to the ab plane. The layers are linked by C—H···O hydrogen bonds, forming a supramolecular three-dimensional framework. An analysis of the Hirshfeld surfaces points to the importance of the O—H···O and O—H···N hydrogen bonding in the packing mechanism of the crystal structure.

1. Chemical context

The isoxazole ring system is a component of many natural and medicinally active molecules that exhibit interesting biological activities (Wang et al., 2012). Isoxazole derivatives have been shown to possess anticonvulsant (Balalaie et al., 2000), antifungal (Santos et al., 2010), HDAC inhibitory (Conti et al., 2010), analgesic (Kano et al., 1967), antimicrobial (Padmaja et al., 2009), antituberculosis (Lee et al., 2009), antimonial (Mao et al., 2010) and many other biological properties. They are also used for the treatment of leishmaniasis (Changtam et al., 2010) and for the treatment of patients with active arthritis (Suryawanshi et al., 2012). Furthermore, the isoxazole unit can be used as the basis for the design and construction of merocyanine dyes, which are used in optical recording and non-linear optical research (Zhang et al., 2011). In the present study, we report on the synthesis, crystal structure and Hirshfeld surface analysis of the title isoxazole derivative.

2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The molecule is composed of an isoxazole ring (O1/N1/
C1–C3) that is almost coplanar with the benzene ring (C6–C11) of the 4-hydroxybenzylidene substituent; the two rings are inclined to each other by 3.18 (8)°. The configuration about the C2=C5 bond is Z, and within the molecule there is a short intramolecular C11—H11···O2 contact (Table 1), forming an S(7) ring motif. The bond lengths and bond angles agree well with those observed for a similar compound, the 2-hydroxybenzylidene analogue, (Z)-4-(2-hydroxybenzylidene)-3-methylisoxazol-5(4H)-one (Cheng et al., 2009). Here the hydroxyl group is in the ortho position, compared to the para position in the title compound.

3. Supramolecular features

In the crystal, molecules stack head-to-tail along the b-axis direction (Fig. 2), and are linked by offset π–π interactions: Cg1· · · Cg2iii,iv intercentroid distances are 3.676 (1) and 3.723 (1) Å, interplanar distances are 3.426 (1) and 3.489 (1) Å, slippages are 1.287 and 1.458 Å with the rings inclined to each other by 3.18 (8)°; symmetry codes: (iii) x + 1, y, z; (iv) −x, −y, −z + 1. The molecular columns are linked by O—H···O and O—H···N hydrogen bonds, forming a supramolecular three-dimensional framework (Table 1 and Fig. 2).

4. Analysis of the Hirshfeld surfaces

Additional insight into the intermolecular interactions was obtained from an analysis of the Hirshfeld surface (Spackman & Jayatilaka, 2009) and the two-dimensional fingerprint plots (McKinnon et al., 2007). The program CrystalExplorer (Turner et al., 2017) was used to generate Hirshfeld surfaces mapped over dnorm, de and the electrostatic potential for the title compound.

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**Figure 1**
The molecular structure of the title compound, with atom labelling and displacement ellipsoids drawn at the 50% probability level. The intramolecular C—H···O contact (see Table 1) is shown as a dashed line.

**Figure 2**
A view along the b axis of the crystal packing of the title compound. Only the H atoms (grey balls) involved in hydrogen bonding (see Table 1) have been included.

**Figure 3**
A view of the Hirshfeld surface mapped over dnorm, with neighbouring interactions shown as green dashed lines.

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

| D—H···A      | D—H     | D···A     | D···A     |
|--------------|---------|-----------|-----------|
| C11—H11···O2 | 0.93    | 2.15      | 3.089 (2) |
| O3—H3O···O1i | 0.86 (2) | 2.41 (2)  | 2.9119 (18) |
| O3—H3O···N1i | 0.86 (2) | 2.00 (2)  | 2.7984 (19) |
| C5—H5···O2ii | 0.93    | 2.47      | 3.3655 (17) |
| C7—H7···O2ii | 0.93    | 2.55      | 3.4038 (19) |

