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3-Ethyl 1-methyl pyrrolo[2,1-*a*]-isoquinoline-1,3-dicarboxylate

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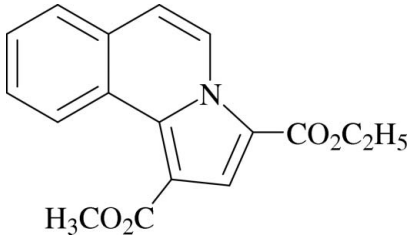
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.101; data-to-parameter ratio = 18.0.

In the molecular structure of the title compound, $C_{17}H_{15}NO_4$, the pyrrolo[2,1-*a*]isoquinoline unit is planar. The crystal structure is stabilized by weak intra- and intermolecular C—H···O interactions, and also by π–π interactions with centroid–centroid distances of 3.5680–3.6683 Å.

Related literature

For reference values of bond lengths, see: Allen *et al.* (1987). For related literature on ring motifs, see: Bernstein *et al.* (1995). For examples of related structures, see: Usman *et al.* (2002); Shen *et al.* (2006); Wang (2006*a*,*b*); Liu *et al.* (2007). For related literature on indolizine derivatives and activities, see, for example: Basketter & Plunkett (1971); Saeva & Luss (1988); Tominaga *et al.* (1990); Gundersen *et al.* (2007).



Experimental

Crystal data

$C_{17}H_{15}NO_4$
M_r = 297.30
Monoclinic, *Cc*
a = 20.5388 (4) Å
b = 4.4550 (1) Å
c = 16.5764 (5) Å
β = 114.955 (2)°
V = 1375.14 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100.0 (1) K
0.53 × 0.27 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.948, T_{max} = 0.985
23077 measured reflections
3627 independent reflections
3404 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.101
S = 1.03
3627 reflections
201 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5*A*···O2	0.93	2.14	2.9691 (19)	148
C11—H11*A*···O4	0.93	2.27	2.8891 (16)	123
C14—H14*A*···O2^{i}	0.97	2.60	3.2930 (17)	129
C14—H14*B*···O4^{ii}	0.97	2.58	3.3817 (16)	140

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2179).

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supplementary materials

Acta Cryst. (2007). E**63**, o3434 [**doi:10.1107/S1600536807032357**]

3-Ethyl 1-methyl pyrrolo[2,1-*a*]isoquinoline-1,3-dicarboxylate

H.-K. Fun, S. Chantrapromma, Y. Liu and J.-H. Xu

Comment

Indolizines are found in several naturally occurring alkaloids with important biological activities and are important synthetic targets (Saeva & Luss, 1988). As an extension of our research on the direct one-pot syntheses of indolizine derivatives (Liu *et al.*, 2007), we have recently researched a general and versatile synthesis of indolizines and obtained the title compound, as one of the products. An *x*-ray crystallographic analysis was undertaken to elucidate its three-dimensional molecular and crystal structures.

The bond lengths and angles in the structure of the title compound (I) are within normal ranges (Allen *et al.*, 1987), and comparable with those in related structures (Usman *et al.*, 2002; Shen *et al.*, 2006; Wang, 2006*a*; 2006*b*; Liu *et al.*, 2007). In the title structure (Fig. 1), the pyrrolo[2,1-*a*]isoquinoline ring system [N1/C1–C12] is planar with the mean deviation of 0.006 (1) Å. The methoxycarbonyl group (O1/O2/C16/C17) is almost coplanar with the pyrrolo[2,1-*a*]isoquinoline ring. The plane of the methoxycarbonyl group is twisted about the C2—C16 bond by an angle of 8.00 (7)°. The ethoxycarbonyl group (O3/O4/C13–C15) is co-planarly attached at atom C12 of the pyrrole ring, as indicated by the torsion angles N1–C12–C13–O3 = −178.13 (10)° and C13–O3–C14–C15 = 179.39 (10)°. The planarity of the molecule is influenced by weak intramolecular C5—H5A···O2 and C11—H11A···O4 interactions (Fig. 1) which generate S(7) and S(6) ring motifs, respectively (Bernstein *et al.*, 1995). The dihedral angle between the planes of two carboxylate groups is 8.33 (6)°.

