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4-Methylphenyl quinoline-2-carboxylate

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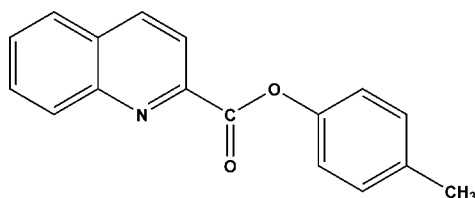
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 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.124; data-to-parameter ratio = 14.5.

In the title compound, $\text{C}_{17}\text{H}_{13}\text{NO}_2$, two molecules crystallize in the asymmetric unit. The dihedral angle between the mean planes of the quinoline and benzene rings are 78.3 (4) and 88.2 (3)°. The carboxylate group is twisted slightly from the quinoline ring by 7.1 (2) and 13.3 (4)°, respectively. In the crystal, weak $\text{C}-\text{H}\cdots\text{O}$ interactions are observed. Further stabilization is provided by weak $\pi-\pi$ stacking interactions, with centroid-centroid distances of 3.564 (9)/ 3.689 (2) and 3.830 (1)/ 3.896 (5) Å, respectively.

Related literature

For heterocycles in natural products, see: Morimoto *et al.* (1991); Michael (1997). For heterocycles in fragrances and dyes, see: Padwa *et al.* (1999). For heterocycles in biologically active compounds, see: Markees *et al.* (1970); Campbell *et al.* (1988). For quinoline alkaloids used as efficient drugs for the treatment of malaria, see: Robert & Meunier, (1998). For quinoline as a privileged scaffold in cancer drug discovery, see: Solomon & Lee (2011). For related structures, see: Dobrzyńska & Jerzykiewicz, (2004); Butcher *et al.* (2007); Jing & Qin (2008); Jasinski *et al.* (2010). For bond lengths, see Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{13}\text{NO}_2$
 $M_r = 263.28$
 Orthorhombic, $Pbca$
 $a = 11.5421$ (2) Å
 $b = 17.3191$ (3) Å
 $c = 26.6667$ (5) Å
 $V = 5330.65$ (16) Å³
 $Z = 16$
 Cu $K\alpha$ radiation
 $\mu = 0.70$ mm⁻¹
 $T = 173$ K
 $0.22 \times 0.14 \times 0.12$ mm

Data collection

Oxford Diffraction Xcalibur (Eos, Gemini) diffractometer
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
 $T_{\min} = 0.726$, $T_{\max} = 1.000$
 34626 measured reflections
 5265 independent reflections
 4303 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.124$
 $S = 1.02$
 5265 reflections
 363 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C15B}-\text{H15B}\cdots\text{O2A}^i$ | 0.93 | 2.59 | 3.343 (2) | 138 |

 Symmetry code: (i) $x + \frac{1}{2}, y, -z + \frac{1}{2}$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

EF thanks Yuvaraja's college, UOM for providing the research facilities and also to Dr. S. Nagarajan, Senior Scientist at CFTRI for giving valuable suggestions. JPJ acknowledges the NSF-MRI program (grant No-CHE1039027) for funds to purchase the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2377).

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supporting information

Acta Cryst. (2012). E68, o3231–o3232 [doi:10.1107/S1600536812044030]

4-Methylphenyl quinoline-2-carboxylate

E. Fazal, Jerry P. Jasinski, Shannon T. Krauss, B. S. Sudha and H. S. Yathirajan

S1. Comment

Quinoline-2-carboxylic acid derivatives are a class of important materials as anti-tuberculosis agents, as fluorescent reagents, hydrophobic field-detection reagents, visualization reagents, fluorescent labeled peptide probes and as antihyperglycemics. Quinoline derivatives represent a major class of heterocycles and are found in natural products (Morimoto *et al.*, 1991; Michael, 1997), numerous commercial products, including fragrances, dyes (Padwa *et al.*, 1999) and biologically active compounds (Markees *et al.*, 1970; Campbell *et al.*, 1988). Quinoline alkaloids such as quinine, chloroquin, mefloquine and amodiaquine are used as efficient drugs for the treatment of malaria (Robert & Meunier, 1998). Quinoline as a privileged scaffold in cancer drug discovery is published (Solomon & Lee, 2011). The crystal structures of quinoline-2-carboxylic acid (Dobrzyńska & Jerzykiewicz, 2004), 1-(quinolin-2-yl)ethanone (Butcher *et al.*, 2007) and methyl quinoline-2-carboxylate (Jing & Qin, 2008) and the synthesis, crystal structures and theoretical studies of four Schiff bases derived from 4-hydrazinyl-8-(trifluoromethyl) quinoline (Jasinski *et al.*, 2010) have been reported. In view of the importance of quinolines, the paper reports the crystal structure of the title compound, 4-methylphenyl quinoline-2-carboxylate, (I).