Symmetry codes: (i) x + 1, y, z; (ii) −x, −y, −z + 1, (iii) x + 3, −y + 1, −z + 1; (iv) −x, −y, + 2, −z + 1.
The analysis of the Hirshfeld surface mapped over $d_{norm}$ is shown in Fig. 3. The $O3-H3\cdots O1^i$ and $O3-H3\cdots N1^i$ interactions between the corresponding donor and acceptor atoms are visualized as bright-red spots on both sides (zones 1 and 2) of the Hirshfeld surfaces (Fig. 4). Two other red spots exist, corresponding to the $C5-H5\cdots O2^ii$ and $C7-H7\cdots O^ii$ interactions (Fig. 4, zones 3 and 4); these are considered to be weak interactions by comparing them to the sum of the van der Waals radii. The donors and acceptors of intermolecular hydrogen bonds appear as blue and red regions, respectively, around the participating atoms on the Hirshfeld surface mapped over the calculated electrostatic potential (Fig. 5).

The overall two-dimensional fingerprint plot is illustrated in Fig. 6a, and the $H\cdots O/O\cdots H$, $H\cdots H$, $C\cdots H/H\cdots C$, and $N\cdots H/H\cdots N$ contacts are illustrated in Fig. 6b–f, respectively. The $H\cdots O/O\cdots H$ contacts (Fig. 6b) account for 33.9% of the Hirshfeld surface, representing the largest contribution and is displayed on the fingerprint plots by a pair of short spikes at $d_e + d_i = 2.3 \text{ Å}$. This distance is ca. 0.5 Å shorter than the sum of the van der Waals radii of the individual atoms, which means it is a very strong interaction. A contribution of 31.0% was found for the interatomic $H\cdots H$ contacts (Fig. 6c), with a distinctive peak in the fingerprint plot at $d_e + d_i = 2.2 \text{ Å}$; the van der Waals radius for this interaction is 2.4 Å. The $H\cdots C/C\cdots H$ contacts (9.6% contribution; Fig. 6d) are indicated by a pair of short peaks at $d_e + d_i = 2.7 \text{ Å}$, equal to the sum of the van der Waals radii. The $H\cdots N/N\cdots H$ contacts (Fig. 6e), which account for only 8.4% of the Hirshfeld surface, are displayed on the fingerprint plot as a pair of long spikes at $d_e + d_i = 2.0 \text{ Å}$. This distance differs by ca. 0.7 Å from the sum of the van der Waals radii, which means it is the strongest interaction present. The $C\cdots C$ contacts (Fig. 6f), which account for 11.7% of the Hirshfeld surface with $d_e + d_i = 3.4 \text{ Å}$, indicate the presence of π–π stacking.
5. Database survey

A search of the Cambridge Structural Database (CSD, V3.59, last update February 2018; Groom et al., 2016) for 4-substituted 3-methyl-isoxazol-5(4H)-ones gave 22 hits. Of these, six compounds involve a benzylidene substituent. The configuration about the C==C bond is Z in all six compounds and the benzene ring is inclined to the isoxazole ring by angles as small as 1.14° in (Z)-4-benzylidene-3-methylisoxazol-5(4H)-one (MBYIOZ01; Chandra et al., 2012) compared to ca 11.59° in (Z)-4-(4-methoxybenzylidene)-3-methyl-2-oxazol-5(4H)- one (YIMWIC; Saikh et al., 2013). The most relevant structure is the 2-hydroxybenzylidene analogue, viz. (Z)-4-(2-hydroxybenzylidene)-3-methylisoxazol-5(4H)-one (AJESAK; Cheng et al., 2009), in which the two rings are inclined to each other by ca 6.53°, compared to 3.18° in the title compound. The Z configuration of all six molecules indicates that there is an intramolecular C—H⋯O contact present forming an S(7) ring motif, as in the title compound (Fig. 1 and Table 1).

6. Synthesis and crystallization

4-Hydroxybenzaldehyde (1 mmol), hydroxylamine hydrochloride (1 mmol), ethylacetoacetate (1 mmol) and K₂CO₃ (5 ml) were mixed in a 25 ml flask equipped with a magnetic stirrer. The mixture was refluxed in 5 ml of water for 1 h (the reaction was monitored by TLC). On completion of the reaction, the mixture was gradually poured into ice-cold water. Stirring was maintained for a few minutes and the obtained solid was filtered and purified by crystallization from ethanol (yield 83%), yielding pale-yellow needle-like crystals on slow evaporation of the solvent.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydroxyl H atom was located in a difference-Fourier map and freely refined. The C-bound H atoms were included in calculated positions and treated as riding: C—H = 0.93–0.96 Å with U_H(C-methyl) and 1.2U_H(C) for other H atoms.

