

Methyl 2-[(*E*)-8-oxo-5,8-dihydroquinolin-5-ylidene]hydrazino]benzoate

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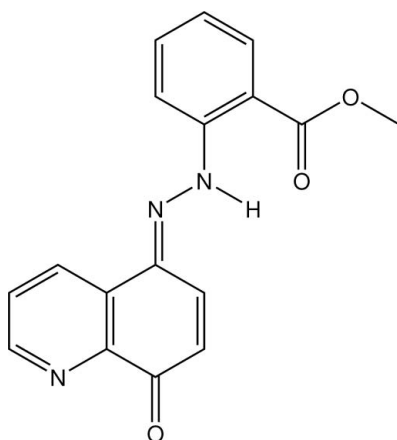
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Key indicators: single-crystal X-ray study; *T* = 98 K; mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$; *R* factor = 0.062; *wR* factor = 0.132; data-to-parameter ratio = 11.1.

The non-H atoms in the title compound, $\text{C}_{17}\text{H}_{13}\text{N}_3\text{O}_3$, are almost coplanar, a conformation stabilized by intramolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ interactions. A supramolecular chain mediated by $\text{C}-\text{H}\cdots\text{O}$ interactions is found in the crystal structure and these pack side-by-side into layers. The layers are consolidated into the crystal structure by further $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{N}$ interactions.

Related literature

For related literature, see: Sawicki (1957); Basu Baul, Mizar, Lyčka *et al.* (2006); Basu Baul, Mizar, Song *et al.* (2006). For a related structure, see: Basu Baul *et al.* (2005).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{13}\text{N}_3\text{O}_3$
 $M_r = 307.30$

Monoclinic, $C2/c$
 $a = 26.695 (3) \text{ \AA}$

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$b = 8.2513 (9) \text{ \AA}$
 $c = 13.5873 (16) \text{ \AA}$
 $\beta = 116.589 (5)^\circ$
 $V = 2676.3 (5) \text{ \AA}^3$
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 0.11 \text{ mm}^{-1}$
 $T = 98 (2) \text{ K}$
 $0.35 \times 0.13 \times 0.05 \text{ mm}$

Data collection

Rigaku AFC12K/SATURN724 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.798$, $T_{\max} = 1$
(expected range = 0.794–0.995)

4109 measured reflections
2329 independent reflections
2165 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.132$
 $S = 1.36$
2329 reflections

209 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.24 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
N3—H3N···O2	0.88	1.96	2.633 (3)	132
C12—H12···O3	0.95	2.32	2.662 (3)	101
C8—H8···O1 ⁱ	0.95	2.51	3.425 (3)	161
C17—H17B···O1 ⁱⁱ	0.98	2.49	3.227 (4)	132
C17—H17C···N1 ⁱⁱⁱ	0.98	2.59	3.379 (3)	138

Symmetry codes: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z - \frac{1}{2}$; (ii) $x + \frac{1}{2}, -y + \frac{5}{2}, z + \frac{1}{2}$; (iii) $-x + 1, y, -z + \frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku Americas Corporation, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2564).

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supplementary materials

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Methyl 2- $\{[(E)-8\text{-oxo-5,8-dihydroquinolin-5-ylidene}]hydrazino\}$ benzoate

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Comment

The title compound, C₁₇H₁₃O₃N₃ (I) was prepared during an on-going study of the coordination chemistry of organotin(IV) 5- $[(E)-2\text{-}((\text{aryl})\text{-1-diazenyl})\text{quinolin-8-olates}]$ (Basu Baul, Mizar, Lyčka *et al.* (2006); Basu Baul, Mizar, Song *et al.* (2006).