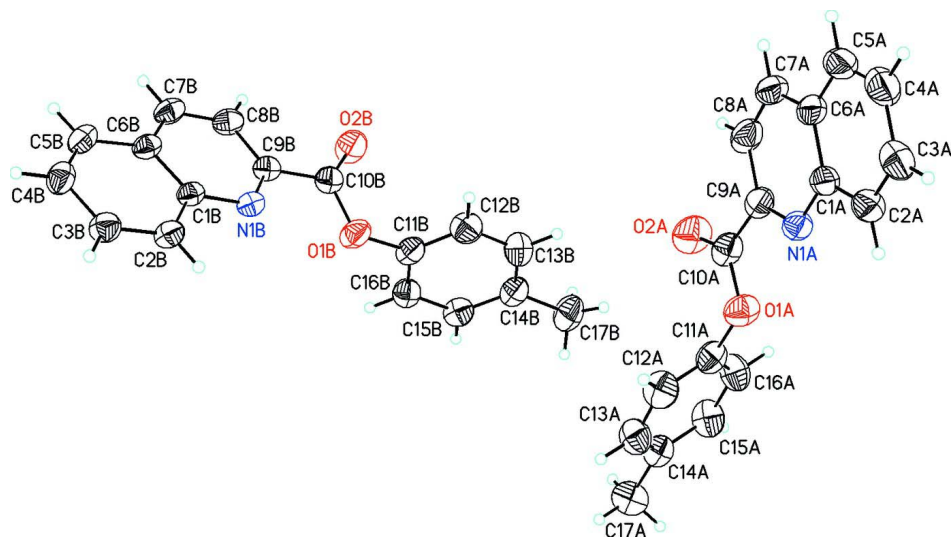
In the title compound, $C_{17}H_{13}NO_2$, two molecules (A & B) crystallize in the asymmetric unit (Fig. 1). The dihedral angle between the mean planes of the quinoline and benzene rings are $78.3(4)^\circ$ (A) and $88.2(3)^\circ$ (B). The carboxylate group is twisted slightly from the quinoline ring by $7.1(2)^\circ$ (A) and $13.3(4)^\circ$ (B), respectively. Bond lengths are in normal ranges (Allen *et al.*, (1987). In the crystal weak C—H \cdots O intermolecular interactions are observed (Fig. 2). Further stabilization is provided by weak π – π stacking interactions with centroid to centroid distances of $3.564(9)\text{\AA}$ (Cg2–Cg1), $3.689(2)\text{\AA}$ (Cg2–Cg6), $3.830(1)\text{\AA}$ (Cg1–Cg5) and $3.896(5)\text{\AA}$ (Cg1–Cg1) [where Cg1 = N1A/C1A/C6A/C7A/C8A/C9A; Cg2 = C1A–C6A; Cg5 = N1B/C1B/C6B/C7B/C8B/C9B; C6 = C1B–C6B].

S2. Experimental

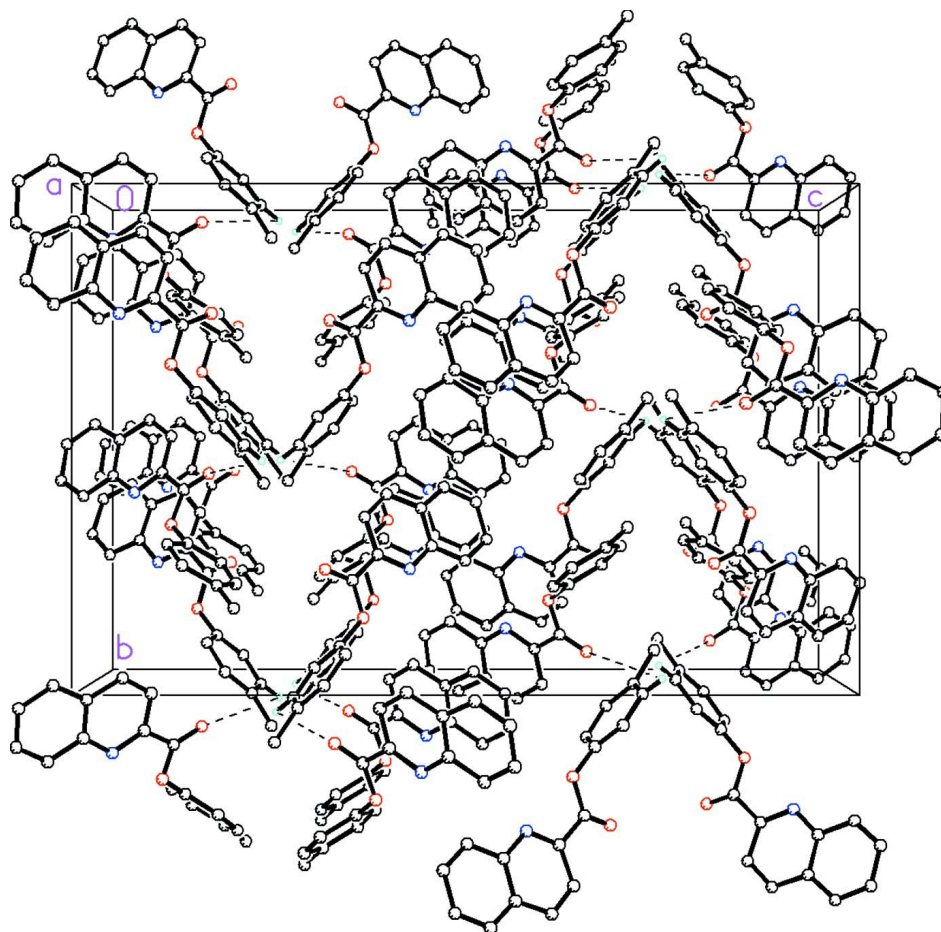
To a mixture of (1.73 g, 10 mmole) of quinaldic acid and p-cresol (1.08 g, 10 mmole) in a round-bottomed flask fitted with a reflux condenser with a drying tube, 0.75 g (5 mmole) of phosphorous oxychloride was added. The mixture was heated with occasional swirling, and temperature maintained at 348–353 K. At the end of six hours, the reaction mixture was poured into a solution of 2 g of sodium bicarbonate in 25 mL of water. The precipitated ester was filtered and washed with water. The yield of crude, air dried p-tolyl quinoline-2-carboxylate was 1.75 to 1.85 g (65–70%). X-ray quality crystals were obtained by recrystallization from absolute ethanol (m.p.: 396–398 K).