Acknowledgements

We thank Mr F. Saidi, Engineer at the Laboratory of Crystallography, University Constantine 1, for assistance with the data collection.

Funding information

This work was supported by the Laboratoire de Cristallographie, Departement de Physique, Université Constantine 1, Algeria.

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Crystal structure and Hirshfeld surface analysis of \((Z)-4-(4\text{-hydroxybenzylidene})-3\text{-methylisoxazol-5(4}H\text{-one)}\)

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Computing details

Data collection: CrysAlis PRO (Agilent, 2013); cell refinement: CrysAlis PRO (Agilent, 2013); data reduction: CrysAlis PRO (Agilent, 2013); program(s) used to solve structure: SIR92 (Altomare et al., 1994); program(s) used to refine structure: SHELXL2018/3 (Sheldrick, 2015); molecular graphics: PLATON (Spek, 2009) and Mercury (Macrae et al., 2008); software used to prepare material for publication: SHELXL2018/3 (Sheldrick, 2015), PLATON (Spek, 2009) and publCIF (Westrip, 2010).

\((Z)-4-(4\text{-Hydroxybenzylidene})-3\text{-methylisoxazol-5(4}H\text{-one)}\)

Crystal data

\(\text{C}_{11}\text{H}_{9}\text{NO}_{3}\)

\(M_r = 203.19\)

Monoclinic, \(C2/c\)

\(a = 21.191 (2) \text{Å}\)

\(b = 7.2352 (11) \text{Å}\)

\(c = 12.9569 (14) \text{Å}\)

\(\beta = 103.920 (11)°\)

\(V = 1928.2 (4) \text{Å}^3\)

\(Z = 8\)

\(F(000) = 848\)

\(D_x = 1.400 \text{Mg m}^{-3}\)

Mo \(K\alpha\) radiation, \(\lambda = 0.71073 \text{Å}\)

Cell parameters from 1714 reflections

\(\theta = 4.1–32°\)

\(\mu = 0.10 \text{mm}^{-1}\)

\(T = 293 \text{K}\)

Needle, pale yellow

\(0.36 \times 0.23 \times 0.11 \text{mm}\)

Data collection

Agilent Technologies Xcalibur, Eos diffractometer

4536 measured reflections

1891 independent reflections

1465 reflections with \(I > 2\sigma(I)\)

\(R_{int} = 0.021\)

\(\theta_{\text{max}} = 26.0°, \theta_{\text{min}} = 3.2°\)

\(h = -26\rightarrow23\)

\(k = -8\rightarrow8\)

\(l = -15\rightarrow12\)

Refinement

Refinement on \(F^2\)

0 restraints

Primary atom site location: structure-invariant direct methods

Least-squares matrix: full

Secondary atom site location: difference Fourier map

\(R[F^2 > 2\sigma(F^2)] = 0.040\)

Hydrogen site location: mixed

\(wR(F^2) = 0.113\)

\(S = 1.04\)

1891 reflections

142 parameters
H atoms treated by a mixture of independent and constrained refinement

\[ w = 1/\left[\sigma^2(F_o^2) + (0.0595P)^2\right] \]

where \( P = (F_o^2 + 2F_c^2)/3 \)

\( (\Delta/\sigma)_{\text{max}} = 0.001 \)

\( \Delta\rho_{\text{min}} = -0.13 \text{ e Å}^{-3} \)

\( \Delta\rho_{\text{max}} = 0.19 \text{ e Å}^{-3} \)

Extinction correction: (SHELXL-2018/3; Sheldrick, 2015),

\[ F_c^\ast = kF_c\left[1 + 0.001x\lambda^2\sin(2\theta)/\sin(\theta)^{1/4}\right] \]

Extinction coefficient: 0.0020 (6)