In the crystal packing of (I) in Fig. 2, the molecules are arranged into molecular sheets parallel to the *ac* plane and these molecular sheets are stacked along the *b* axis. π···π interactions are also presented in the crystal with the distances of $Cg_1\cdots Cg_2$ = 3.6683 (7) Å, $Cg_1\cdots Cg_3$ = 3.5680 (7)Å and $Cg_2\cdots Cg_3$ = 3.6514 (7)Å [symmetry codes; *x*, −1 + *y*, *z* and *x*, 1 + *y*, *z* for all π···π interactions]; Cg_1, Cg_2 and Cg_3 are the centroids of N1/C1–C3/C12, N1/C3–C4/C9–C11 and C4–C9 rings, respectively. The crystal is stabilized by weak intramolecular and intermolecular C—H···O interactions (Table 1) and further stabilized by π···π interactions.

Experimental

A mixture of *N*-(carboxyethyl)quinolinium bromide (1 mmol), methyl acrylate (3 mmol), TPCD (1.6 mmol) and sodium carbonate (2.5 mmol) in DMF (15 ml) was heated at 363 K for 4 h with magnetic stirring. The title compound (I) was isolated by column chromatography of the reaction mixture on silica gel in 60% yield. Single crystals of the title compound in colorless block shape were obtained by slow evaporation of a solution in petroleum ether/ethyl acetate (3:1, V/V), m.p. 404–406 K. Compound (I) has been reported before. [Basketter & Plunkett (1971); Tominaga *et al.*, (1990)]. However there is no *x*-ray structure published yet. We prepared (I) with the same starting materials as in Tominaga *et al.*, 1990, but under different reaction conditions and with the better yield. The spectroscopic and analytical data of (I) was found to be the same as the published data (Tominaga *et al.*, 1990).

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. 3573 Friedel pairs were merged as there is no significant anomalous dispersion to determine the absolute structure. The highest residual peak is located 0.68 Å from O2 and the deepest hole is located 0.67 Å from O2.

Figures

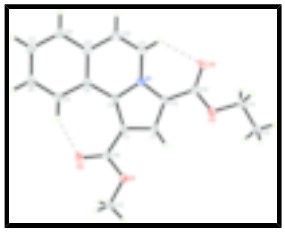

Fig. 1. The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Weak C—H···O intramolecular interactions were shown as dash lines.

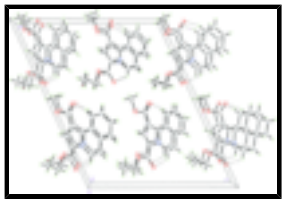

Fig. 2. The crystal packing of (I), viewed along the *b* axis. C—H···O weak interactions were shown as dash lines.

3-ethyl 1-methyl pyrrolo[2,1-*a*]isoquinoline-1,3-dicarboxylate

Crystal data

$C_{17}H_{15}NO_4$	F_{000} = 624
M_r = 297.30	D_x = 1.436 Mg m^{-3}
Monoclinic, *Cc*	Melting point: 404-406 K
Hall symbol: C -2yc	Mo *K*α radiation λ = 0.71073 Å
a = 20.5388 (4) Å	Cell parameters from 3627 reflections
b = 4.45500 (10) Å	θ = 2.2–37.5º
c = 16.5764 (5) Å	μ = 0.10 mm^{-1}
β = 114.955 (2)º	*T* = 100.0 (1) K
V = 1375.14 (6) Å^3	Block, colourless
Z = 4	0.53 × 0.27 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	3627 independent reflections
Radiation source: fine-focus sealed tube	3404 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044

Detector resolution: 8.33 pixels mm^{-1}

T = 100.0(1) K

ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

T_{min} = 0.948, T_{max} = 0.985

23077 measured reflections

θ_{max} = 37.5º

θ_{min} = 2.2º

h = −34→34

k = −7→7

l = −28→28

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.038

$wR(F^2)$ = 0.101

S = 1.03

3627 reflections

201 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0691P)^2 + 0.0494P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.42 e $Å^{-3}$