S3. Refinement

All of the H atoms were placed in their calculated positions and then refined using the riding model with Atom—H lengths of 0.93\AA (CH) or 0.96\AA (CH₃). Isotropic displacement parameters for these atoms were set to 1.19–1.21 (CH) or 1.50 (CH₃) times U_{eq} of the parent atom.

**Figure 1**

Molecular structure of the title compound showing the atom labeling scheme of two molecules (A & B) in the asymmetric unit and 50% probability displacement ellipsoids.

**Figure 2**

Packing diagram of the title compound viewed along the *a* axis. Dashed lines indicate weak C—H...O intermolecular interactions. The remaining H atoms have been removed for clarity.

4-Methylphenyl quinoline-2-carboxylate

Crystal data

$C_{17}H_{13}NO_2$

$M_r = 263.28$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 11.5421$ (2) Å

$b = 17.3191$ (3) Å

$c = 26.6667$ (5) Å

$V = 5330.65$ (16) Å³

$Z = 16$

$F(000) = 2208$

$D_x = 1.312$ Mg m⁻³

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

Cell parameters from 10490 reflections

$\theta = 3.8$ – 72.7°

$\mu = 0.70$ mm⁻¹

$T = 173$ K

Chunk, colorless

$0.22 \times 0.14 \times 0.12$ mm

Data collection

Oxford Diffraction Xcalibur (Eos, Gemini)
diffractometer

Radiation source: Enhance (Cu) X-ray Source

Graphite monochromator

Detector resolution: 16.0416 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO* and *CrysAlis RED*; Oxford
Diffraction, 2010)

$T_{\min} = 0.726$, $T_{\max} = 1.000$

34626 measured reflections
 5265 independent reflections
 4303 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

$\theta_{\text{max}} = 72.8^\circ$, $\theta_{\text{min}} = 4.9^\circ$
 $h = -14 \rightarrow 10$
 $k = -20 \rightarrow 21$
 $l = -32 \rightarrow 31$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.124$
 $S = 1.02$
 5265 reflections
 363 parameters
 0 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
 $w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 1.783P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.19 \text{ e } \text{Å}^{-3}$

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 $F^2 > \sigma(F^2)$ 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)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|--------------|-------------|----------------------------------|
| O1A | 0.12216 (11) | 0.67251 (7) | 0.39057 (4) | 0.0511 (3) |
| O2A | 0.13223 (13) | 0.56857 (9) | 0.34041 (5) | 0.0657 (4) |
| N1A | -0.03742 (11) | 0.60902 (8) | 0.44686 (5) | 0.0390 (3) |
| C1A | -0.11347 (13) | 0.57558 (9) | 0.47931 (6) | 0.0396 (4) |
| C2A | -0.14961 (14) | 0.61816 (10) | 0.52172 (6) | 0.0450 (4) |
| H2A | -0.1244 | 0.6688 | 0.5259 | 0.054* |
| C3A | -0.22121 (15) | 0.58568 (11) | 0.55662 (7) | 0.0525 (4) |
| H3A | -0.2437 | 0.6142 | 0.5845 | 0.063* |
| C4A | -0.26120 (16) | 0.50963 (12) | 0.55081 (8) | 0.0578 (5) |
| H4A | -0.3097 | 0.4880 | 0.5749 | 0.069* |
| C5A | -0.22915 (15) | 0.46757 (11) | 0.51017 (8) | 0.0569 (5) |
| H5A | -0.2561 | 0.4172 | 0.5067 | 0.068* |
| C6A | -0.15524 (14) | 0.49894 (9) | 0.47282 (7) | 0.0464 (4) |
| C7A | -0.11881 (16) | 0.45862 (10) | 0.43001 (8) | 0.0537 (5) |
| H7A | -0.1462 | 0.4090 | 0.4238 | 0.064* |
| C8A | -0.04317 (17) | 0.49246 (10) | 0.39768 (7) | 0.0526 (5) |
| H8A | -0.0185 | 0.4664 | 0.3691 | 0.063* |
| C9A | -0.00251 (14) | 0.56800 (9) | 0.40816 (6) | 0.0422 (4) |
| C10A | 0.08924 (16) | 0.60161 (10) | 0.37521 (6) | 0.0455 (4) |
| C11A | 0.21459 (15) | 0.70870 (10) | 0.36529 (6) | 0.0447 (4) |
| C12A | 0.32213 (17) | 0.70819 (12) | 0.38707 (7) | 0.0555 (5) |