**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)**

| Atom | x     | y     | z     | Uiso* / Ueq |
|------|-------|-------|-------|-------------|
| O1   | 0.62276 (5) | 0.14571 (16) | 0.60644 (8) | 0.0492 (4) |
| O2   | 0.72284 (6) | 0.0722 (2) | 0.69282 (8) | 0.0626 (4) |
| O3   | 0.99352 (6) | -0.2162 (2) | 0.60528 (10) | 0.0613 (4) |
| H3O  | 1.0132 (11) | -0.237 (3) | 0.5558 (16) | 0.092 (8)* |
| N1   | 0.58554 (6) | 0.16143 (18) | 0.49783 (10) | 0.0448 (4) |
| C1   | 0.68443 (8) | 0.0886 (2) | 0.60805 (12) | 0.0392 (4) |
| C2   | 0.68735 (7) | 0.06174 (19) | 0.49745 (11) | 0.0308 (3) |
| C3   | 0.62309 (7) | 0.11381 (19) | 0.43784 (12) | 0.0347 (4) |
| C4   | 0.59837 (9) | 0.1192 (2) | 0.32016 (13) | 0.0521 (5) |
| H4A  | 0.558271 | 0.187052 | 0.302307 | 0.078* |
| H4B  | 0.591061 | -0.004561 | 0.293277 | 0.078* |
| H4C  | 0.629819 | 0.178707 | 0.289026 | 0.078* |
| C5   | 0.73584 (7) | 0.00370 (18) | 0.45337 (10) | 0.0321 (4) |
| H5   | 0.723867 | 0.000592 | 0.329466 | 0.039* |
| C6   | 0.80178 (7) | -0.05397 (19) | 0.49675 (11) | 0.0309 (3) |
| C7   | 0.83942 (7) | -0.0968 (2) | 0.42463 (11) | 0.0374 (4) |
| H7   | 0.820742 | -0.088293 | 0.352088 | 0.045* |
| C8   | 0.90318 (8) | -0.1508 (2) | 0.45834 (12) | 0.0407 (4) |
| H8   | 0.927260 | -0.178127 | 0.409006 | 0.049* |
| C9   | 0.93153 (7) | -0.1646 (2) | 0.56651 (13) | 0.0391 (4) |
| C10  | 0.89502 (8) | -0.1264 (2) | 0.63930 (12) | 0.0414 (4) |
| H10  | 0.913860 | -0.136911 | 0.711693 | 0.050* |
| C11  | 0.83143 (7) | -0.0732 (2) | 0.60553 (11) | 0.0374 (4) |
| H11  | 0.807415 | -0.049326 | 0.655426 | 0.045* |

**Atomic displacement parameters (Å²)**

|  | U^11 | U^22 | U^33 | U^12 | U^13 | U^23 |
|---|------|------|------|------|------|------|
| O1 | 0.0342 (7) | 0.0808 (8) | 0.0351 (6) | 0.0182 (6) | 0.0132 (5) | 0.0031 (5) |
| O2 | 0.0413 (7) | 0.1180 (11) | 0.0271 (6) | 0.0283 (7) | 0.0056 (5) | -0.0015 (6) |
| O3 | 0.0259 (6) | 0.1075 (11) | 0.0490 (8) | 0.0214 (6) | 0.0061 (6) | -0.0077 (7) |
| N1 | 0.0290 (8) | 0.0631 (8) | 0.0410 (8) | 0.0122 (7) | 0.0057 (6) | 0.0029 (6) |
| C1 | 0.0271 (8) | 0.0580 (9) | 0.0335 (8) | 0.0104 (7) | 0.0091 (7) | 0.0017 (7) |
C2 0.0256 (8) 0.0372 (7) 0.0289 (7) 0.0031 (6) 0.0050 (6) 0.0010 (6)
C3 0.0274 (8) 0.0405 (7) 0.0363 (8) 0.0049 (7) 0.0076 (7) 0.0005 (6)
C4 0.0392 (10) 0.0710 (11) 0.0397 (9) 0.0134 (9) −0.0027 (8) −0.0004 (8)
C5 0.0294 (8) 0.0403 (7) 0.0263 (7) 0.0033 (6) 0.0061 (6) −0.0002 (6)
C6 0.0258 (8) 0.0373 (7) 0.0297 (7) 0.0036 (6) 0.0067 (6) −0.0021 (6)
C7 0.0324 (9) 0.0516 (9) 0.0283 (7) 0.0051 (7) 0.0075 (6) −0.0001 (6)
C8 0.0297 (9) 0.0582 (9) 0.0374 (8) 0.0057 (7) 0.0141 (7) −0.0051 (7)
C9 0.0225 (8) 0.0503 (9) 0.0435 (8) 0.0067 (7) 0.0062 (7) −0.0045 (7)
C10 0.0323 (9) 0.0588 (9) 0.0304 (8) 0.0081 (7) 0.0025 (7) −0.0033 (7)
C11 0.0292 (8) 0.0531 (9) 0.0310 (8) 0.0083 (7) 0.0093 (6) −0.0032 (6)