$\Delta\rho_{min}$ = −0.28 e $Å^{-3}$

Extinction correction: none

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.45336 (5)	−0.2384 (3)	0.04828 (6)	0.0244 (2)
O2	0.49888 (5)	0.0351 (4)	0.17187 (8)	0.0383 (3)
O3	0.19014 (4)	−0.5425 (2)	−0.07101 (6)	0.01815 (15)
O4	0.12973 (5)	−0.3018 (2)	−0.00418 (6)	0.02089 (16)
N1	0.26700 (5)	−0.0079 (2)	0.11014 (6)	0.01466 (15)
C1	0.31898 (5)	−0.2445 (2)	0.03391 (7)	0.01531 (16)
H1A	0.3262	−0.3652	−0.0074	0.018*
C2	0.37166 (5)	−0.0660 (3)	0.09913 (7)	0.01470 (16)

C3	0.33828 (5)	0.0848 (2)	0.14737 (7)	0.01413 (16)
C4	0.36033 (5)	0.2977 (3)	0.21988 (7)	0.01494 (16)
C5	0.43085 (6)	0.4134 (3)	0.26242 (7)	0.01794 (18)
H5A	0.4656	0.3528	0.2437	0.022*
C6	0.44863 (6)	0.6159 (3)	0.33164 (8)	0.0202 (2)
H6A	0.4953	0.6901	0.3588	0.024*
C7	0.39770 (7)	0.7114 (3)	0.36171 (8)	0.0213 (2)
H7A	0.4105	0.8472	0.4085	0.026*
C8	0.32840 (7)	0.6021 (3)	0.32128 (8)	0.0201 (2)
H8A	0.2944	0.6646	0.3411	0.024*
C9	0.30869 (6)	0.3964 (3)	0.25012 (7)	0.01647 (17)
C10	0.23655 (6)	0.2863 (3)	0.20788 (8)	0.01857 (18)
H10A	0.2028	0.3514	0.2277	0.022*
C11	0.21679 (6)	0.0901 (3)	0.13991 (8)	0.01766 (18)
H11A	0.1697	0.0206	0.1130	0.021*
C12	0.25490 (5)	−0.2107 (2)	0.04127 (7)	0.01502 (17)
C13	0.18567 (5)	−0.3499 (3)	−0.01099 (7)	0.01573 (17)
C14	0.12267 (6)	−0.6880 (3)	−0.12763 (8)	0.01850 (18)
H14A	0.0869	−0.5390	−0.1607	0.022*
H14B	0.1047	−0.8042	−0.0918	0.022*
C15	0.13787 (7)	−0.8902 (3)	−0.19044 (8)	0.0207 (2)
H15A	0.0969	−1.0153	−0.2220	0.031*
H15B	0.1788	−1.0139	−0.1572	0.031*
H15C	0.1477	−0.7704	−0.2322	0.031*
C16	0.44691 (6)	−0.0746 (3)	0.11232 (7)	0.01717 (18)
C17	0.52569 (7)	−0.2785 (4)	0.05696 (9)	0.0269 (3)
H17A	0.5248	−0.3920	0.0073	0.040*
H17B	0.5534	−0.3843	0.1111	0.040*
H17C	0.5471	−0.0859	0.0583	0.040*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0118 (3)	0.0404 (6)	0.0210 (4)	−0.0009 (3)	0.0068 (3)	−0.0103 (4)
O2	0.0145 (4)	0.0634 (9)	0.0361 (6)	−0.0096 (4)	0.0099 (4)	−0.0281 (6)
O3	0.0124 (3)	0.0199 (4)	0.0192 (3)	−0.0015 (3)	0.0037 (2)	−0.0022 (3)
O4	0.0123 (3)	0.0260 (4)	0.0239 (4)	−0.0014 (3)	0.0072 (3)	−0.0017 (3)
N1	0.0110 (3)	0.0175 (4)	0.0152 (3)	0.0005 (3)	0.0052 (3)	0.0006 (3)
C1	0.0116 (3)	0.0187 (4)	0.0144 (4)	−0.0003 (3)	0.0043 (3)	0.0001 (3)
C2	0.0111 (3)	0.0183 (4)	0.0137 (4)	−0.0006 (3)	0.0043 (3)	−0.0002 (3)
C3	0.0116 (3)	0.0161 (4)	0.0142 (4)	0.0006 (3)	0.0049 (3)	0.0014 (3)
C4	0.0136 (4)	0.0162 (4)	0.0138 (4)	0.0006 (3)	0.0047 (3)	0.0005 (3)
C5	0.0157 (4)	0.0196 (5)	0.0164 (4)	−0.0008 (3)	0.0048 (3)	−0.0006 (3)
C6	0.0186 (4)	0.0216 (5)	0.0162 (4)	−0.0012 (4)	0.0034 (3)	−0.0018 (3)
C7	0.0240 (5)	0.0215 (5)	0.0161 (4)	0.0015 (4)	0.0062 (4)	−0.0016 (4)
C8	0.0223 (5)	0.0204 (5)	0.0173 (4)	0.0028 (4)	0.0081 (4)	0.0001 (3)
C9	0.0168 (4)	0.0169 (4)	0.0158 (4)	0.0016 (3)	0.0070 (3)	0.0015 (3)
C10	0.0162 (4)	0.0202 (5)	0.0209 (4)	0.0016 (3)	0.0094 (4)	−0.0004 (4)

