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4-Chloro-1-(4-methylphenylsulfonyl)-1*H*-pyrrolo[2,3-*b*]pyridine

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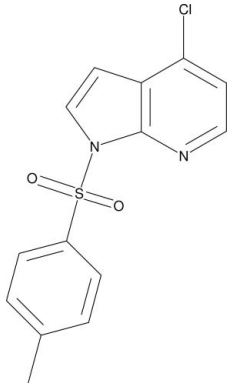
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.119; data-to-parameter ratio = 14.3.

The crystal structure of the title compound, $C_{14}H_{11}ClN_2O_2S$, features a three-dimensional network stabilized by π–π interactions between the rings of the 4-methylphenylsulfonyl protecting group [centroid–centroid distance = 3.623 (1) Å]. The 4-methylphenylsulfonyl ring makes a dihedral angle of 79.60 (6)° with the 4-chloro-1*H*-pyrrolo[2,3-*b*]pyridine unit.

Related literature

For the synthesis of the title compound, see: Desarbre *et al.* (1997).



Experimental

Crystal data

$C_{14}H_{11}ClN_2O_2S$
M_r = 306.76
Monoclinic, $C2/c$
a = 21.7342 (12) Å
b = 7.6313 (2) Å
c = 16.4649 (8) Å
β = 91.531 (2)°
V = 2729.9 (2) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 3.94 mm^{-1}
T = 193 K
0.52 × 0.24 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: numerical (*PLATON*; Spek, 2009)
T_{min} = 0.319, T_{max} = 0.519
2580 measured reflections
2580 independent reflections
2435 reflections with $I > 2\sigma(I)$
3 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.119
S = 1.09
2580 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CORINC* (Dräger & Gattow, 1971); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5113).

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Acta Cryst. (2009). E**65**, o3018 [doi:10.1107/S1600536809044559]

4-Chloro-1-(4-methylphenylsulfonyl)-1*H*-pyrrolo[2,3-*b*]pyridine

Roland Selig, Dieter Schollmeyer, Wolfgang Albrecht and Stefan Laufer

S1. Comment

In recent years, compounds with the 1*H*-pyrrolo[2,3-*b*]pyridine moiety have been shown to display significant biological activities. The N-protected 4-chloro-1*H*-pyrrolo[2,3-*b*]pyridine is an important precursor for NH sensitive reactions like coupling reactions or metalation experiments. The title compound forms a three dimensional network stabilized by π -π interactions between two phenyl moieties of the 4-methylphenylsulfonyl protecting group (distance between centroids 3.623 (1) Å). The 4-methylphenylsulfonyl ring makes a dihedral angle of 79.60 (6)° to the 4-chloro-1*H*-pyrrolo[2,3-*b*]pyridine.

S2. Experimental

Finely powdered sodium hydroxide (1.9 g, 34 mmol) was added to a solution of dichloromethane containing benzyltriethylammonium chloride (67 mg, 0.30 mmol) and 4-chlor-1*H*-pyrrolo[2,3-*b*]pyridine (1.5 g, 9.8 mmol). *p*-Toluensulfonylchloride (2.2 g, 12 mmol) was slowly added at 273 K and the resulting suspension was stirred at this temperature for 2 h at room temperature. The suspension was filtered through celite, washed with dichloromethane and the filtrate was evaporated *in vacuo*. The residue was suspendet in methanol and filtered off. The filtrate was dried *in vacuo* to give the pure title compound in a good yield of 78%.

S3. Refinement

Hydrogen atoms were placed at calculated positions with $C_{aromatic}$—H = 0.95 Å or C_{methyl}—H = 0.98Å and they were refined in the riding-model approximation with isotropic displacement parameters (set at 1.2–1.5 times of the U_{eq} of the parent atom).