| | | | | |
|------|--------------|--------------|-------------|------------|
| H12A | 0.3348 | 0.6815 | 0.4168 | 0.067* |
| C13A | 0.41123 (16) | 0.74779 (11) | 0.36423 (7) | 0.0534 (4) |
| H13A | 0.4842 | 0.7475 | 0.3790 | 0.064* |
| C14A | 0.39551 (15) | 0.78789 (10) | 0.32009 (6) | 0.0452 (4) |
| C15A | 0.28520 (17) | 0.78769 (12) | 0.29953 (7) | 0.0553 (5) |
| H15A | 0.2719 | 0.8147 | 0.2699 | 0.066* |
| C16A | 0.19469 (16) | 0.74867 (12) | 0.32171 (7) | 0.0535 (5) |
| H16A | 0.1212 | 0.7494 | 0.3074 | 0.064* |
| C17A | 0.49506 (18) | 0.82914 (13) | 0.29514 (7) | 0.0600 (5) |
| H17D | 0.5132 | 0.8040 | 0.2640 | 0.090* |
| H17E | 0.5616 | 0.8278 | 0.3167 | 0.090* |
| H17F | 0.4738 | 0.8818 | 0.2888 | 0.090* |
| O1B | 0.66824 (11) | 0.35457 (6) | 0.36695 (4) | 0.0466 (3) |
| O2B | 0.60648 (11) | 0.25607 (7) | 0.31907 (5) | 0.0540 (3) |
| N1B | 0.78323 (10) | 0.26045 (7) | 0.42615 (4) | 0.0322 (3) |
| C1B | 0.85486 (12) | 0.21554 (8) | 0.45442 (5) | 0.0309 (3) |
| C2B | 0.90207 (13) | 0.24698 (9) | 0.49877 (6) | 0.0365 (3) |
| H2B | 0.8820 | 0.2968 | 0.5085 | 0.044* |
| C3B | 0.97676 (14) | 0.20498 (9) | 0.52742 (6) | 0.0396 (3) |
| H3B | 1.0079 | 0.2265 | 0.5564 | 0.048* |
| C4B | 1.00717 (14) | 0.12907 (9) | 0.51339 (6) | 0.0409 (4) |
| H4B | 1.0576 | 0.1007 | 0.5334 | 0.049* |
| C5B | 0.96322 (14) | 0.09699 (9) | 0.47076 (6) | 0.0392 (4) |
| H5B | 0.9842 | 0.0470 | 0.4618 | 0.047* |
| C6B | 0.88617 (13) | 0.13903 (8) | 0.44012 (5) | 0.0330 (3) |
| C7B | 0.83864 (14) | 0.11000 (9) | 0.39521 (6) | 0.0385 (3) |
| H7B | 0.8567 | 0.0603 | 0.3846 | 0.046* |
| C8B | 0.76635 (14) | 0.15486 (9) | 0.36753 (6) | 0.0385 (3) |
| H8B | 0.7338 | 0.1363 | 0.3380 | 0.046* |
| C9B | 0.74165 (12) | 0.23042 (8) | 0.38463 (5) | 0.0332 (3) |
| C10B | 0.66380 (13) | 0.27980 (9) | 0.35296 (5) | 0.0362 (3) |
| C11B | 0.60426 (15) | 0.40810 (9) | 0.33826 (6) | 0.0396 (4) |
| C12B | 0.48842 (16) | 0.41864 (10) | 0.34708 (6) | 0.0481 (4) |
| H12B | 0.4496 | 0.3877 | 0.3701 | 0.058* |
| C13B | 0.42981 (16) | 0.47621 (11) | 0.32107 (6) | 0.0488 (4) |
| H13B | 0.3511 | 0.4835 | 0.3269 | 0.059* |
| C14B | 0.48616 (15) | 0.52276 (9) | 0.28679 (6) | 0.0428 (4) |
| C15B | 0.60316 (15) | 0.51005 (10) | 0.27840 (6) | 0.0428 (4) |
| H15B | 0.6422 | 0.5403 | 0.2551 | 0.051* |
| C16B | 0.66314 (15) | 0.45307 (9) | 0.30413 (6) | 0.0416 (4) |
| H16B | 0.7418 | 0.4454 | 0.2984 | 0.050* |
| C17B | 0.42202 (19) | 0.58559 (11) | 0.25908 (8) | 0.0597 (5) |
| H17A | 0.4712 | 0.6300 | 0.2558 | 0.089* |
| H17B | 0.3536 | 0.5994 | 0.2775 | 0.089* |
| H17C | 0.4005 | 0.5673 | 0.2264 | 0.089* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|-------------|-------------|-------------|--------------|--------------|-------------|
| O1A | 0.0630 (8) | 0.0459 (7) | 0.0443 (6) | -0.0036 (6) | 0.0113 (6) | -0.0089 (5) |
| O2A | 0.0815 (10) | 0.0666 (9) | 0.0489 (7) | 0.0005 (8) | 0.0093 (7) | -0.0219 (6) |
| N1A | 0.0399 (7) | 0.0362 (7) | 0.0408 (7) | 0.0000 (5) | -0.0069 (6) | -0.0042 (5) |
| C1A | 0.0340 (8) | 0.0357 (8) | 0.0491 (9) | 0.0000 (6) | -0.0108 (7) | 0.0032 (7) |
| C2A | 0.0400 (9) | 0.0456 (9) | 0.0493 (9) | -0.0030 (7) | -0.0047 (7) | -0.0009 (7) |
| C3A | 0.0397 (9) | 0.0626 (12) | 0.0552 (10) | 0.0000 (8) | -0.0009 (8) | 0.0061 (9) |
