Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-[5-(2,2-Dimethylpropanamido)-1*H*-pyrazol-3-yl]pyridinium chloride

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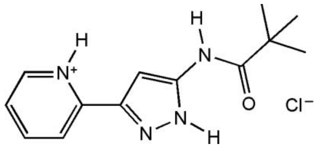
Received 12 April 2007; accepted 9 May 2007

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.054; wR factor = 0.156; data-to-parameter ratio = 18.3.

The title compound, $C_{13}H_{17}N_4O^+\cdot Cl^-$, contains a cation protonated at its pyridine ring N atom. Pyridinium and amide NH groups (from two different cations) form hydrogen bonds with the Cl^- ion, together with three weaker C—H···Cl interactions. The pyrazole rings of the cations associate into cyclic dimers through N—H···N hydrogen bonding. The N—H···N and N—H···Cl interactions combine to associate the cations and anions into alternating chains along the [101] vector, generated by crystallographic inversion centres. These chains are then further aggregated by a C—H···Cl interaction into two-dimensional layers parallel to (010).

Related literature

For related literature, see: Jones *et al.* (2006, 2007); Liu *et al.* (2004); Llamas-Saiz *et al.* (1994); Nieto *et al.* (2006); Pask, Camm, Bullen *et al.* (2006); Pask, Camm, Kilner & Halcrow (2006); Reger *et al.* (1994); Renard *et al.* (2002, 2006); Sobolev & White (2004).



Experimental

Crystal data

$C_{13}H_{17}N_4O^+\cdot Cl^-$
M_r = 280.76
Triclinic, $P\overline{1}$
a = 9.0226 (1) Å
b = 9.1638 (2) Å
c = 9.7919 (2) Å
α = 91.6357 (7)°
β = 95.5828 (7)°
γ = 115.9693 (13)°
V = 722.11 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 150 (2) K
0.60 × 0.51 × 0.35 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min}$ = 0.665, $T_{\max}$ = 1.070 (expected range = 0.567–0.912)
10651 measured reflections
3204 independent reflections
2590 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.092

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.156
S = 1.07
3204 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.32 e Å^{-3}
$\Delta\rho_{\min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl19$^{\rm i}$	0.88	2.15	2.9978 (16)	163
C4—H4···Cl19$^{\rm ii}$	0.95	2.76	3.6112 (18)	149
N9—H9···N8$^{\rm iii}$	0.88	2.33	2.936 (2)	127
N9—H9···O18	0.88	2.11	2.655 (2)	119
C11—H11···Cl19$^{\rm i}$	0.95	2.78	3.5620 (19)	140
N12—H12···Cl19	0.88	2.35	3.1799 (15)	157
C16—H16B···Cl19	0.98	2.91	3.837 (2)	157

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

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2003); software used to prepare material for publication: local program.

The authors acknowledge the Leverhulme Trust and the University of Leeds for funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG3091).

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doi:10.1107/S1600536807022866

supplementary materials

Acta Cryst. (2007). E**63**, o2933 [**doi:10.1107/S1600536807022866**]

2-[5-(2,2-Dimethylpropanamido)-1*H*-pyrazol-3-yl]pyridinium chloride

C. M. Pask, **C. A. Kilner** and **M. A. Halcrow**

Comment

The pyrazole ring is an attractive functionality for transition metal supramolecular chemistry in that it possesses a Lewis basic pyridinic N-donor and a Lewis acidic pyrrolic N—H group in adjacent sites. It can therefore be a ditopic ligand for metal salts, binding a metal cation and anion simultaneously, placing the two guests 3.5–4.5Å apart (Reger *et al.*, 1994; Renard *et al.*, 2002; Nieto *et al.*, 2006). As a continuation of our own studies of metal-organic supramolecular chemistry of 5-substituted N—H pyrazoles (Liu *et al.*, 2004; Renard *et al.*, 2002 & 2006 and refs. therein), we have investigated the synthesis of bidentate ligands derived from 3-(pyrid-2-yl)-1*H*-pyrazole and their metal complexes (Pask, Camm, Kilner & Halcrow, 2006; Pask, Camm, Bullen *et al.*, 2006; Jones *et al.*, 2006 & 2007). During this work, we were interested in investigating how these ligands interact with anions in the absence of a metal cation and have examined the crystal structure of the hydrochloride salt of the title compound (I).

The asymmetric unit of (I) contains one formula unit, with the cation and anion both lying on general positions. All bond lengths and angles in the organic cation lie within the expected ranges. The pyridinium and pyrazole rings in (I) are not coplanar, having a dihedral angle of 17.45 (10)° between their least squares planes. The dihedral angle between the pyrazole and amido groups is smaller, at 10.48 (11)°. The conformation of (I), and of the related neutral compound *N*-(5-{pyridin-2-yl}pyrazol-3-yl)methylamide (II) (Pask, Camm, Kilner & Halcrow, 2006), differ in two important ways: a) the N atoms of the pyridine and pyrazole rings have an anti disposition in (I), but are *syn* to each other in (II) and b) the amide N—H group is anti to the pyrazole N atoms in (I). Since the amide bond has its usual *transoid* conformation, that facilitates the formation of an intramolecular hydrogen bond from the pyrazole ring to the amide carbonyl group, N8—H8···O18. However, in (II) these two groups are *syn* to each other, placing the carbonyl group on the opposite side of the pyrazole ring so that no intramolecular hydrogen bond is formed.