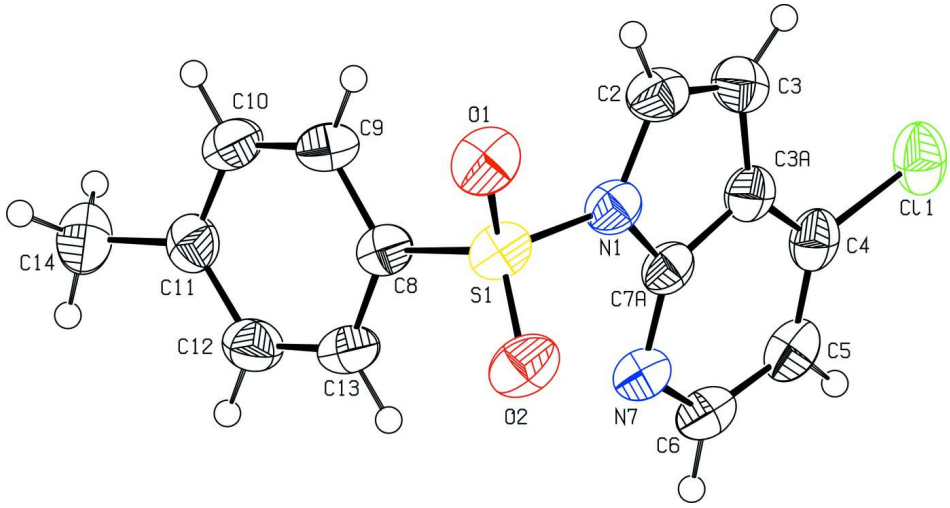

Figure 1

View of compound **I**. Displacement ellipsoids are drawn at the 50% probability level.

4-Chloro-1-(4-methylphenylsulfonyl)-1*H*-pyrrolo[2,3-*b*]pyridine

Crystal data

$C_{14}H_{11}ClN_2O_2S$
$M_r = 306.76$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 21.7342\ (12)$ Å
$b = 7.6313\ (2)$ Å
$c = 16.4649\ (8)$ Å
$\beta = 91.531\ (2)°$
$V = 2729.9\ (2)$ Å^3
$Z = 8$
$F(000) = 1264$
$D_x = 1.493$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 65–70°$
$\mu = 3.94$ mm^{-1}
$T = 193$ K
Block, colourless
0.52 × 0.24 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: rotating anode
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: numerical (*PLATON*; Spek, 2009)
$T_{min} = 0.319$, $T_{max} = 0.519$
2580 measured reflections
2580 independent reflections
2435 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 69.9°$, $\theta_{min} = 4.1°$
$h = 0\rightarrow26$
$k = 0\rightarrow9$
$l = -20\rightarrow20$
3 standard reflections every 60 min
intensity decay: 2%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.119$
$S = 1.09$
2580 reflections
181 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