The crystal structure shows (I), (Fig. 1), to exist as the phenylhydrazone tautomer rather than in the azo form (Sawicki, 1957). The non-H atoms in (I) are effectively co-planar and the dihedral angle between the N1/C1—C9 and C10—C15 ring planes is 1.33 (10)°. An intramolecular N3—H \cdots O2 hydrogen bond contributes to the stability of the observed conformation; an intramolecular C12—H \cdots O3 interaction is also noted. Intermolecular C8—H \cdots O1 interactions lead to the formation of supramolecular chains aligned along the *b* axis (Fig. 2 & Table 1). These stack side-by-side to form layers and interactions between these layers are of the type C—H \cdots O and C—H \cdots N and involve the methyl groups (Fig. 3).

A very closely related molecule characterized in the tautomeric form shown in the Scheme has been observed previously in a dimeric dibenzyltin structure (Basu Baul *et al.*, 2005).

Experimental

Methyl anthranilate (5.0 g, 33.1 mmol) was mixed with HCl (11 ml) and water (11 ml) and digested in a water bath for 1 h. The hydrochloride was cooled to 278 K and diazotized with ice-cold aqueous NaNO₂ solution (5.0 g, 72.45 mmol, 25 ml). A cold solution of quinolin-8-ol (5.0 g, 34.4 mmol), previously dissolved in methanol solution (70 ml), was then added to the cold diazonium salt solution with vigorous stirring maintaining the temperature around 273 K. A light-orange colour developed and the stirring was continued for 1 h. A saturated solution of potassium acetate was then added to neutralize the hydrochloric acid, thereupon a deep-red precipitate appeared and stirring was continued for an additional hour. The reaction mixture was kept overnight in a refrigerator followed by 2 h at room temperature. The precipitate was filtered, washed several times with water to remove soluble starting materials, and then dried in air. The crude product was washed with hexane to remove any tarry materials, dried *in vacuo* and recrystallization from a methanol solution afforded orange microcrystalline (I) in 53.6% (5.67 g) yield. Red crystals (m.p. 434–435 K) of (I) suitable for an X-ray crystal structure determination were obtained from the slow evaporation of an ethylacetate/methanol (*v/v*, 1:1) solution. Elemental analysis, found: C 66.40, H 4.23, N 13.56%; C₁₇H₁₃O₃N₃ requires C 66.44, H 4.26, N 13.67%.

Refinement

All H atoms were included in the riding-model approximation, with N—H = 0.88 Å and C—H = 0.95 to 0.98 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ or $1.5U_{\text{eq}}(\text{methyl-C})$.

Figures

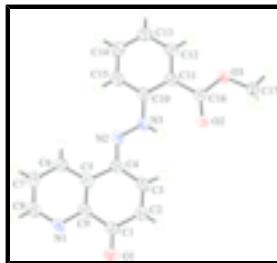


Fig. 1. The molecular structure of (I) showing displacement ellipsoids at the 50% probability level (arbitrary spheres for the H atoms).

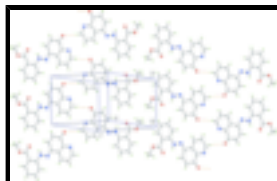


Fig. 2. View of the supramolecular chain in (I) mediated by hydrogen bonds, shown as orange-dashed lines. Colour code: red (oxygen), blue (nitrogen), grey (carbon) and green (hydrogen).

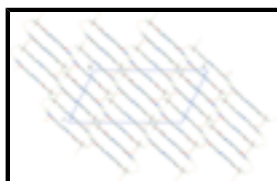


Fig. 3. Unit cell packing diagram in (I) highlighting the stacking of layers. Hydrogen bonds are shown as orange-dashed lines. Colour code in Fig. 2.

