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2-Chloro-3-(2,2,2-trifluoroethoxy)-pyridine

Pin-Liang Wang,* Hai-Su Zeng, Si-Shun Kang and Hai-Bo Wang

College of Science, Nanjing University of Technology, Xinmofan Road No. 5, Nanjing 210009, People's Republic of China

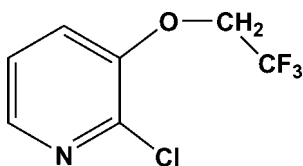
Correspondence e-mail: wanghaibo@njut.edu.cn

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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.079; wR factor = 0.237; data-to-parameter ratio = 14.8.

The title compound, $\text{C}_7\text{H}_5\text{ClF}_3\text{NO}$, was synthesized by the reaction of 2-chloro-3-hydroxypyridine with 2-chloro-1,1,1-trifluoroethane. The pyridine ring and the O and Cl atoms are nearly coplanar, the largest deviation from the mean plane being 0.078 (6) Å. Bioassay indicates that the title compound has a good fungicidal activity.

Related literature

For related literature, see: Sandford *et al.* (2004).

Experimental

Crystal data

$\text{C}_7\text{H}_5\text{ClF}_3\text{NO}$
 $M_r = 211.57$
 Orthorhombic, $Pbca$
 $a = 12.907$ (3) Å
 $b = 7.469$ (2) Å
 $c = 18.520$ (4) Å

$V = 1785.4$ (7) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.43$ mm⁻¹
 $T = 293$ (2) K
 $0.40 \times 0.30 \times 0.30$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.846$, $T_{\max} = 0.881$
 1749 measured reflections

1749 independent reflections
 814 reflections with $I > 2\sigma(I)$
 3 standard reflections every 200 reflections
 intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.080$
 $wR(F^2) = 0.237$
 $S = 1.04$
 1749 reflections

118 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.26$ e Å⁻³
 $\Delta\rho_{\min} = -0.31$ e Å⁻³

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2242).

References

- Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, U. S.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sandford, G., Wilson, I. & Timperley, C. M. (2004). *J. Fluorine Chem.* **125**, 1425–1430.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

supplementary materials

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2-Chloro-3-(2,2,2-trifluoroethoxy)pyridine

P.-L. Wang, H.-S. Zeng, S.-S. Kang and H.-B. Wang

Comment

The organic fluorine derivatives have been investigated as pesticide (Sandford *et al.*, 2004). This article focus on new pesticide containing trifluoroethoxy group.

The title compound, C₇H₅ClF₃NO, which is a derivative of 2-chloropyridine, was synthesized by the reaction of 2-Chloro-3-hydroxypyridine with 2-chloro-1,1,1-trifluoroethane, and characterized by ¹H NMR, IR, elemental analysis and single-crystal X-ray diffraction. The structure analysis reveals that the benzene ring, oxygen and chlorine atoms are nerally coplanar with the largest deviation from the mean plane being 0.078 (6)Å at C1(Fig. 1). Bioassay indicates that the title compound has a good fungicidal activity.

Experimental

2-Chloro-3-hydroxypyridine (30 mmol) was dissolved in DMF (40 ml) and potassium carbonate (40 mmol) was added in one portion. A solution of 2-chloro-1,1,1-trifluoroethane (30 mmol) in DMF (20 ml) was then added to this mixture at 293 K. The resulting mixture was refluxed for 32 h. The mixture was filtered and the filtrate concentrated under reduced pressure to afford crude compound (I). Pure (I) was obtained by recrystallization from ethyl acetate. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. Spectroscopic analysis, ¹H NMR (CDCl₃, δ, p.p.m.): 8.54–8.55 (m, 1H), 7.82–7.84 (m, 1H), 7.65–7.67 (m, 1H), 4.48 (m, 2H).

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.97 Å (methylene) and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

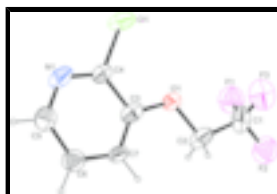


Fig. 1. Molecular structure of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

2-Chloro-3-(2,2,2-trifluoroethoxy)pyridine

Crystal data

$C_7H_5ClF_3NO$

$M_r = 211.57$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 12.907$ (3) Å

$b = 7.469$ (2) Å

$c = 18.520$ (4) Å

$V = 1785.4$ (7) Å³

$Z = 8$

$F_{000} = 848$

$D_x = 1.574$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9\text{--}12^\circ$

$\mu = 0.43$ mm⁻¹

$T = 293$ (2) K

Block, colourless

$0.40 \times 0.30 \times 0.30$ mm

Data collection

Enraf-Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.846$, $T_{\max} = 0.881$

1749 measured reflections

1749 independent reflections

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

$R_{\text{int}} = 0.0000$

$\theta_{\max} = 26.0^\circ$

$\theta_{\min} = 2.2^\circ$

$h = 0 \rightarrow 15$

$k = 0 \rightarrow 9$

$l = 0 \rightarrow 22$

3 standard reflections

every 200 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.080$

$wR(F^2) = 0.237$

$S = 1.04$

1749 reflections

118 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0827P)^2 + 3.9201P]$$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.26$ e Å⁻³