| C4A | 0.0396 (9) | 0.0603 (12) | 0.0735 (13) | 0.0004 (8) | -0.0007 (9) | 0.0205 (10) |
| C5A | 0.0391 (9) | 0.0409 (9) | 0.0908 (15) | -0.0038 (7) | -0.0120 (9) | 0.0193 (10) |
| C6A | 0.0384 (8) | 0.0339 (8) | 0.0670 (11) | 0.0023 (7) | -0.0151 (8) | 0.0030 (8) |
| C7A | 0.0485 (10) | 0.0333 (8) | 0.0793 (13) | -0.0010 (7) | -0.0175 (9) | -0.0060 (8) |
| C8A | 0.0572 (11) | 0.0427 (9) | 0.0580 (11) | 0.0080 (8) | -0.0130 (9) | -0.0156 (8) |
| C9A | 0.0438 (9) | 0.0388 (8) | 0.0439 (8) | 0.0053 (7) | -0.0119 (7) | -0.0067 (7) |
| C10A | 0.0544 (10) | 0.0463 (9) | 0.0357 (8) | 0.0079 (8) | -0.0076 (7) | -0.0088 (7) |
| C11A | 0.0535 (10) | 0.0437 (9) | 0.0368 (8) | 0.0051 (8) | 0.0066 (7) | -0.0038 (7) |
| C12A | 0.0650 (12) | 0.0597 (11) | 0.0418 (9) | 0.0046 (9) | -0.0070 (9) | 0.0130 (8) |
| C13A | 0.0509 (10) | 0.0619 (11) | 0.0475 (10) | 0.0043 (9) | -0.0101 (8) | 0.0077 (8) |
| C14A | 0.0526 (10) | 0.0464 (9) | 0.0366 (8) | 0.0057 (8) | 0.0002 (7) | -0.0019 (7) |
| C15A | 0.0584 (11) | 0.0684 (12) | 0.0390 (9) | 0.0069 (9) | -0.0039 (8) | 0.0129 (8) |
| C16A | 0.0452 (9) | 0.0704 (12) | 0.0447 (9) | 0.0062 (9) | -0.0045 (8) | 0.0059 (9) |
| C17A | 0.0640 (12) | 0.0682 (13) | 0.0477 (10) | -0.0078 (10) | 0.0010 (9) | 0.0003 (9) |
| O1B | 0.0612 (7) | 0.0323 (6) | 0.0463 (6) | 0.0021 (5) | -0.0194 (5) | -0.0004 (5) |
| O2B | 0.0662 (8) | 0.0464 (7) | 0.0494 (7) | 0.0073 (6) | -0.0224 (6) | -0.0120 (5) |
| N1B | 0.0362 (6) | 0.0270 (6) | 0.0333 (6) | -0.0016 (5) | 0.0012 (5) | -0.0006 (5) |
| C1B | 0.0337 (7) | 0.0259 (7) | 0.0330 (7) | -0.0026 (6) | 0.0037 (6) | 0.0012 (5) |
| C2B | 0.0435 (8) | 0.0283 (7) | 0.0378 (8) | -0.0004 (6) | -0.0009 (6) | -0.0010 (6) |
| C3B | 0.0460 (9) | 0.0366 (8) | 0.0363 (8) | -0.0016 (7) | -0.0036 (7) | 0.0017 (6) |
| C4B | 0.0433 (9) | 0.0379 (8) | 0.0417 (8) | 0.0057 (7) | -0.0005 (7) | 0.0083 (7) |
| C5B | 0.0466 (9) | 0.0270 (7) | 0.0441 (8) | 0.0049 (6) | 0.0069 (7) | 0.0036 (6) |
| C6B | 0.0371 (8) | 0.0267 (7) | 0.0351 (7) | -0.0019 (6) | 0.0083 (6) | 0.0007 (5) |
| C7B | 0.0488 (9) | 0.0265 (7) | 0.0400 (8) | 0.0000 (6) | 0.0061 (7) | -0.0051 (6) |
| C8B | 0.0467 (9) | 0.0343 (8) | 0.0343 (7) | -0.0045 (7) | 0.0007 (6) | -0.0068 (6) |
| C9B | 0.0346 (7) | 0.0318 (7) | 0.0332 (7) | -0.0036 (6) | 0.0020 (6) | -0.0009 (6) |
| C10B | 0.0385 (8) | 0.0367 (8) | 0.0335 (7) | -0.0013 (6) | 0.0014 (6) | -0.0026 (6) |
| C11B | 0.0514 (9) | 0.0332 (8) | 0.0341 (8) | 0.0026 (7) | -0.0108 (7) | -0.0023 (6) |
| C12B | 0.0519 (10) | 0.0484 (10) | 0.0440 (9) | -0.0026 (8) | 0.0000 (8) | 0.0086 (7) |
| C13B | 0.0458 (9) | 0.0545 (10) | 0.0462 (9) | 0.0072 (8) | -0.0011 (8) | 0.0022 (8) |
| C14B | 0.0528 (10) | 0.0387 (8) | 0.0369 (8) | 0.0061 (7) | -0.0073 (7) | -0.0029 (6) |
| C15B | 0.0533 (10) | 0.0414 (9) | 0.0337 (8) | -0.0005 (7) | -0.0022 (7) | 0.0030 (6) |
| C16B | 0.0444 (9) | 0.0426 (9) | 0.0378 (8) | 0.0034 (7) | -0.0022 (7) | -0.0044 (7) |
| C17B | 0.0685 (12) | 0.0532 (11) | 0.0573 (11) | 0.0158 (9) | -0.0089 (10) | 0.0076 (9) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------|-----------|----------|-------------|
| O1A—C10A | 1.349 (2) | O1B—C10B | 1.3486 (18) |
| O1A—C11A | 1.409 (2) | O1B—C11B | 1.4108 (18) |