Methyl 2-[(*E*)-8-oxo-5,8-dihydroquinolin-5-ylidene]hydrazino}benzoate

Crystal data

$C_{17}H_{13}N_3O_3$

$M_r = 307.30$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 26.695 (3) \text{ \AA}$

$b = 8.2513 (9) \text{ \AA}$

$c = 13.5873 (16) \text{ \AA}$

$\beta = 116.589 (5)^\circ$

$V = 2676.3 (5) \text{ \AA}^3$

$Z = 8$

$F_{000} = 1280$

$D_x = 1.525 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 7939 reflections

$\theta = 2.6\text{--}29.6^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 98 (2) \text{ K}$

Prism, red

$0.35 \times 0.13 \times 0.05 \text{ mm}$

Data collection

Rigaku AFC12K/SATURN724
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 98(2) \text{ K}$

ω scans

Absorption correction: multi-scan
(ABSCOR; Higashi, 1995)

2329 independent reflections

2165 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.6^\circ$

$h = 0 \rightarrow 31$

$T_{\min} = 0.798$, $T_{\max} = 1$
4109 measured reflections

$k = -9 \rightarrow 9$
 $l = -16 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.062$	H-atom parameters constrained
$wR(F^2) = 0.132$	$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 4.7653P]$
$S = 1.36$	where $P = (F_o^2 + 2F_c^2)/3$
2329 reflections	$(\Delta/\sigma)_{\max} < 0.001$
209 parameters	$\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.34053 (7)	1.3572 (2)	-0.08723 (14)	0.0274 (4)
O2	0.61943 (7)	1.0578 (2)	0.29517 (13)	0.0204 (4)
O3	0.69937 (6)	0.9308 (2)	0.39955 (13)	0.0206 (4)
N1	0.30057 (8)	1.0510 (3)	-0.15349 (16)	0.0214 (5)
N2	0.48060 (8)	0.8551 (3)	0.09386 (16)	0.0196 (5)
N3	0.53313 (8)	0.8769 (3)	0.16930 (16)	0.0215 (5)
H3N	0.5460	0.9756	0.1903	0.026*
C1	0.37145 (10)	1.2430 (3)	-0.04737 (19)	0.0203 (5)
C2	0.42878 (10)	1.2680 (3)	0.0373 (2)	0.0252 (6)
H2	0.4414	1.3755	0.0604	0.030*
C3	0.46412 (10)	1.1451 (3)	0.0834 (2)	0.0254 (6)
H3	0.5011	1.1680	0.1378	0.030*
C4	0.44828 (9)	0.9807 (3)	0.05351 (19)	0.0189 (5)
C5	0.39130 (10)	0.9464 (3)	-0.02835 (18)	0.0187 (5)
C6	0.37130 (10)	0.7905 (3)	-0.05822 (19)	0.0214 (5)
H6	0.3953	0.7003	-0.0262	0.026*