C11	0.0136 (4)	0.0202 (5)	0.0204 (5)	0.0011 (3)	0.0084 (3)	0.0001 (3)
C12	0.0111 (3)	0.0175 (4)	0.0149 (4)	−0.0004 (3)	0.0040 (3)	0.0003 (3)
C13	0.0124 (4)	0.0169 (4)	0.0157 (4)	0.0001 (3)	0.0038 (3)	0.0018 (3)
C14	0.0133 (4)	0.0192 (5)	0.0194 (4)	−0.0029 (3)	0.0033 (3)	−0.0013 (3)
C15	0.0199 (4)	0.0212 (5)	0.0189 (5)	−0.0027 (4)	0.0062 (4)	−0.0011 (4)
C16	0.0120 (3)	0.0241 (5)	0.0152 (4)	−0.0010 (3)	0.0056 (3)	−0.0017 (3)
C17	0.0137 (4)	0.0451 (8)	0.0230 (5)	0.0001 (4)	0.0089 (4)	−0.0068 (5)

Geometric parameters (Å, °)

O1—C16	1.3401 (14)	C6—H6A	0.9300
O1—C17	1.4419 (14)	C7—C8	1.3810 (18)
O2—C16	1.2097 (14)	C7—H7A	0.9300
O3—C13	1.3458 (14)	C8—C9	1.4114 (16)
O3—C14	1.4574 (13)	C8—H8A	0.9300
O4—C13	1.2199 (13)	C9—C10	1.4322 (16)
N1—C11	1.3882 (14)	C10—C11	1.3462 (17)
N1—C3	1.3900 (13)	C10—H10A	0.9300
N1—C12	1.3935 (14)	C11—H11A	0.9300
C1—C12	1.3803 (14)	C12—C13	1.4551 (15)
C1—C2	1.4088 (15)	C14—C15	1.5058 (17)
C1—H1A	0.9300	C14—H14A	0.9700
C2—C3	1.4224 (15)	C14—H14B	0.9700
C2—C16	1.4673 (14)	C15—H15A	0.9600
C3—C4	1.4457 (15)	C15—H15B	0.9600
C4—C5	1.4137 (15)	C15—H15C	0.9600
C4—C9	1.4207 (15)	C17—H17A	0.9600
C5—C6	1.3819 (16)	C17—H17B	0.9600
C5—H5A	0.9300	C17—H17C	0.9600
C6—C7	1.4014 (18)		
C16—O1—C17	115.50 (9)	C11—C10—H10A	119.5
C13—O3—C14	114.63 (9)	C9—C10—H10A	119.5
C11—N1—C3	123.35 (9)	C10—C11—N1	119.67 (10)
C11—N1—C12	126.35 (9)	C10—C11—H11A	120.2
C3—N1—C12	110.29 (8)	N1—C11—H11A	120.2
C12—C1—C2	108.84 (10)	C1—C12—N1	107.21 (9)
C12—C1—H1A	125.6	C1—C12—C13	129.03 (10)
C2—C1—H1A	125.6	N1—C12—C13	123.76 (9)
C1—C2—C3	107.56 (9)	O4—C13—O3	122.75 (10)
C1—C2—C16	121.27 (10)	O4—C13—C12	126.30 (11)
C3—C2—C16	130.99 (10)	O3—C13—C12	110.94 (9)
N1—C3—C2	106.09 (9)	O3—C14—C15	107.05 (9)
N1—C3—C4	117.78 (9)	O3—C14—H14A	110.3
C2—C3—C4	136.13 (9)	C15—C14—H14A	110.3
C5—C4—C9	118.12 (10)	O3—C14—H14B	110.3
C5—C4—C3	123.35 (10)	C15—C14—H14B	110.3
C9—C4—C3	118.53 (9)	H14A—C14—H14B	108.6
C6—C5—C4	120.56 (11)	C14—C15—H15A	109.5
C6—C5—H5A	119.7	C14—C15—H15B	109.5

