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4-{[(5-Methyl-2-furyl)methylene]-hydrazinocarbonyl}pyridinium chloride monohydrate

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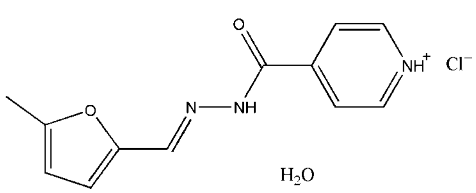
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.107; data-to-parameter ratio = 17.7.

The title compound, $C_{12}H_{12}N_3O_2^+\cdot Cl^-\cdot H_2O$, was prepared by the reaction of N'-[(5-methyl-2-furyl)methylene]isonicotinohydrazide and hydrochloric acid at room temperature. The entire molecule is approximately planar with a maximum deviation of 0.047 (2) Å. An intramolecular C—H···O interaction is observed. O—H···Cl, N—H···Cl, N—H···O, N—H···N, C—H···Cl and C—H···O hydrogen-bonds stabilize the crystal structure.

Related literature

Schiff bases have been used extensively as ligands in the field of coordination chemistry, see: Cui *et al.* (2005). For their antimicrobial and anticancer applications, see: Tarafder *et al.* (2000) and Deschamps *et al.* (2003), respectively.



Experimental

Crystal data

$C_{12}H_{12}N_3O_2^+\cdot Cl^-\cdot H_2O$
M_r = 283.71
Monoclinic, $P2_1/c$
a = 8.5258 (17) Å
b = 14.435 (3) Å
c = 13.625 (4) Å
β = 123.55 (2)°
V = 1397.5 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 293 K
0.20 × 0.15 × 0.11 mm

Data collection

Bruker P4 diffractometer
Absorption correction: none
13328 measured reflections
3187 independent reflections
2715 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.107
S = 1.07
3187 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H2W1···Cl1^{i}	0.86 (3)	2.40 (3)	3.229 (3)	162 (3)
O1W—H1W1···Cl1^{ii}	0.72 (3)	2.51 (3)	3.225 (3)	177 (2)
N2—H2A···Cl1^{i}	0.86	2.39	3.2243 (15)	164
N3—H3A···O2^{ii}	0.86	1.89	2.639 (2)	144
N3—H3A···N1^{ii}	0.86	2.50	3.2238 (18)	142
C3—H3B···Cl1^{iii}	0.93	2.76	3.6574 (19)	162
C6—H6A···Cl1^{i}	0.93	2.69	3.5374 (18)	151
C9—H9A···Cl1^{i}	0.93	2.64	3.5656 (18)	171
C11—H11A···O1^{ii}	0.93	2.45	3.1694 (19)	135
C12—H12A···O2	0.93	2.39	2.713 (2)	100

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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: AT2845).

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Deschamps, P., Kulkarni, P. P. & Sarkar, B. (2003). *Inorg. Chem.* **42**, 7366–7368.
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Tarafder, M. T. H., Ali, M. A., Wee, D. J., Azahari, K., Silong, S. & Crouse, K. A. (2000). *Transition Met. Chem.* **25**, 456–460.

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S1. Comment

Schiff bases have been used extensively as ligands in the field of coordination chemistry (Cui *et al.*, 2005). And they have antimicrobial (Tarafder *et al.*, 2000) and anticancer applications (Deschamps *et al.*, 2003). The title compound (I) was synthesized and we report its crystal structure here.

In the crystal structure of (I) (Fig. 1), the carbon and nitrogen atoms are nearly the same plane with a maximum deviation of 0.047Å for N2. There are intra- and intermolecular O—H···Cl, N—H···Cl, N—H···O, N—H···N, C—H···Cl and C—H···O hydrogen-bonds to stabilize the crystal structure (Table 1).

