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Methyl 5-bromo-6-methylpicolinate

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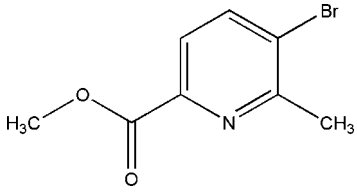
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.061; wR factor = 0.066; data-to-parameter ratio = 14.6.

The title compound, $C_8H_8BrNO_2$, does not show any significant intermolecular π–π or C—H$\cdots\pi$ interactions in the crystal packing except for one weak Br$\cdots$Br [3.715 (1) Å] interaction.

Related literature

The title compound is an important intermediate for the construction of novel supported PyOX ligands, see: Oila *et al.* (2005). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_8H_8BrNO_2$
M_r = 230.06
Monoclinic, $P2_1/c$
a = 18.518 (4) Å
b = 4.1040 (8) Å
c = 12.442 (3) Å
β = 109.52 (3)°
V = 891.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.57 mm^{-1}
T = 293 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.462, T_{max} = 0.658
1602 measured reflections
1602 independent reflections
975 reflections with $I > 2\sigma(I)$
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.066
S = 1.75
1602 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2083).

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supporting information

Acta Cryst. (2009). E**65**, o134 [doi:10.1107/S1600536808042104]

Methyl 5-bromo-6-methylpicolinate

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S1. Comment

The title compound is one of important intermediates for construction of novel supported PyOX-ligands (Oila *et al.*, 2005). Here we report the crystal structure of the title compound, methyl 5-bromo-6-methylpicolinate (Fig. 1).

In the title compound, the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The crystal structure is stabilized by a weak Br···Br^{i} interaction at 3.715 (1) Å (Fig. 2; symmetry code as in Fig. 2).

S2. Experimental

The title compound, (I) was prepared by a method reported in literature (Oila *et al.*, 2005) with some modification. The crystals were obtained by dissolving I (0.2 g) in methanol (50 ml) and evaporating the solvent slowly at room temperature for about 3 d.

S3. Refinement

H atoms were positioned geometrically, with O—H = 0.82 and C—H = 0.93 Å for aromatic H, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C/O), where x = 1.2 for aromatic H and x = 1.5 for other H.

