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2-Hydroxy-3-methoxybenzaldehyde (pyridinium-4-ylcarbonyl)hydrazone chloride hemihydrate

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The crystal structure of the title compound, $C_{14}H_{14}ClN_3O_3^+\cdot Cl^-\cdot 0.5H_2O$, exhibits O—H···O, C—H···O, C—H···Cl, N—H···Cl and O—H···Cl hydrogen bonds. The chloride anions participate in extensive hydrogen bonding with the aminium cations and link molecules through multiple $N—H^+\cdots Cl^-$ interactions.

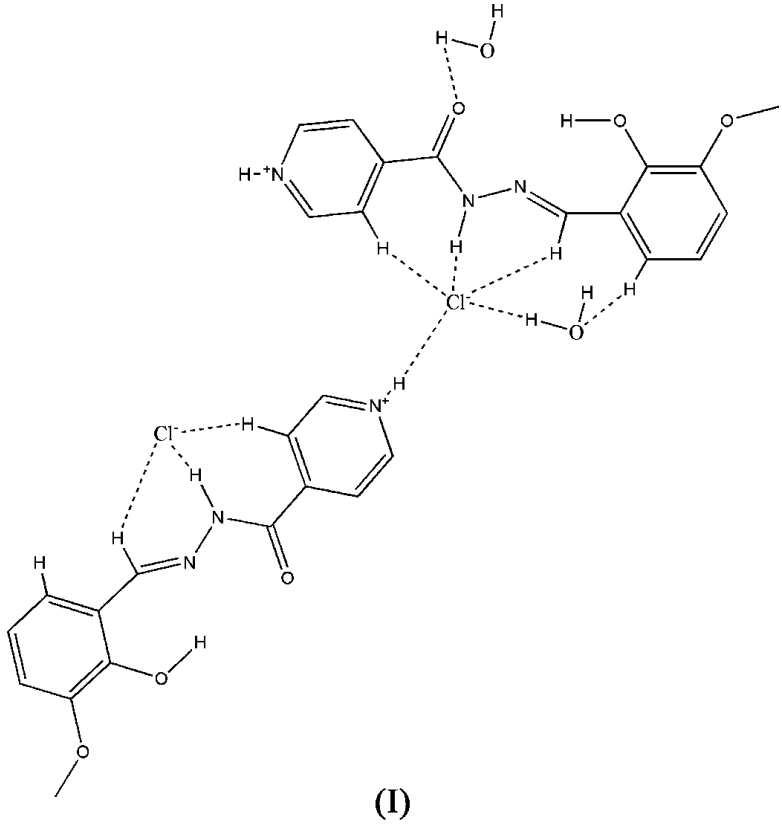
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
Disorder in solvent or counterion
R factor = 0.050
wR factor = 0.146
Data-to-parameter ratio = 13.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The molecular geometry of the title compound (Fig. 1) is listed in Table 1. The crystal packing (Table 2 and Fig. 2) is dominated by $N—H^+\cdots Cl^-$, C—H···O, C—H···Cl and O—H···Cl hydrogen bonds, including also a three-centre C—H···O hydrogen bond (Jeffrey & Saenger, 1997). The intermolecular N—H···Cl, C—H···Cl and N—H···Cl bonds directly link two molecules (see scheme) through the chloride ion. The benzene rings are stacked along the c-axis direction by π–π interactions, forming a lipophilic layer, whereas hydrophilic layers are interconnected by $N—H^+\cdots Cl^-$ hydrogen bonds.



(I)

Experimental

The title compound was prepared by the condensation of the *o*-vanillic and isonicotinic acid hydrazide (molar ratio 1:1) in ethanol/hydrochloric acid (3:1) at room temperature. The resulting solid was recrystallized from dichloromethane–ethanol (1:1, *v*/*v*). Yield 85%, m.p. 577–579 K. Analysis calculated for $C_{14}H_{15}N_3O_{3.5}Cl$: C 53.09, H 4.77, N 13.26%; found: C 52.98, H 4.69, N 23.15%.