S2. Experimental

A mixture of *N*′-[(5-methyl-2-furyl)methylene]isonicotinohydrazide (0.02 mol) and hydrochloric acid (0.01 mol) was stirred with ethanol (50 ml) at 298 K for 2 h, then afford the title compound (2.61 g, yield 92%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol and trichloromethane (1:1) at room temperature.

S3. Refinement

The H atoms of the water molecule were found from a difference Fourier map and refined freely. The other H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H and N—H distances of 0.93–0.96 and 0.86 Å, and with U_{iso} = 1.2–1.5U_{eq}(C,N).

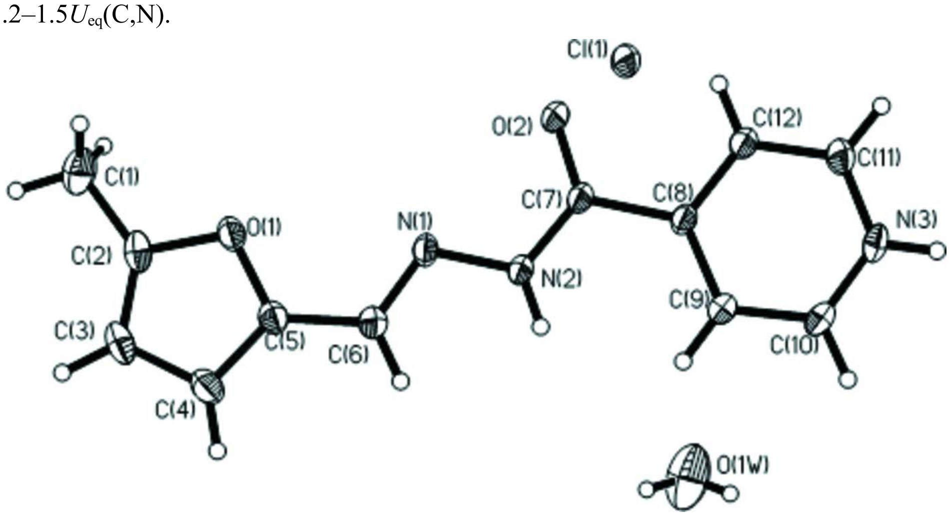

Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

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Crystal data

$C_{12}H_{12}N_3O_2^+\cdot Cl^-\cdot H_2O$
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β = 123.55 (2)°
V = 1397.5 (7) Å^3
Z = 4
F(000) = 592
D_x = 1.348 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2715 reflections
θ = 3.1–27.5°
μ = 0.28 mm^{-1}
T = 293 K
Bar, yellow
0.20 × 0.15 × 0.11 mm

Data collection

Bruker P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 3 pixels mm^{-1}
ω scans
13328 measured reflections
3187 independent reflections
2715 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −10→11
k = −18→18
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.107
S = 1.07
3187 reflections
180 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 0.3029P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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}
Cl1	0.22892 (6)	−0.42533 (3)	−0.18006 (4)	0.06023 (16)
O1	0.51022 (13)	0.04123 (6)	0.13453 (8)	0.0380 (2)
O2	0.05971 (14)	−0.22008 (7)	−0.02944 (10)	0.0536 (3)
N1	0.16922 (14)	−0.04732 (7)	−0.03026 (9)	0.0327 (2)
N2	−0.00554 (14)	−0.08118 (7)	−0.11953 (9)	0.0327 (2)