Geometric parameters (Å, º)

| Bond/Distance | Value |
|---------------|-------|
| O1—C1         | 1.3660 (19) |
| O1—N1         | 1.4426 (15) |
| O2—C1         | 1.2054 (18) |
| O3—C9         | 1.3417 (18) |
| N1—C3         | 1.2860 (19) |
| C1—C2         | 1.462 (2) |
| C2—C3         | 1.445 (2) |
| C3—C4         | 1.489 (2) |
| C4—H4A        | 0.9600 |
| C4—H4B        | 0.9600 |
| C4—H4C        | 0.9600 |
| C1—O1—N1      | 109.59 (11) |
| C9—O3—H3O     | 112.2 (14) |
| C3—N1—O1      | 107.22 (11) |
| O2—C1—O1      | 118.53 (14) |
| O2—C1—C2      | 134.54 (15) |
| O1—C1—C2      | 106.93 (12) |
| C5—C2—C3      | 124.58 (13) |
| C5—C2—C1      | 131.96 (13) |
| C3—C2—C1      | 103.46 (13) |
| N1—C3—C2      | 112.79 (13) |
| N1—C3—C4      | 119.70 (14) |
| C2—C3—C4      | 127.50 (14) |
| C3—C4—H4A     | 109.5 |
| C3—C4—H4B     | 109.5 |
| C4—H4A—C4     | 109.5 |
| C4—H4B—C4     | 109.5 |
| C2—C5—C6      | 133.54 (13) |
| C2—C5—H5      | 113.2 |
C1—O1—N1—C3 0.71 (16)  C1—C2—C5—C6 0.4 (3)  
N1—O1—C1—O2 178.29 (14)  C2—C5—C6—C11 4.0 (3)  
N1—O1—C1—C2 −1.37 (16)  C2—C5—C6—C11 −176.83 (15)  
O2—C1—C2—C5 1.7 (3)  C11—C6—C7—C8 −1.6 (2)  
O1—C1—C2—C5 −178.74 (15)  C5—C6—C7—C8 179.15 (14)  
O2—C1—C2—C3 −178.13 (19)  C6—C7—C8—C9 0.2 (2)  
O1—C1—C2—C3 1.45 (16)  C7—C8—C9—O3 −179.90 (15)  
O1—N1—C3—C2 0.28 (17)  C7—C8—C9—O3 1.0 (2)  
O1—N1—C3—C4 −178.93 (13)  O3—C9—C10—C11 −179.90 (14)  
C5—C2—C3—N1 179.09 (14)  C8—C9—C10—C11 −0.7 (2)  
C1—C2—C3—N1 −1.08 (17)  C9—C10—C11—C6 −0.7 (2)  
C5—C2—C3—C4 −1.8 (2)  C7—C6—C11—C10 1.8 (2)  
C1—C2—C3—C4 178.06 (14)  C5—C6—C11—C10 −178.97 (14)  
C3—C2—C5—C6 −179.81 (14)

**Hydrogen-bond geometry (Å, °)**

| D—H—A | D—H | H—A | D···A | D—H···A |
|--------|------|------|-------|---------|
| C11—H11···O2 | 0.93 | 2.15 | 2.989 (2) | 149 |
| O3—H3···O1^i | 0.86 (2) | 2.41 (2) | 2.9119 (18) | 118 (2) |
| O3—H3···N1^i | 0.86 (2) | 2.00 (2) | 2.7984 (19) | 154 (2) |
| C5—H5···O2^a | 0.93 | 2.47 | 3.3655 (17) | 162 |
| C7—H7···O2^a | 0.93 | 2.55 | 3.4038 (19) | 152 |

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