$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2 + 2.2448P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
S1	0.37591 (3)	0.21601 (7)	0.24460 (3)	0.04415 (18)
Cl1	0.17076 (3)	0.02839 (8)	0.52042 (4)	0.0552 (2)
O1	0.35059 (9)	0.2966 (2)	0.17315 (9)	0.0568 (4)
O2	0.40887 (9)	0.0548 (2)	0.23960 (10)	0.0574 (4)
N1	0.31423 (8)	0.1778 (2)	0.30138 (10)	0.0419 (4)
C2	0.25596 (11)	0.2537 (3)	0.28739 (14)	0.0471 (5)
H2	0.2448	0.3249	0.2420	0.057*
C3	0.21846 (10)	0.2106 (3)	0.34773 (14)	0.0452 (5)
H3	0.1766	0.2440	0.3520	0.054*
C3A	0.25318 (9)	0.1048 (3)	0.40466 (12)	0.0393 (4)
C4	0.24293 (10)	0.0231 (3)	0.47865 (13)	0.0428 (5)
C5	0.29115 (11)	−0.0600 (3)	0.51803 (13)	0.0463 (5)
H5	0.2853	−0.1165	0.5686	0.056*
C6	0.34872 (11)	−0.0608 (3)	0.48308 (13)	0.0458 (5)
H6	0.3814	−0.1178	0.5120	0.055*
N7	0.36143 (8)	0.0130 (2)	0.41150 (11)	0.0427 (4)
C7A	0.31332 (9)	0.0897 (2)	0.37609 (12)	0.0374 (4)
C8	0.41925 (9)	0.3694 (3)	0.30021 (11)	0.0381 (4)
C9	0.40403 (10)	0.5448 (3)	0.29162 (14)	0.0466 (5)
H9	0.3696	0.5785	0.2585	0.056*
C10	0.43905 (10)	0.6698 (3)	0.33118 (14)	0.0478 (5)
H10	0.4284	0.7899	0.3252	0.057*
C11	0.48956 (9)	0.6237 (3)	0.37969 (12)	0.0434 (5)
C12	0.50321 (10)	0.4468 (3)	0.38851 (14)	0.0484 (5)
H12	0.5373	0.4129	0.4223	0.058*
C13	0.46853 (10)	0.3189 (3)	0.34933 (13)	0.0451 (5)
H13	0.4785	0.1985	0.3561	0.054*
C14	0.52904 (13)	0.7648 (4)	0.41851 (17)	0.0615 (6)
H14A	0.5628	0.7105	0.4502	0.092*
H14B	0.5040	0.8362	0.4544	0.092*
H14C	0.5461	0.8392	0.3761	0.092*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0603 (3)	0.0409 (3)	0.0313 (3)	0.0037 (2)	0.0033 (2)	−0.00179 (18)
Cl1	0.0548 (3)	0.0523 (3)	0.0593 (4)	−0.0143 (2)	0.0148 (3)	−0.0159 (2)
O1	0.0779 (11)	0.0603 (11)	0.0319 (7)	−0.0021 (8)	−0.0050 (7)	0.0023 (7)
O2	0.0793 (12)	0.0432 (9)	0.0503 (9)	0.0096 (8)	0.0149 (8)	−0.0083 (7)
N1	0.0500 (10)	0.0398 (9)	0.0359 (8)	0.0004 (7)	−0.0024 (7)	0.0025 (7)
C2	0.0551 (12)	0.0437 (11)	0.0417 (11)	0.0038 (10)	−0.0146 (9)	−0.0013 (9)
C3	0.0437 (11)	0.0434 (12)	0.0479 (11)	−0.0010 (9)	−0.0102 (9)	−0.0115 (9)
C3A	0.0450 (10)	0.0315 (9)	0.0411 (10)	−0.0049 (8)	−0.0031 (8)	−0.0102 (8)
C4	0.0503 (11)	0.0348 (10)	0.0433 (11)	−0.0095 (8)	0.0045 (9)	−0.0113 (8)
C5	0.0645 (13)	0.0360 (10)	0.0383 (10)	−0.0070 (10)	0.0012 (9)	−0.0001 (8)
C6	0.0584 (12)	0.0348 (10)	0.0440 (11)	0.0037 (9)	−0.0036 (9)	0.0026 (9)
N7	0.0509 (10)	0.0355 (9)	0.0416 (9)	0.0038 (7)	0.0007 (7)	0.0006 (7)
C7A	0.0473 (10)	0.0285 (9)	0.0362 (9)	−0.0016 (8)	−0.0013 (8)	−0.0044 (7)
C8	0.0450 (10)	0.0387 (10)	0.0310 (9)	0.0052 (8)	0.0077 (7)	0.0022 (7)
C9	0.0504 (12)	0.0422 (11)	0.0469 (11)	0.0105 (9)	−0.0042 (9)	0.0061 (9)
C10	0.0551 (12)	0.0367 (11)	0.0519 (12)	0.0071 (9)	0.0051 (10)	0.0053 (9)
C11	0.0431 (10)	0.0476 (12)	0.0401 (10)	−0.0026 (9)	0.0107 (8)	0.0034 (9)
C12	0.0431 (11)	0.0534 (13)	0.0483 (12)	0.0063 (10)	−0.0025 (9)	0.0077 (10)
C13	0.0505 (11)	0.0402 (11)	0.0447 (11)	0.0120 (9)	0.0038 (9)	0.0061 (9)
C14	0.0609 (14)	0.0572 (15)	0.0665 (16)	−0.0147 (12)	0.0030 (12)	0.0037 (12)

Geometric parameters (Å, º)

S1—O1	1.4250 (16)	C6—H6	0.9500
S1—O2	1.4269 (17)	N7—C7A	1.320 (3)
S1—N1	1.6803 (18)	C8—C13	1.380 (3)
S1—C8	1.746 (2)	C8—C9	1.385 (3)
Cl1—C4	1.730 (2)	C9—C10	1.374 (3)
N1—C7A	1.402 (3)	C9—H9	0.9500
N1—C2	1.406 (3)	C10—C11	1.386 (3)
C2—C3	1.343 (3)	C10—H10	0.9500
C2—H2	0.9500	C11—C12	1.389 (3)
C3—C3A	1.436 (3)	C11—C14	1.508 (3)
C3—H3	0.9500	C12—C13	1.382 (3)
C3A—C4	1.392 (3)	C12—H12	0.9500
C3A—C7A	1.406 (3)	C13—H13	0.9500
C4—C5	1.373 (3)	C14—H14A	0.9800
C5—C6	1.391 (3)	C14—H14B	0.9800
C5—H5	0.9500	C14—H14C	0.9800
C6—N7	1.342 (3)		
O1—S1—O2	120.52 (10)	N7—C7A—N1	124.77 (19)
O1—S1—N1	103.77 (10)	N7—C7A—C3A	128.40 (19)
O2—S1—N1	106.95 (10)	N1—C7A—C3A	106.81 (17)
O1—S1—C8	109.55 (10)	C13—C8—C9	120.6 (2)