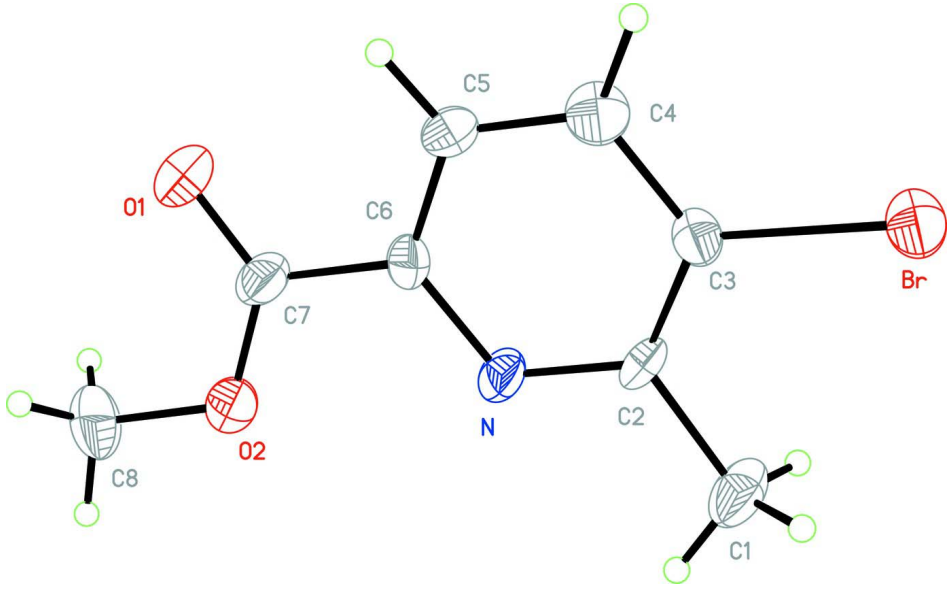

Figure 1

A drawing of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

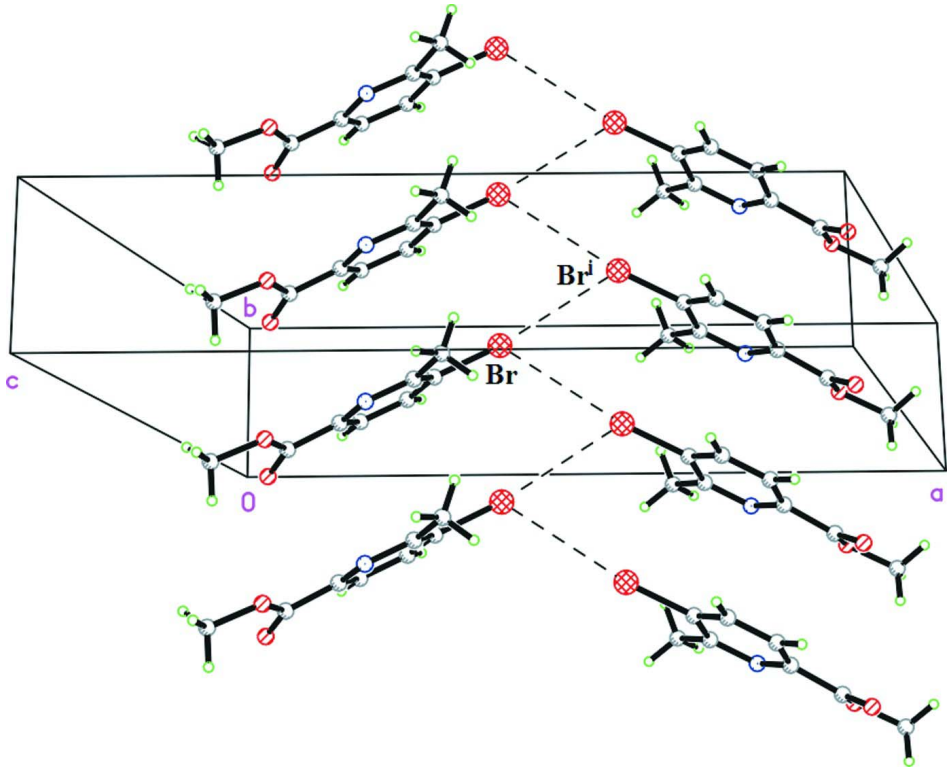

Figure 2

Br···Br interaction in the title compound. [Symmetry code: (i) -x+1, y+1/2, -z+1/2.]

Methyl 5-bromo-6-methylpicolinate

Crystal data

$C_8H_8BrNO_2$
$M_r = 230.06$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 18.518$ (4) Å
$b = 4.1040$ (8) Å
$c = 12.442$ (3) Å
$\beta = 109.52$ (3)°
$V = 891.2$ (4) Å^3
$Z = 4$
$F(000) = 456$
$D_x = 1.715$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–14°
$\mu = 4.57$ mm^{-1}
$T = 293$ K
Block, colorless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.462$, $T_{max} = 0.658$
1602 measured reflections
1602 independent reflections
975 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 25.3°$, $\theta_{min} = 1.2°$
$h = -22 \rightarrow 20$
$k = 0 \rightarrow 4$
$l = 0 \rightarrow 14$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.066$
$S = 1.75$
1602 reflections
110 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2)]$
$(\Delta/\sigma)_{max} < 0.000$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ e 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 $F^2 > 2$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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.41815 (4)	0.6507 (2)	0.25731 (6)	0.0546 (3)
O1	0.0882 (2)	−0.1451 (13)	0.2125 (3)	0.0691 (16)
O2	0.1349 (2)	−0.0435 (11)	0.3986 (4)	0.0533 (15)
N	0.2635 (3)	0.2009 (15)	0.3797 (4)	0.0450 (17)
C1	0.3893 (3)	0.4149 (17)	0.4811 (4)	0.067 (2)
H1A	0.3707	0.3787	0.5434	0.101*
H1B	0.4059	0.6370	0.4822	0.101*
H1C	0.4317	0.2717	0.4882	0.101*
C2	0.3260 (3)	0.3477 (18)	0.3698 (5)	0.0344 (16)
C3	0.3290 (3)	0.4444 (16)	0.2646 (5)	0.039 (2)
C4	0.2680 (3)	0.3789 (19)	0.1651 (5)	0.059 (2)
H4A	0.2702	0.4358	0.0939	0.071*
C5	0.2053 (3)	0.2298 (18)	0.1755 (5)	0.050 (2)
H5A	0.1632	0.1866	0.1109	0.060*
C6	0.2038 (3)	0.1425 (19)	0.2814 (5)	0.0426 (18)
C7	0.1372 (4)	−0.0335 (18)	0.2958 (6)	0.050 (2)
C8	0.0676 (3)	−0.1896 (19)	0.4127 (5)	0.070 (2)
H8A	0.0724	−0.1875	0.4920	0.104*
H8B	0.0627	−0.4104	0.3857	0.104*
H8C	0.0230	−0.0679	0.3698	0.104*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0482 (4)	0.0590 (5)	0.0589 (5)	−0.0020 (6)	0.0211 (3)	0.0028 (6)