C4—C5—H5A	119.7	H15A—C15—H15B	109.5
C5—C6—C7	121.20 (11)	C14—C15—H15C	109.5
C5—C6—H6A	119.4	H15A—C15—H15C	109.5
C7—C6—H6A	119.4	H15B—C15—H15C	109.5
C8—C7—C6	119.44 (11)	O2—C16—O1	121.21 (10)
C8—C7—H7A	120.3	O2—C16—C2	128.49 (11)
C6—C7—H7A	120.3	O1—C16—C2	110.26 (9)
C7—C8—C9	120.53 (11)	O1—C17—H17A	109.5
C7—C8—H8A	119.7	O1—C17—H17B	109.5
C9—C8—H8A	119.7	H17A—C17—H17B	109.5
C8—C9—C4	120.15 (10)	O1—C17—H17C	109.5
C8—C9—C10	120.25 (10)	H17A—C17—H17C	109.5
C4—C9—C10	119.60 (10)	H17B—C17—H17C	109.5
C11—C10—C9	121.06 (10)		
C12—C1—C2—C3	−0.68 (12)	C8—C9—C10—C11	179.98 (11)
C12—C1—C2—C16	174.97 (10)	C4—C9—C10—C11	0.09 (17)
C11—N1—C3—C2	179.63 (10)	C9—C10—C11—N1	0.08 (17)
C12—N1—C3—C2	0.30 (11)	C3—N1—C11—C10	0.38 (17)
C11—N1—C3—C4	−0.95 (15)	C12—N1—C11—C10	179.60 (11)
C12—N1—C3—C4	179.72 (9)	C2—C1—C12—N1	0.86 (12)
C1—C2—C3—N1	0.23 (12)	C2—C1—C12—C13	−179.75 (10)
C16—C2—C3—N1	−174.85 (11)	C11—N1—C12—C1	179.97 (10)
C1—C2—C3—C4	−179.03 (12)	C3—N1—C12—C1	−0.72 (12)
C16—C2—C3—C4	5.9 (2)	C11—N1—C12—C13	0.54 (17)
N1—C3—C4—C5	−178.91 (10)	C3—N1—C12—C13	179.84 (9)
C2—C3—C4—C5	0.29 (19)	C14—O3—C13—O4	0.30 (15)
N1—C3—C4—C9	1.06 (14)	C14—O3—C13—C12	−178.83 (9)
C2—C3—C4—C9	−179.74 (11)	C1—C12—C13—O4	−176.52 (12)
C9—C4—C5—C6	0.23 (16)	N1—C12—C13—O4	2.79 (18)
C3—C4—C5—C6	−179.79 (11)	C1—C12—C13—O3	2.57 (16)
C4—C5—C6—C7	0.17 (18)	N1—C12—C13—O3	−178.13 (10)
C5—C6—C7—C8	−0.23 (19)	C13—O3—C14—C15	179.39 (10)
C6—C7—C8—C9	−0.12 (18)	C17—O1—C16—O2	1.39 (19)
C7—C8—C9—C4	0.53 (17)	C17—O1—C16—C2	−176.48 (12)
C7—C8—C9—C10	−179.36 (11)	C1—C2—C16—O2	−169.91 (15)
C5—C4—C9—C8	−0.57 (15)	C3—C2—C16—O2	4.6 (2)
C3—C4—C9—C8	179.45 (10)	C1—C2—C16—O1	7.77 (15)
C5—C4—C9—C10	179.32 (10)	C3—C2—C16—O1	−177.72 (11)
C3—C4—C9—C10	−0.66 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5A···O2	0.93	2.14	2.9691 (19)	148
C11—H11A···O4	0.93	2.27	2.8891 (16)	123
C14—H14A···O2^{i}	0.97	2.60	3.2930 (17)	129
C14—H14B···O4^{ii}	0.97	2.58	3.3817 (16)	140