supplementary materials

C7	0.31712 (10)	0.7666 (3)	-0.13366 (19)	0.0232 (6)
H7	0.3028	0.6601	-0.1546	0.028*
C8	0.28330 (10)	0.9001 (3)	-0.17926 (19)	0.0232 (6)
H8	0.2456	0.8822	-0.2320	0.028*
C9	0.35390 (10)	1.0730 (3)	-0.07851 (19)	0.0198 (5)
C10	0.56749 (10)	0.7457 (3)	0.21470 (19)	0.0203 (5)
C11	0.62287 (10)	0.7696 (3)	0.29446 (19)	0.0191 (5)
C12	0.65655 (10)	0.6352 (3)	0.3393 (2)	0.0220 (6)
H12	0.6940	0.6503	0.3941	0.026*
C13	0.63740 (11)	0.4825 (3)	0.3067 (2)	0.0273 (6)
H13	0.6611	0.3919	0.3387	0.033*
C14	0.58308 (11)	0.4604 (3)	0.2267 (2)	0.0317 (6)
H14	0.5697	0.3539	0.2025	0.038*
C15	0.54847 (10)	0.5896 (3)	0.1819 (2)	0.0277 (6)
H15	0.5110	0.5723	0.1279	0.033*
C16	0.64564 (10)	0.9334 (3)	0.32801 (19)	0.0180 (5)
C17	0.72608 (10)	1.0850 (3)	0.4336 (2)	0.0230 (5)
H17A	0.7156	1.1550	0.3691	0.034*
H17B	0.7668	1.0704	0.4703	0.034*
H17C	0.7141	1.1354	0.4849	0.034*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0231 (9)	0.0265 (10)	0.0283 (10)	0.0061 (8)	0.0075 (8)	0.0022 (8)
O2	0.0177 (8)	0.0177 (9)	0.0228 (9)	0.0024 (7)	0.0066 (7)	0.0004 (7)
O3	0.0147 (8)	0.0211 (9)	0.0202 (8)	-0.0003 (7)	0.0026 (7)	-0.0005 (7)
N1	0.0138 (10)	0.0327 (12)	0.0159 (10)	0.0013 (9)	0.0050 (8)	-0.0004 (9)
N2	0.0147 (10)	0.0246 (11)	0.0180 (10)	-0.0007 (9)	0.0062 (8)	-0.0007 (9)
N3	0.0143 (10)	0.0199 (11)	0.0247 (11)	-0.0008 (8)	0.0038 (8)	0.0003 (9)
C1	0.0169 (12)	0.0255 (13)	0.0197 (12)	0.0033 (11)	0.0093 (10)	0.0021 (10)
C2	0.0222 (13)	0.0220 (13)	0.0293 (14)	-0.0037 (11)	0.0094 (11)	-0.0024 (11)
C3	0.0173 (12)	0.0280 (14)	0.0235 (13)	-0.0019 (11)	0.0025 (10)	-0.0007 (11)
C4	0.0166 (12)	0.0208 (13)	0.0187 (12)	0.0011 (10)	0.0074 (10)	0.0024 (10)
C5	0.0178 (12)	0.0271 (14)	0.0136 (11)	-0.0016 (10)	0.0091 (10)	-0.0011 (10)
C6	0.0214 (13)	0.0238 (13)	0.0185 (12)	0.0003 (10)	0.0084 (10)	0.0005 (10)
C7	0.0240 (13)	0.0245 (13)	0.0193 (12)	-0.0062 (11)	0.0081 (10)	-0.0032 (10)
C8	0.0174 (12)	0.0335 (15)	0.0176 (12)	-0.0044 (11)	0.0069 (10)	-0.0035 (11)
C9	0.0149 (11)	0.0289 (14)	0.0155 (11)	0.0004 (10)	0.0067 (10)	-0.0009 (10)
C10	0.0173 (12)	0.0225 (13)	0.0207 (12)	0.0001 (10)	0.0081 (10)	0.0028 (10)
C11	0.0170 (12)	0.0215 (13)	0.0189 (11)	0.0003 (10)	0.0082 (10)	0.0006 (10)
C12	0.0190 (12)	0.0235 (14)	0.0212 (12)	0.0023 (10)	0.0069 (10)	0.0024 (10)
C13	0.0250 (14)	0.0199 (13)	0.0321 (14)	0.0040 (11)	0.0085 (12)	0.0050 (11)
C14	0.0273 (14)	0.0191 (13)	0.0420 (16)	-0.0034 (11)	0.0094 (12)	0.0007 (12)
C15	0.0184 (12)	0.0257 (14)	0.0326 (14)	-0.0021 (11)	0.0056 (11)	0.0003 (12)
C16	0.0176 (12)	0.0215 (13)	0.0154 (11)	0.0012 (10)	0.0079 (9)	0.0011 (10)
C17	0.0191 (12)	0.0214 (13)	0.0244 (13)	-0.0048 (10)	0.0061 (10)	-0.0018 (11)

Geometric parameters (Å, °)