O1	0.048 (3)	0.092 (4)	0.055 (4)	−0.011 (4)	0.001 (3)	−0.013 (4)
O2	0.043 (3)	0.070 (4)	0.046 (3)	−0.007 (3)	0.013 (2)	−0.002 (3)
N	0.029 (3)	0.065 (5)	0.033 (3)	−0.006 (4)	0.000 (3)	0.004 (4)
C1	0.056 (4)	0.087 (7)	0.043 (4)	−0.027 (5)	−0.004 (4)	0.012 (5)
C2	0.026 (4)	0.031 (4)	0.035 (4)	−0.003 (4)	−0.004 (3)	−0.004 (5)
C3	0.034 (4)	0.045 (6)	0.040 (4)	0.002 (4)	0.014 (4)	0.009 (4)
C4	0.049 (4)	0.088 (7)	0.033 (4)	0.004 (6)	0.005 (4)	0.031 (5)
C5	0.038 (4)	0.076 (7)	0.029 (4)	0.001 (4)	0.002 (3)	0.002 (4)
C6	0.025 (4)	0.060 (5)	0.042 (4)	0.004 (5)	0.011 (3)	−0.001 (5)
C7	0.037 (5)	0.060 (6)	0.043 (5)	0.004 (4)	0.000 (4)	−0.010 (5)
C8	0.051 (4)	0.079 (7)	0.089 (5)	−0.011 (5)	0.037 (4)	0.010 (6)

Geometric parameters (Å, º)

Br—Br^{i}	3.715 (1)	C2—C3	1.387 (6)
Br—C3	1.884 (5)	C3—C4	1.396 (6)
O1—C7	1.218 (6)	C4—C5	1.356 (7)
O2—C7	1.294 (6)	C4—H4A	0.9300
O2—C8	1.446 (6)	C5—C6	1.375 (7)
N—C2	1.347 (6)	C5—H5A	0.9300
N—C6	1.368 (6)	C6—C7	1.491 (8)
C1—C2	1.510 (6)	C8—H8A	0.9600
C1—H1A	0.9600	C8—H8B	0.9600
C1—H1B	0.9600	C8—H8C	0.9600
C1—H1C	0.9600		
C7—O2—C8	116.6 (5)	C3—C4—H4A	121.0
C2—N—C6	117.3 (5)	C4—C5—C6	120.0 (6)
C2—C1—H1A	109.5	C4—C5—H5A	120.0
C2—C1—H1B	109.5	C6—C5—H5A	120.0
H1A—C1—H1B	109.5	N—C6—C5	122.9 (6)
C2—C1—H1C	109.5	N—C6—C7	115.5 (6)
H1A—C1—H1C	109.5	C5—C6—C7	121.6 (6)
H1B—C1—H1C	109.5	O1—C7—O2	124.6 (7)
N—C2—C3	121.4 (5)	O1—C7—C6	119.4 (7)
N—C2—C1	115.2 (5)	O2—C7—C6	115.9 (6)
C3—C2—C1	123.3 (6)	O2—C8—H8A	109.5
C4—C3—C2	120.4 (5)	O2—C8—H8B	109.5
C4—C3—Br	120.5 (5)	H8A—C8—H8B	109.5
C2—C3—Br	119.1 (5)	O2—C8—H8C	109.5
C5—C4—C3	117.9 (6)	H8A—C8—H8C	109.5
C5—C4—H4A	121.0	H8B—C8—H8C	109.5
C6—N—C2—C3	1.8 (10)	C2—N—C6—C7	177.4 (6)
C6—N—C2—C1	178.4 (6)	C4—C5—C6—N	0.4 (11)
N—C2—C3—C4	−2.7 (11)	C4—C5—C6—C7	−177.5 (7)
C1—C2—C3—C4	−179.0 (6)	C8—O2—C7—O1	−2.1 (11)
N—C2—C3—Br	179.6 (5)	C8—O2—C7—C6	175.0 (6)

C1—C2—C3—Br	3.3 (9)	N—C6—C7—O1	−167.1 (7)
C2—C3—C4—C5	2.3 (11)	C5—C6—C7—O1	11.0 (11)
Br—C3—C4—C5	180.0 (5)	N—C6—C7—O2	15.6 (9)
C3—C4—C5—C6	−1.2 (11)	C5—C6—C7—O2	−166.3 (7)
C2—N—C6—C5	−0.6 (10)		

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.