$\Delta\rho_{\min} = -0.30$ e Å⁻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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3876 (8)	0.2387 (13)	0.4626 (4)	0.089 (2)
C2	0.3770 (5)	0.2123 (10)	0.3840 (4)	0.078 (2)
H2A	0.4131	0.3065	0.3581	0.094*
H2B	0.4065	0.0980	0.3700	0.094*
C3	0.2438 (5)	0.1764 (7)	0.2976 (3)	0.0588 (15)
C4	0.1336 (4)	0.1758 (8)	0.2832 (3)	0.0561 (15)
C5	0.1602 (5)	0.1046 (11)	0.1698 (4)	0.081 (2)
H5	0.1328	0.0785	0.1245	0.097*
C6	0.2693 (5)	0.1007 (9)	0.1781 (3)	0.0692 (18)
H6	0.3129	0.0767	0.1393	0.083*
C7	0.3077 (4)	0.1340 (8)	0.2457 (3)	0.0562 (15)
H7	0.3785	0.1265	0.2546	0.067*
C11	0.04948 (12)	0.2192 (3)	0.35288 (11)	0.0830 (7)
F1	0.3504 (4)	0.3979 (8)	0.4816 (3)	0.1198 (17)
F2	0.4852 (4)	0.2529 (7)	0.4787 (2)	0.1119 (16)
F3	0.3469 (4)	0.1264 (8)	0.4989 (3)	0.1251 (19)
N1	0.0970 (4)	0.1421 (7)	0.2211 (3)	0.0710 (16)
O1	0.2678 (3)	0.2168 (6)	0.36733 (19)	0.0668 (12)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.101 (7)	0.091 (7)	0.074 (5)	0.009 (6)	-0.031 (5)	0.004 (5)
C2	0.049 (3)	0.083 (5)	0.102 (5)	0.015 (4)	-0.011 (3)	-0.015 (4)
C3	0.045 (3)	0.042 (3)	0.089 (4)	-0.003 (3)	-0.005 (4)	0.006 (3)
C4	0.041 (3)	0.060 (4)	0.067 (4)	0.004 (3)	-0.003 (3)	0.002 (3)
C5	0.067 (5)	0.109 (6)	0.067 (4)	-0.010 (4)	-0.012 (4)	-0.008 (4)
C6	0.063 (4)	0.075 (5)	0.070 (4)	-0.002 (4)	0.003 (3)	-0.012 (3)
C7	0.031 (3)	0.068 (4)	0.070 (4)	0.002 (3)	0.005 (3)	0.019 (3)
C11	0.0444 (8)	0.0978 (14)	0.1067 (14)	0.0030 (9)	0.0116 (9)	-0.0115 (11)
F1	0.128 (4)	0.118 (4)	0.114 (4)	0.006 (3)	-0.005 (3)	-0.004 (3)
F2	0.108 (4)	0.121 (4)	0.107 (4)	0.003 (3)	-0.016 (3)	-0.011 (3)

supplementary materials

F3	0.145 (5)	0.122 (4)	0.108 (4)	-0.003 (4)	-0.013 (3)	0.005 (3)
N1	0.043 (3)	0.079 (4)	0.091 (4)	-0.001 (3)	-0.015 (3)	-0.020 (3)
O1	0.040 (2)	0.105 (4)	0.056 (2)	-0.001 (2)	-0.0037 (18)	-0.002 (2)

Geometric parameters (Å, °)

C1—F3	1.197 (9)	C3—C4	1.447 (8)
C1—F2	1.299 (10)	C4—N1	1.269 (7)
C1—F1	1.330 (9)	C4—C11	1.717 (6)
C1—C2	1.474 (10)	C5—N1	1.284 (8)
C2—O1	1.443 (7)	C5—C6	1.417 (9)
C2—H2A	0.9700	C5—H5	0.9300
C2—H2B	0.9700	C6—C7	1.370 (8)
C3—C7	1.305 (8)	C6—H6	0.9300
C3—O1	1.363 (7)	C7—H7	0.9300
F3—C1—F2	110.7 (7)	N1—C4—C3	122.2 (5)
F3—C1—F1	108.6 (8)	N1—C4—C11	118.9 (5)
F2—C1—F1	102.5 (8)	C3—C4—C11	118.9 (5)
F3—C1—C2	114.9 (8)	N1—C5—C6	123.8 (6)
F2—C1—C2	109.1 (8)	N1—C5—H5	118.1
F1—C1—C2	110.4 (7)	C6—C5—H5	118.1
O1—C2—C1	107.4 (6)	C7—C6—C5	117.1 (6)
O1—C2—H2A	110.2	C7—C6—H6	121.5
C1—C2—H2A	110.2	C5—C6—H6	121.5
O1—C2—H2B	110.2	C3—C7—C6	119.2 (6)
C1—C2—H2B	110.2	C3—C7—H7	120.4
H2A—C2—H2B	108.5	C6—C7—H7	120.4
C7—C3—O1	127.4 (5)	C4—N1—C5	118.6 (5)
C7—C3—C4	119.0 (6)	C3—O1—C2	114.9 (5)
O1—C3—C4	113.5 (5)		

Fig. 1