O2—S1—C8	110.09 (10)	C13—C8—S1	121.32 (17)
N1—S1—C8	104.58 (9)	C9—C8—S1	118.05 (16)
C7A—N1—C2	107.91 (18)	C10—C9—C8	119.7 (2)
C7A—N1—S1	126.97 (15)	C10—C9—H9	120.2
C2—N1—S1	124.46 (16)	C8—C9—H9	120.2
C3—C2—N1	109.85 (19)	C9—C10—C11	121.2 (2)
C3—C2—H2	125.1	C9—C10—H10	119.4
N1—C2—H2	125.1	C11—C10—H10	119.4
C2—C3—C3A	107.59 (19)	C10—C11—C12	118.1 (2)
C2—C3—H3	126.2	C10—C11—C14	119.7 (2)
C3A—C3—H3	126.2	C12—C11—C14	122.1 (2)
C4—C3A—C7A	115.35 (19)	C13—C12—C11	121.7 (2)
C4—C3A—C3	136.9 (2)	C13—C12—H12	119.2
C7A—C3A—C3	107.75 (19)	C11—C12—H12	119.2
C5—C4—C3A	118.8 (2)	C8—C13—C12	118.8 (2)
C5—C4—Cl1	120.78 (17)	C8—C13—H13	120.6
C3A—C4—Cl1	120.37 (17)	C12—C13—H13	120.6
C4—C5—C6	119.4 (2)	C11—C14—H14A	109.5
C4—C5—H5	120.3	C11—C14—H14B	109.5
C6—C5—H5	120.3	H14A—C14—H14B	109.5
N7—C6—C5	124.8 (2)	C11—C14—H14C	109.5
N7—C6—H6	117.6	H14A—C14—H14C	109.5
C5—C6—H6	117.6	H14B—C14—H14C	109.5
C7A—N7—C6	113.26 (19)		

O1—S1—N1—C7A	175.64 (17)	C2—N1—C7A—C3A	3.3 (2)
O2—S1—N1—C7A	47.22 (19)	S1—N1—C7A—C3A	174.17 (14)
C8—S1—N1—C7A	−69.55 (19)	C4—C3A—C7A—N7	−2.3 (3)
O1—S1—N1—C2	−14.9 (2)	C3—C3A—C7A—N7	175.68 (19)
O2—S1—N1—C2	−143.28 (18)	C4—C3A—C7A—N1	179.38 (16)
C8—S1—N1—C2	99.95 (18)	C3—C3A—C7A—N1	−2.6 (2)
C7A—N1—C2—C3	−2.7 (2)	O1—S1—C8—C13	−151.45 (17)
S1—N1—C2—C3	−173.92 (15)	O2—S1—C8—C13	−16.72 (19)
N1—C2—C3—C3A	1.0 (2)	N1—S1—C8—C13	97.86 (17)
C2—C3—C3A—C4	178.3 (2)	O1—S1—C8—C9	26.4 (2)
C2—C3—C3A—C7A	1.0 (2)	O2—S1—C8—C9	161.17 (17)
C7A—C3A—C4—C5	1.5 (3)	N1—S1—C8—C9	−84.25 (18)
C3—C3A—C4—C5	−175.7 (2)	C13—C8—C9—C10	1.2 (3)
C7A—C3A—C4—Cl1	−179.07 (14)	S1—C8—C9—C10	−176.68 (17)
C3—C3A—C4—Cl1	3.8 (3)	C8—C9—C10—C11	0.0 (3)
C3A—C4—C5—C6	−0.1 (3)	C9—C10—C11—C12	−1.2 (3)
Cl1—C4—C5—C6	−179.55 (16)	C9—C10—C11—C14	176.6 (2)
C4—C5—C6—N7	−0.9 (3)	C10—C11—C12—C13	1.1 (3)
C5—C6—N7—C7A	0.3 (3)	C14—C11—C12—C13	−176.7 (2)
C6—N7—C7A—N1	179.39 (18)	C9—C8—C13—C12	−1.3 (3)
C6—N7—C7A—C3A	1.4 (3)	S1—C8—C13—C12	176.54 (17)
C2—N1—C7A—N7	−175.14 (19)	C11—C12—C13—C8	0.1 (3)
S1—N1—C7A—N7	−4.2 (3)		