| | | | |
|---------------|-------------|---------------|-------------|
| O2A—C10A | 1.198 (2) | O2B—C10B | 1.1930 (18) |
| N1A—C9A | 1.316 (2) | N1B—C9B | 1.3139 (18) |
| N1A—C1A | 1.362 (2) | N1B—C1B | 1.3626 (18) |
| C1A—C2A | 1.413 (2) | C1B—C2B | 1.412 (2) |
| C1A—C6A | 1.423 (2) | C1B—C6B | 1.4254 (19) |
| C2A—C3A | 1.366 (2) | C2B—C3B | 1.362 (2) |
| C2A—H2A | 0.9300 | C2B—H2B | 0.9300 |
| C3A—C4A | 1.404 (3) | C3B—C4B | 1.411 (2) |
| C3A—H3A | 0.9300 | C3B—H3B | 0.9300 |
| C4A—C5A | 1.357 (3) | C4B—C5B | 1.363 (2) |
| C4A—H4A | 0.9300 | C4B—H4B | 0.9300 |
| C5A—C6A | 1.419 (3) | C5B—C6B | 1.410 (2) |
| C5A—H5A | 0.9300 | C5B—H5B | 0.9300 |
| C6A—C7A | 1.403 (3) | C6B—C7B | 1.410 (2) |
| C7A—C8A | 1.360 (3) | C7B—C8B | 1.358 (2) |
| C7A—H7A | 0.9300 | C7B—H7B | 0.9300 |
| C8A—C9A | 1.418 (2) | C8B—C9B | 1.415 (2) |
| C8A—H8A | 0.9300 | C8B—H8B | 0.9300 |
| C9A—C10A | 1.494 (3) | C9B—C10B | 1.501 (2) |
| C11A—C12A | 1.370 (2) | C11B—C12B | 1.370 (2) |
| C11A—C16A | 1.372 (2) | C11B—C16B | 1.377 (2) |
| C12A—C13A | 1.378 (3) | C12B—C13B | 1.390 (2) |
| C12A—H12A | 0.9300 | C12B—H12B | 0.9300 |
| C13A—C14A | 1.379 (2) | C13B—C14B | 1.382 (2) |
| C13A—H13A | 0.9300 | C13B—H13B | 0.9300 |
| C14A—C15A | 1.386 (2) | C14B—C15B | 1.386 (2) |
| C14A—C17A | 1.508 (3) | C14B—C17B | 1.509 (2) |
| C15A—C16A | 1.378 (3) | C15B—C16B | 1.387 (2) |
| C15A—H15A | 0.9300 | C15B—H15B | 0.9300 |
| C16A—H16A | 0.9300 | C16B—H16B | 0.9300 |
| C17A—H17D | 0.9600 | C17B—H17A | 0.9600 |
| C17A—H17E | 0.9600 | C17B—H17B | 0.9600 |
| C17A—H17F | 0.9600 | C17B—H17C | 0.9600 |
| | | | |
| C10A—O1A—C11A | 118.21 (13) | C10B—O1B—C11B | 117.46 (12) |
| C9A—N1A—C1A | 117.78 (14) | C9B—N1B—C1B | 117.50 (12) |
| N1A—C1A—C2A | 118.48 (14) | N1B—C1B—C2B | 118.52 (12) |
| N1A—C1A—C6A | 122.53 (16) | N1B—C1B—C6B | 122.45 (13) |
| C2A—C1A—C6A | 118.96 (16) | C2B—C1B—C6B | 119.01 (13) |
| C3A—C2A—C1A | 120.60 (17) | C3B—C2B—C1B | 120.54 (14) |
| C3A—C2A—H2A | 119.7 | C3B—C2B—H2B | 119.7 |
| C1A—C2A—H2A | 119.7 | C1B—C2B—H2B | 119.7 |
| C2A—C3A—C4A | 120.66 (19) | C2B—C3B—C4B | 120.41 (15) |
| C2A—C3A—H3A | 119.7 | C2B—C3B—H3B | 119.8 |
| C4A—C3A—H3A | 119.7 | C4B—C3B—H3B | 119.8 |
| C5A—C4A—C3A | 120.14 (18) | C5B—C4B—C3B | 120.54 (15) |
| C5A—C4A—H4A | 119.9 | C5B—C4B—H4B | 119.7 |
| C3A—C4A—H4A | 119.9 | C3B—C4B—H4B | 119.7 |