H2A	−0.0844	−0.0467	−0.1778	0.039*
N3	−0.57485 (15)	−0.28167 (8)	−0.36865 (9)	0.0392 (3)
H3A	−0.6822	−0.3057	−0.4198	0.047*
C1	0.8247 (2)	0.07121 (14)	0.30399 (16)	0.0616 (5)
H1B	0.9141	0.1209	0.3397	0.092*
H1C	0.8779	0.0215	0.2847	0.092*
H1D	0.7941	0.0493	0.3580	0.092*
C2	0.65193 (19)	0.10503 (11)	0.19547 (13)	0.0419 (3)
C3	0.5985 (2)	0.18739 (10)	0.14057 (14)	0.0469 (4)
H3B	0.6695	0.2415	0.1641	0.056*
C4	0.4137 (2)	0.17617 (10)	0.04032 (13)	0.0441 (3)
H4A	0.3395	0.2216	−0.0145	0.053*
C5	0.36538 (19)	0.08648 (9)	0.03927 (12)	0.0354 (3)
C6	0.19437 (19)	0.03842 (9)	−0.04240 (11)	0.0359 (3)
H6A	0.0978	0.0707	−0.1067	0.043*
C7	−0.04668 (16)	−0.16914 (9)	−0.11160 (10)	0.0321 (3)
C8	−0.23608 (16)	−0.20547 (8)	−0.20798 (10)	0.0301 (3)
C9	−0.37266 (17)	−0.15335 (9)	−0.30253 (11)	0.0365 (3)
H9A	−0.3495	−0.0920	−0.3115	0.044*
C10	−0.54274 (18)	−0.19394 (10)	−0.38265 (11)	0.0404 (3)
H10A	−0.6356	−0.1601	−0.4468	0.049*
C11	−0.4484 (2)	−0.33342 (10)	−0.27924 (13)	0.0441 (3)
H11A	−0.4768	−0.3942	−0.2721	0.053*
C12	−0.27438 (19)	−0.29683 (9)	−0.19686 (12)	0.0413 (3)
H12A	−0.1837	−0.3330	−0.1346	0.050*
O1W	−0.3179 (3)	−0.06393 (18)	−0.5308 (2)	0.1060 (7)
H2W1	−0.282 (4)	−0.0189 (19)	−0.481 (2)	0.093 (8)*
H1W1	−0.419 (4)	−0.0676 (18)	−0.566 (2)	0.084 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0599 (3)	0.0341 (2)	0.0513 (2)	−0.00819 (16)	0.00847 (19)	−0.00564 (15)
O1	0.0337 (5)	0.0317 (5)	0.0423 (5)	−0.0068 (4)	0.0170 (4)	−0.0055 (4)
O2	0.0291 (5)	0.0402 (5)	0.0497 (6)	−0.0051 (4)	−0.0044 (4)	0.0125 (5)
N1	0.0253 (5)	0.0331 (5)	0.0312 (5)	−0.0053 (4)	0.0104 (4)	−0.0060 (4)
N2	0.0240 (5)	0.0305 (5)	0.0292 (5)	−0.0022 (4)	0.0056 (4)	−0.0012 (4)
N3	0.0252 (5)	0.0466 (7)	0.0317 (5)	−0.0085 (5)	0.0070 (4)	−0.0110 (5)
C1	0.0368 (8)	0.0758 (12)	0.0565 (10)	−0.0073 (8)	0.0159 (7)	−0.0122 (9)
C2	0.0348 (6)	0.0454 (7)	0.0472 (8)	−0.0143 (6)	0.0236 (6)	−0.0167 (6)
C3	0.0515 (8)	0.0396 (7)	0.0572 (9)	−0.0204 (7)	0.0348 (7)	−0.0155 (7)
C4	0.0531 (8)	0.0334 (7)	0.0484 (8)	−0.0089 (6)	0.0297 (7)	−0.0025 (6)
C5	0.0377 (7)	0.0321 (6)	0.0359 (6)	−0.0053 (5)	0.0200 (6)	−0.0047 (5)
C6	0.0348 (6)	0.0332 (6)	0.0335 (6)	−0.0030 (5)	0.0151 (5)	−0.0032 (5)
C7	0.0221 (5)	0.0321 (6)	0.0305 (6)	−0.0002 (5)	0.0072 (5)	−0.0007 (5)
C8	0.0217 (5)	0.0319 (6)	0.0282 (5)	−0.0003 (5)	0.0084 (5)	−0.0021 (5)
C9	0.0278 (6)	0.0365 (6)	0.0326 (6)	−0.0007 (5)	0.0089 (5)	0.0038 (5)
C10	0.0262 (6)	0.0476 (8)	0.0302 (6)	0.0008 (6)	0.0047 (5)	0.0022 (5)