O1—C1	1.209 (3)	C6—C7	1.362 (3)
O2—C16	1.210 (3)	C6—H6	0.9500
O3—C16	1.325 (3)	C7—C8	1.382 (4)
O3—C17	1.431 (3)	C7—H7	0.9500
N1—C8	1.320 (3)	C8—H8	0.9500
N1—C9	1.341 (3)	C10—C15	1.383 (4)
N2—N3	1.327 (3)	C10—C11	1.402 (3)
N2—C4	1.302 (3)	C11—C12	1.385 (3)
N3—C10	1.373 (3)	C11—C16	1.468 (3)
N3—H3N	0.8800	C12—C13	1.357 (4)
C1—C2	1.460 (3)	C12—H12	0.9500
C1—C9	1.479 (4)	C13—C14	1.381 (4)
C2—C3	1.335 (4)	C13—H13	0.9500
C2—H2	0.9500	C14—C15	1.363 (4)
C3—C4	1.424 (4)	C14—H14	0.9500
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.455 (3)	C17—H17A	0.9800
C5—C6	1.383 (4)	C17—H17B	0.9800
C5—C9	1.394 (3)	C17—H17C	0.9800
C16—O3—C17	116.24 (19)	N1—C9—C5	123.7 (2)
C8—N1—C9	117.1 (2)	N1—C9—C1	116.0 (2)
C4—N2—N3	119.3 (2)	C5—C9—C1	120.3 (2)
N2—N3—C10	120.2 (2)	N3—C10—C15	120.9 (2)
N2—N3—H3N	119.9	N3—C10—C11	119.8 (2)
C10—N3—H3N	119.9	C15—C10—C11	119.2 (2)
O1—C1—C2	120.5 (2)	C12—C11—C10	118.7 (2)
O1—C1—C9	123.1 (2)	C12—C11—C16	120.2 (2)
C2—C1—C9	116.4 (2)	C10—C11—C16	121.1 (2)
C3—C2—C1	122.2 (2)	C13—C12—C11	121.6 (2)
C3—C2—H2	118.9	C13—C12—H12	119.2
C1—C2—H2	118.9	C11—C12—H12	119.2
C2—C3—C4	122.2 (2)	C12—C13—C14	119.3 (2)
C2—C3—H3	118.9	C12—C13—H13	120.4
C4—C3—H3	118.9	C14—C13—H13	120.4
N2—C4—C3	125.6 (2)	C15—C14—C13	120.7 (2)
N2—C4—C5	115.8 (2)	C15—C14—H14	119.6
C3—C4—C5	118.6 (2)	C13—C14—H14	119.6
C6—C5—C9	117.1 (2)	C14—C15—C10	120.5 (2)
C6—C5—C4	122.7 (2)	C14—C15—H15	119.8
C9—C5—C4	120.2 (2)	C10—C15—H15	119.8
C7—C6—C5	119.7 (2)	O2—C16—O3	122.8 (2)
C7—C6—H6	120.1	O2—C16—C11	125.2 (2)
C5—C6—H6	120.1	O3—C16—C11	112.0 (2)
C6—C7—C8	118.9 (2)	O3—C17—H17A	109.5
C6—C7—H7	120.6	O3—C17—H17B	109.5
C8—C7—H7	120.6	H17A—C17—H17B	109.5