| | | | |
|-----------------|--------------|-----------------|--------------|
| C4A—C5A—C6A | 121.24 (17) | C4B—C5B—C6B | 120.49 (14) |
| C4A—C5A—H5A | 119.4 | C4B—C5B—H5B | 119.8 |
| C6A—C5A—H5A | 119.4 | C6B—C5B—H5B | 119.8 |
| C7A—C6A—C5A | 124.10 (17) | C7B—C6B—C5B | 123.60 (14) |
| C7A—C6A—C1A | 117.51 (17) | C7B—C6B—C1B | 117.39 (14) |
| C5A—C6A—C1A | 118.38 (17) | C5B—C6B—C1B | 119.00 (14) |
| C8A—C7A—C6A | 119.59 (16) | C8B—C7B—C6B | 119.78 (14) |
| C8A—C7A—H7A | 120.2 | C8B—C7B—H7B | 120.1 |
| C6A—C7A—H7A | 120.2 | C6B—C7B—H7B | 120.1 |
| C7A—C8A—C9A | 119.04 (17) | C7B—C8B—C9B | 118.53 (14) |
| C7A—C8A—H8A | 120.5 | C7B—C8B—H8B | 120.7 |
| C9A—C8A—H8A | 120.5 | C9B—C8B—H8B | 120.7 |
| N1A—C9A—C8A | 123.46 (17) | N1B—C9B—C8B | 124.34 (14) |
| N1A—C9A—C10A | 117.90 (14) | N1B—C9B—C10B | 117.87 (13) |
| C8A—C9A—C10A | 118.56 (15) | C8B—C9B—C10B | 117.79 (13) |
| O2A—C10A—O1A | 123.60 (18) | O2B—C10B—O1B | 124.15 (14) |
| O2A—C10A—C9A | 124.28 (17) | O2B—C10B—C9B | 124.21 (14) |
| O1A—C10A—C9A | 112.06 (14) | O1B—C10B—C9B | 111.63 (12) |
| C12A—C11A—C16A | 120.93 (17) | C12B—C11B—C16B | 121.32 (15) |
| C12A—C11A—O1A | 118.69 (15) | C12B—C11B—O1B | 120.35 (15) |
| C16A—C11A—O1A | 120.19 (16) | C16B—C11B—O1B | 118.14 (15) |
| C11A—C12A—C13A | 119.03 (16) | C11B—C12B—C13B | 119.00 (16) |
| C11A—C12A—H12A | 120.5 | C11B—C12B—H12B | 120.5 |
| C13A—C12A—H12A | 120.5 | C13B—C12B—H12B | 120.5 |
| C12A—C13A—C14A | 122.00 (17) | C14B—C13B—C12B | 121.30 (17) |
| C12A—C13A—H13A | 119.0 | C14B—C13B—H13B | 119.4 |
| C14A—C13A—H13A | 119.0 | C12B—C13B—H13B | 119.4 |
| C13A—C14A—C15A | 117.20 (17) | C13B—C14B—C15B | 118.22 (15) |
| C13A—C14A—C17A | 121.00 (16) | C13B—C14B—C17B | 120.91 (17) |
| C15A—C14A—C17A | 121.79 (16) | C15B—C14B—C17B | 120.87 (16) |
| C16A—C15A—C14A | 121.88 (16) | C14B—C15B—C16B | 121.28 (16) |
| C16A—C15A—H15A | 119.1 | C14B—C15B—H15B | 119.4 |
| C14A—C15A—H15A | 119.1 | C16B—C15B—H15B | 119.4 |
| C11A—C16A—C15A | 118.95 (17) | C11B—C16B—C15B | 118.88 (16) |
| C11A—C16A—H16A | 120.5 | C11B—C16B—H16B | 120.6 |
| C15A—C16A—H16A | 120.5 | C15B—C16B—H16B | 120.6 |
| C14A—C17A—H17D | 109.5 | C14B—C17B—H17A | 109.5 |
| C14A—C17A—H17E | 109.5 | C14B—C17B—H17B | 109.5 |
| H17D—C17A—H17E | 109.5 | H17A—C17B—H17B | 109.5 |
| C14A—C17A—H17F | 109.5 | C14B—C17B—H17C | 109.5 |
| H17D—C17A—H17F | 109.5 | H17A—C17B—H17C | 109.5 |
| H17E—C17A—H17F | 109.5 | H17B—C17B—H17C | 109.5 |
| C9A—N1A—C1A—C2A | 178.47 (14) | C9B—N1B—C1B—C2B | 179.00 (13) |
| C9A—N1A—C1A—C6A | 0.2 (2) | C9B—N1B—C1B—C6B | 0.8 (2) |
| N1A—C1A—C2A—C3A | -176.71 (15) | N1B—C1B—C2B—C3B | -178.08 (13) |
| C6A—C1A—C2A—C3A | 1.6 (2) | C6B—C1B—C2B—C3B | 0.2 (2) |
| C1A—C2A—C3A—C4A | -0.6 (3) | C1B—C2B—C3B—C4B | -0.6 (2) |