C11	0.0386 (7)	0.0333 (7)	0.0435 (7)	−0.0094 (6)	0.0120 (6)	−0.0066 (6)
C12	0.0319 (6)	0.0307 (6)	0.0384 (7)	−0.0011 (5)	0.0050 (5)	0.0010 (5)
O1W	0.0743 (12)	0.152 (2)	0.0905 (13)	−0.0102 (12)	0.0446 (11)	−0.0516 (13)

Geometric parameters (Å, º)

O1—C5	1.3655 (17)	C3—H3B	0.9300
O1—C2	1.3742 (16)	C4—C5	1.3566 (19)
O2—C7	1.2225 (16)	C4—H4A	0.9300
N1—C6	1.2823 (17)	C5—C6	1.4324 (18)
N1—N2	1.3911 (14)	C6—H6A	0.9300
N2—C7	1.3375 (16)	C7—C8	1.5044 (16)
N2—H2A	0.8600	C8—C12	1.3868 (18)
N3—C11	1.3239 (18)	C8—C9	1.3869 (17)
N3—C10	1.3314 (19)	C9—C10	1.3741 (18)
N3—H3A	0.8600	C9—H9A	0.9300
C1—C2	1.479 (2)	C10—H10A	0.9300
C1—H1B	0.9600	C11—C12	1.3780 (18)
C1—H1C	0.9600	C11—H11A	0.9300
C1—H1D	0.9600	C12—H12A	0.9300
C2—C3	1.343 (2)	O1W—H2W1	0.87 (3)
C3—C4	1.412 (2)	O1W—H1W1	0.72 (3)
C5—O1—C2	106.56 (11)	C4—C5—C6	130.05 (13)
C6—N1—N2	113.64 (11)	O1—C5—C6	120.28 (11)
C7—N2—N1	117.70 (10)	N1—C6—C5	122.56 (12)
C7—N2—H2A	121.1	N1—C6—H6A	118.7
N1—N2—H2A	121.1	C5—C6—H6A	118.7
C11—N3—C10	122.76 (11)	O2—C7—N2	123.37 (11)
C11—N3—H3A	118.6	O2—C7—C8	119.01 (11)
C10—N3—H3A	118.6	N2—C7—C8	117.61 (10)
C2—C1—H1B	109.5	C12—C8—C9	119.33 (11)
C2—C1—H1C	109.5	C12—C8—C7	116.11 (11)
H1B—C1—H1C	109.5	C9—C8—C7	124.53 (11)
C2—C1—H1D	109.5	C10—C9—C8	118.83 (13)
H1B—C1—H1D	109.5	C10—C9—H9A	120.6
H1C—C1—H1D	109.5	C8—C9—H9A	120.6
C3—C2—O1	110.02 (13)	N3—C10—C9	120.13 (12)
C3—C2—C1	133.87 (14)	N3—C10—H10A	119.9
O1—C2—C1	116.11 (14)	C9—C10—H10A	119.9
C2—C3—C4	106.92 (13)	N3—C11—C12	119.73 (13)
C2—C3—H3B	126.5	N3—C11—H11A	120.1
C4—C3—H3B	126.5	C12—C11—H11A	120.1
C5—C4—C3	106.83 (14)	C11—C12—C8	119.21 (12)
C5—C4—H4A	126.6	C11—C12—H12A	120.4
C3—C4—H4A	126.6	C8—C12—H12A	120.4
C4—C5—O1	109.67 (12)	H2W1—O1W—H1W1	111 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H2*W*1···Cl1^{i}	0.86 (3)	2.40 (3)	3.229 (3)	162 (3)
O1*W*—H1*W*1···Cl1^{ii}	0.72 (3)	2.51 (3)	3.225 (3)	177 (2)
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