supplementary materials

N1—C8—C7	123.5 (2)	O3—C17—H17C	109.5
N1—C8—H8	118.2	H17A—C17—H17C	109.5
C7—C8—H8	118.2	H17B—C17—H17C	109.5
C4—N2—N3—C10	178.5 (2)	O1—C1—C9—N1	-1.1 (3)
O1—C1—C2—C3	179.9 (2)	C2—C1—C9—N1	177.7 (2)
C9—C1—C2—C3	1.1 (4)	O1—C1—C9—C5	-179.1 (2)
C1—C2—C3—C4	-0.3 (4)	C2—C1—C9—C5	-0.3 (3)
N3—N2—C4—C3	-0.1 (4)	N2—N3—C10—C15	0.9 (4)
N3—N2—C4—C5	-179.75 (19)	N2—N3—C10—C11	-179.9 (2)
C2—C3—C4—N2	179.2 (2)	N3—C10—C11—C12	179.7 (2)
C2—C3—C4—C5	-1.2 (4)	C15—C10—C11—C12	-1.0 (4)
N2—C4—C5—C6	3.1 (3)	N3—C10—C11—C16	-2.1 (3)
C3—C4—C5—C6	-176.5 (2)	C15—C10—C11—C16	177.1 (2)
N2—C4—C5—C9	-178.3 (2)	C10—C11—C12—C13	0.9 (4)
C3—C4—C5—C9	2.0 (3)	C16—C11—C12—C13	-177.3 (2)
C9—C5—C6—C7	0.0 (3)	C11—C12—C13—C14	0.3 (4)
C4—C5—C6—C7	178.5 (2)	C12—C13—C14—C15	-1.3 (4)
C5—C6—C7—C8	0.3 (4)	C13—C14—C15—C10	1.1 (4)
C9—N1—C8—C7	-0.3 (4)	N3—C10—C15—C14	179.3 (3)
C6—C7—C8—N1	-0.2 (4)	C11—C10—C15—C14	0.1 (4)
C8—N1—C9—C5	0.6 (3)	C17—O3—C16—O2	-2.4 (3)
C8—N1—C9—C1	-177.3 (2)	C17—O3—C16—C11	177.35 (19)
C6—C5—C9—N1	-0.5 (3)	C12—C11—C16—O2	-178.7 (2)
C4—C5—C9—N1	-179.1 (2)	C10—C11—C16—O2	3.2 (4)
C6—C5—C9—C1	177.4 (2)	C12—C11—C16—O3	1.6 (3)
C4—C5—C9—C1	-1.2 (3)	C10—C11—C16—O3	-176.5 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N3—H3N...O2	0.88	1.96	2.633 (3)	132
C12—H12...O3	0.95	2.32	2.662 (3)	101
C8—H8...O1 ⁱ	0.95	2.51	3.425 (3)	161
C17—H17B...O1 ⁱⁱ	0.98	2.49	3.227 (4)	132
C17—H17C...N1 ⁱⁱⁱ	0.98	2.59	3.379 (3)	138

Symmetry codes: (i) $-x+1/2, y-1/2, -z-1/2$; (ii) $x+1/2, -y+5/2, z+1/2$; (iii) $-x+1, y, -z+1/2$.