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

Fig. 1

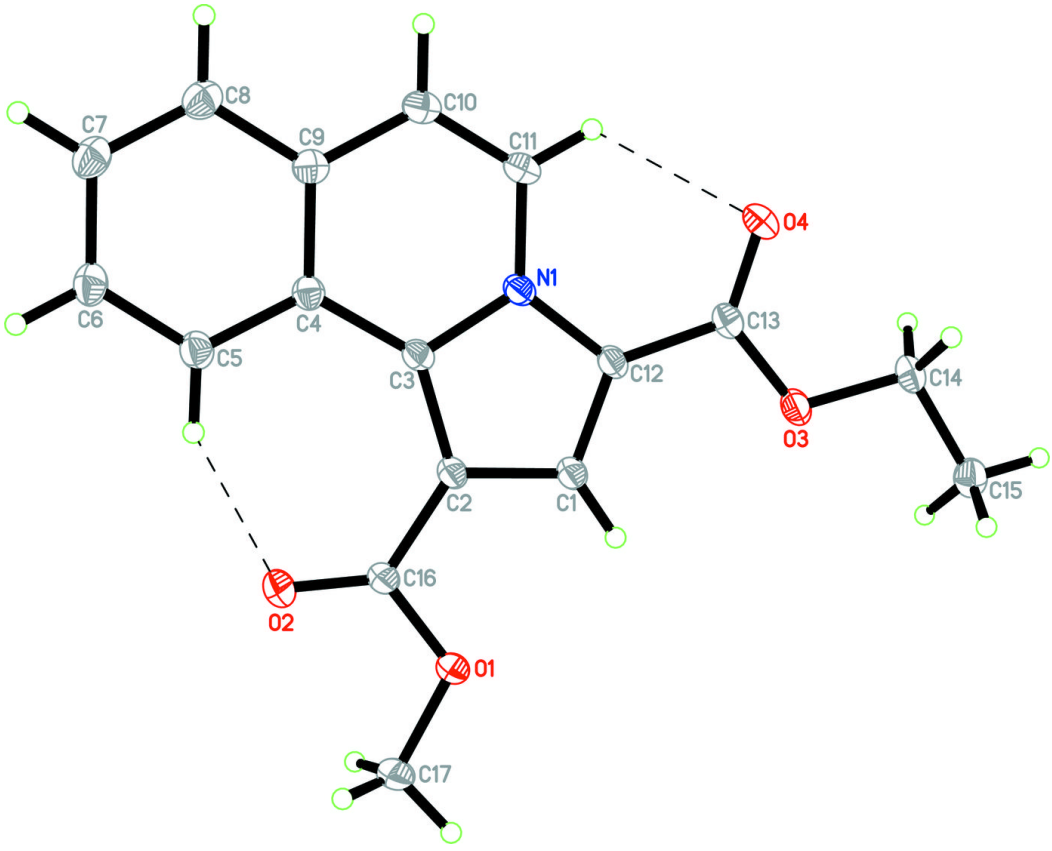
C8
C7
C9
C10
C11
O4
N1
C6
C4
C13
C5
C3
C12
C14
O3
C2
C1
C15
C16
O2
O1
C17

Fig. 2