Crystal data

$C_{14}H_{14}N_3O_3^+\cdot Cl^-\cdot 0.5H_2O$
M_r = 316.74
Monoclinic, $P2_1/n$
a = 12.925 (3) Å
b = 7.4369 (17) Å
c = 15.503 (4) Å
β = 90.978 (4)°
V = 1489.9 (6) Å^3
Z = 4
D_x = 1.412 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 293 (2) K
Block, orange
0.17 × 0.15 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.955, T_{max} = 0.973
7428 measured reflections
2580 independent reflections
1326 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.146
S = 1.00
2580 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.001P)^2 + 0.7491P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.346 (5)	N2—C7	1.280 (5)
N1—N2	1.370 (4)	O1—C1	1.223 (4)
C1—N1—N2	118.9 (3)	O1—C1—N1	122.4 (4)
C7—N2—N1	116.9 (3)	C3—C2—C1	124.1 (4)
C5—N3—C4	121.9 (4)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1^{i}	0.86	2.49	3.312 (3)	161
N3—H3···Cl1^{ii}	0.86	2.17	3.002 (3)	163
O2—H2···N2	0.82	1.91	2.632 (4)	146
O4—H15···Cl1^{iii}	0.85	2.33	3.146 (7)	161
O4—H16···O1	0.85	2.33	3.145 (7)	162
C5—H5···O4^{iv}	0.93	2.21	3.136 (8)	173
C7—H7···Cl1^{i}	0.93	2.71	3.549 (4)	151
C3—H3*A*···Cl1^{i}	0.93	2.74	3.662 (4)	172
C4—H4···O3^{v}	0.93	2.51	3.065 (5)	118
C4—H4···O2^{v}	0.93	2.33	3.248 (5)	170
C13—H13···O4^{vi}	0.93	2.57	3.237 (9)	129

Symmetry codes: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x, y+1, z$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (iv) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (vi) $-x+1, -y+1, -z+1$.

All H atoms were positioned geometrically and treated as riding on their parent atoms, with aromatic C—H distances of 0.93 Å and methyl C—H distances of 0.96 Å (O,N—H as given in Table 2). The U_{iso}(H) values were set at 1.2U_{eq}(C,N) for the aromatic and N-bound H atoms, and 1.5U_{eq}(C,O) for other H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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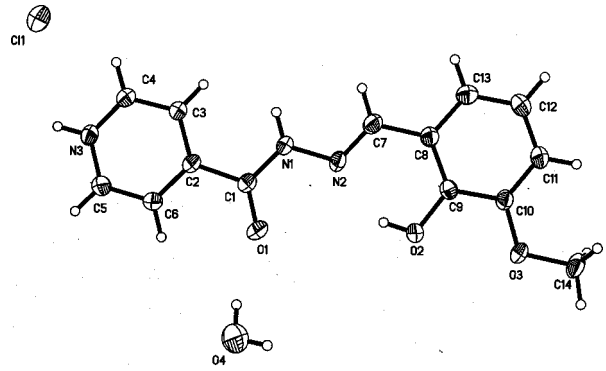

Figure 1
The structure of the title compound, with the atom numbering scheme and displacement ellipsoids shown at the 30% probability level.

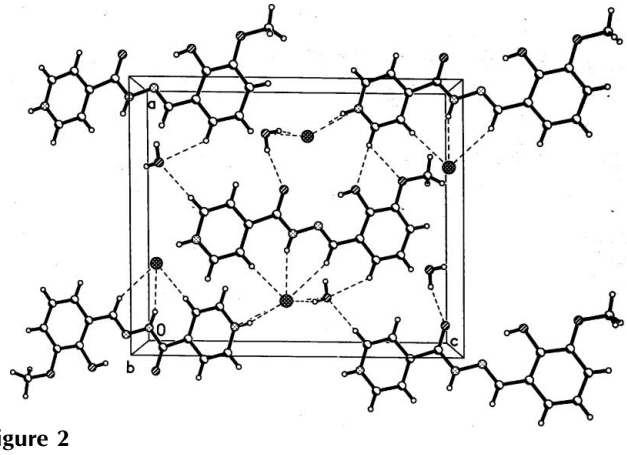

Figure 2
Crystal packing of the title compound. Dashed lines indicate hydrogen bonds.

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