Fig. 1

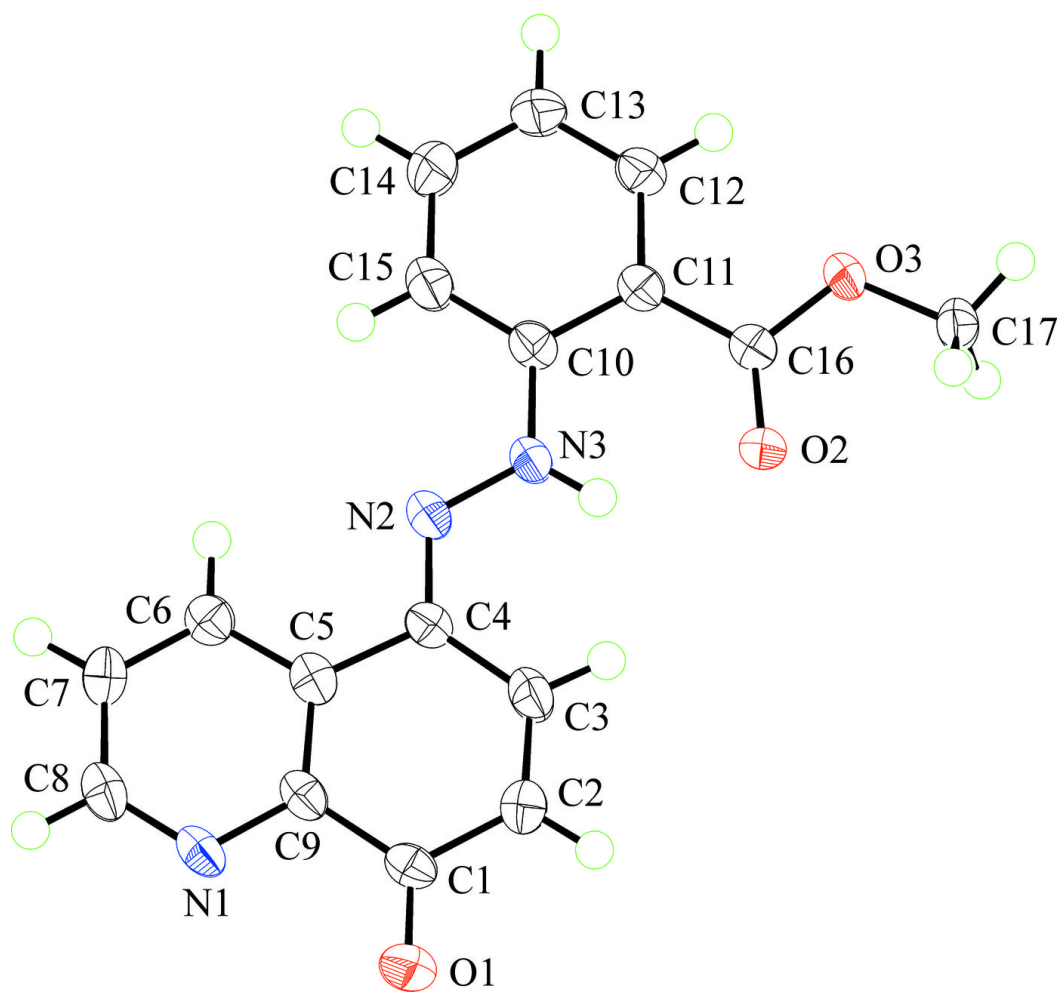


Fig. 2

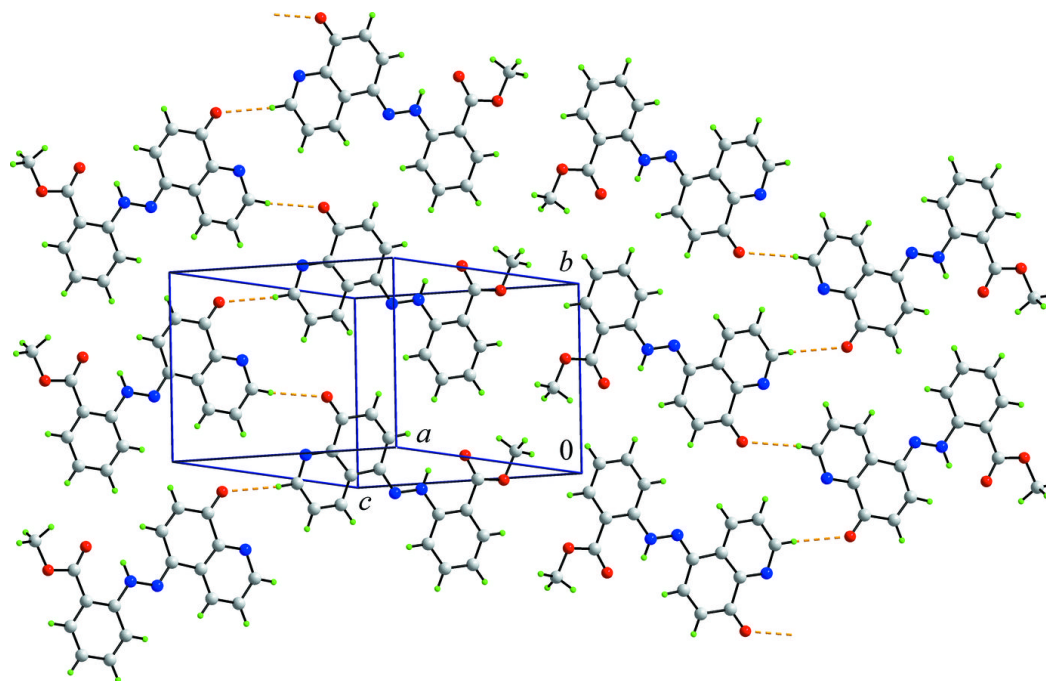


Fig. 3