| | | | |
|---------------------|--------------|---------------------|--------------|
| C2A—C3A—C4A—C5A | -0.2 (3) | C2B—C3B—C4B—C5B | 0.7 (2) |
| C3A—C4A—C5A—C6A | 0.0 (3) | C3B—C4B—C5B—C6B | -0.3 (2) |
| C4A—C5A—C6A—C7A | 179.93 (17) | C4B—C5B—C6B—C7B | 179.09 (15) |
| C4A—C5A—C6A—C1A | 1.1 (3) | C4B—C5B—C6B—C1B | -0.1 (2) |
| N1A—C1A—C6A—C7A | -2.5 (2) | N1B—C1B—C6B—C7B | -0.9 (2) |
| C2A—C1A—C6A—C7A | 179.24 (15) | C2B—C1B—C6B—C7B | -179.09 (13) |
| N1A—C1A—C6A—C5A | 176.45 (14) | N1B—C1B—C6B—C5B | 178.34 (13) |
| C2A—C1A—C6A—C5A | -1.8 (2) | C2B—C1B—C6B—C5B | 0.1 (2) |
| C5A—C6A—C7A—C8A | -176.68 (17) | C5B—C6B—C7B—C8B | -179.01 (14) |
| C1A—C6A—C7A—C8A | 2.2 (2) | C1B—C6B—C7B—C8B | 0.2 (2) |
| C6A—C7A—C8A—C9A | 0.2 (3) | C6B—C7B—C8B—C9B | 0.6 (2) |
| C1A—N1A—C9A—C8A | 2.4 (2) | C1B—N1B—C9B—C8B | 0.0 (2) |
| C1A—N1A—C9A—C10A | -174.34 (13) | C1B—N1B—C9B—C10B | -179.30 (12) |
| C7A—C8A—C9A—N1A | -2.7 (3) | C7B—C8B—C9B—N1B | -0.7 (2) |
| C7A—C8A—C9A—C10A | 174.08 (16) | C7B—C8B—C9B—C10B | 178.62 (14) |
| C11A—O1A—C10A—O2A | -2.1 (3) | C11B—O1B—C10B—O2B | -2.6 (2) |
| C11A—O1A—C10A—C9A | 175.13 (14) | C11B—O1B—C10B—C9B | 176.32 (13) |
| N1A—C9A—C10A—O2A | 176.18 (17) | N1B—C9B—C10B—O2B | -167.71 (15) |
| C8A—C9A—C10A—O2A | -0.7 (3) | C8B—C9B—C10B—O2B | 12.9 (2) |
| N1A—C9A—C10A—O1A | -1.0 (2) | N1B—C9B—C10B—O1B | 13.42 (19) |
| C8A—C9A—C10A—O1A | -177.91 (15) | C8B—C9B—C10B—O1B | -165.96 (13) |
| C10A—O1A—C11A—C12A | -101.26 (19) | C10B—O1B—C11B—C12B | 82.90 (19) |
| C10A—O1A—C11A—C16A | 83.7 (2) | C10B—O1B—C11B—C16B | -101.91 (17) |
| C16A—C11A—C12A—C13A | -0.8 (3) | C16B—C11B—C12B—C13B | -0.3 (3) |
| O1A—C11A—C12A—C13A | -175.76 (16) | O1B—C11B—C12B—C13B | 174.77 (15) |
| C11A—C12A—C13A—C14A | 0.0 (3) | C11B—C12B—C13B—C14B | -0.1 (3) |
| C12A—C13A—C14A—C15A | 0.7 (3) | C12B—C13B—C14B—C15B | 0.8 (3) |
| C12A—C13A—C14A—C17A | -178.47 (18) | C12B—C13B—C14B—C17B | -179.43 (17) |
| C13A—C14A—C15A—C16A | -0.6 (3) | C13B—C14B—C15B—C16B | -1.0 (2) |
| C17A—C14A—C15A—C16A | 178.60 (19) | C17B—C14B—C15B—C16B | 179.21 (16) |
| C12A—C11A—C16A—C15A | 0.9 (3) | C12B—C11B—C16B—C15B | 0.1 (2) |
| O1A—C11A—C16A—C15A | 175.81 (17) | O1B—C11B—C16B—C15B | -175.10 (13) |
| C14A—C15A—C16A—C11A | -0.2 (3) | C14B—C15B—C16B—C11B | 0.6 (2) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------|-------|-------------|-------------|---------------|
| $C15B-H15B\cdots O2A^i$ | 0.93 | 2.59 | 3.343 (2) | 138 |

Symmetry code: (i) $x+1/2, y, -z+1/2$.