There are five intermolecular contacts to the Cl^- ion Cl19 that are shorter than the sum of van der Waals radii of a Cl (1.8 Å) and H (1.2 Å) atom [Pauling (1960). The Nature of the Chemical Bond, 3rd ed. Ithaca: Cornell University Press.] (Fig. 2). These are: two N—H···Cl hydrogen bonds, from N12—H12 and $N1^i$—$H1^i$; and, three weaker C—H···Cl contacts from $C11^i$—$H11^i$, $C4^{ii}$—$H4^{ii}$ and C16—H16B, with H···Cl = 2.76–2.91 Å [symmetry codes: (i) $1 - x$, $2 - y$, $-z$; (ii) $2 - x$, $2 - y$, $-z$]. The geometry about Cl19 is therefore is very distorted, with $\tau = 0.44$ placing it midway between the square pyramidal ($\tau = 0$) and trigonal bipyramidal ($\tau =1$) extremes [Addison, Rao, Reedijk, van Rijn, J. & Verschoor (1984). *J. Chem. Soc. Dalton Trans.* pp. 1349–1356]. In addition to the aforementioned intramolecular hydrogen bond, pyrazole group N8—H8 also donates an intermolecular hydrogen bond to a neighbouring pyrazole pyridinic N atom $N9^{iii}$ [symmetry code: (iii) $2 - x$, $2 - y$, $1 - z$] (Fig. 2). The resultant cyclic dimer of pyrazole rings is a common motif in pyrazole crystal chemistry (see *e.g.* Llamas-Saiz *et al.*, 1994; Sobolev & White, 2004). The N—H···N and N—H···Cl hydrogen bonds combine to associate the cations and anions into alternating chains along the [1 0 1] vector, generated by the crystallographic inversion centres at 1/2, 1, 0 and 1, 1, 0.5 (Fig. 2). These chains are then further aggregated by the C4—H4···$Cl19^{ii}$ interaction into 2-D layers parallel to (0 1 0).

Experimental

Compound (I) was prepared by the previously reported method (Pask, Camm, Kilner & Halcrow, 2006). Recrystallization of the crude solid by slow evaporation of a 2*M* HCl solution yielded colourless rectangular prisms.

Refinement

The pyridinium N1 and C6 sites were distinguished from their isotropic thermal parameters, and on the basis that N1 in the model donates a short hydrogen bond to Cl19 while C6 does not take part in hydrogen bonding. All non-H atoms were refined anisotropically. All H atoms were located in the difference map, but several of them (notably the methyl groups) did not refine satisfactorily when their positions were allowed to refine freely. Hence, in the final model all H atoms were placed in calculated positions and refined using a riding model with methyl group torsions allowed to refine freely. The fixed bond distances and isotropic thermal parameters for the H atom refinements were: *C*—*H*(aryl) = 0.95Å and $U_{\mathrm{iso}} = 1.2U_{\mathrm{eq}}$(C); *C*—*H*(methyl) = 0.98Å and $1.5U_{\mathrm{eq}}$(C); and, N—H = 0.88Å and $U_{\mathrm{iso}} = 1.2U_{\mathrm{eq}}$(N).

Figures

Fig. 1. View of the asymmetric unit of (I), showing the atom numbering scheme employed. Displacement ellipsoids are at the 50% probability level, except for H atoms which have arbitrary radii.

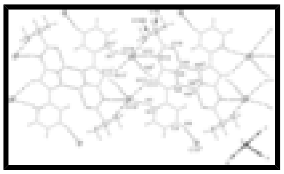


Fig. 2. Partial packing diagram of (I), showing the hydrogen bonds in the crystal lattice. Cl atoms are plotted with 50% displacement ellipsoids, and all other atoms have arbitrary radii. The orientations of the unit cell axes in this arbitrary view are shown in inset. Symmetry codes: (i) $1 - x, 2 - y, -z$; (ii) $2 - x, 2 - y, -z$; (iii) $2 - x, 2 - y, 1 - z$.

2-[5-(2,2-Dimethylpropanamido)-1*H*-pyrazol-3-yl]pyridinium chloride

Crystal data

$C_{13}H_{17}N_4O^+ \cdot Cl^-$
$M_r = 280.76$
Triclinic, $P\bar{1}$
Hall symbol: -P 1
$a = 9.0226$ (1) Å
$b = 9.1638$ (2) Å
$c = 9.7919$ (2) Å
$\alpha = 91.6357$ (7)°
$\beta = 95.5828$ (7)°
$\gamma = 115.9693$ (13)°
$V = 722.11$ (2) Å^3

$Z = 2$
$F_{000} = 296$
$D_x = 1.291$ Mg m^{-3}
Mo *K*α radiation
λ = 0.71073 Å
Cell parameters from 10651 reflections
θ = 2.1–27.5°
$\mu = 0.26$ mm^{-1}
$T = 150$ (2) K
Rectangular prism, colourless
0.60 × 0.51 × 0.35 mm

Data collection

Nonius KappaCCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 9.091 pixels mm^{-1}

T = 150(2) K

ω and φ scans

Absorption correction: multi-scan (SORTAV; Blessing, 1995)

T_{min} = 0.665, T_{max} = 1.070

10651 measured reflections

3204 independent reflections

2590 reflections with $I > 2\sigma(I)$

R_{int} = 0.092

θ_{max} = 27.5°

θ_{min} = 2.1°

$h = -10 \rightarrow 11$

$k = -11 \rightarrow 11$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.054

$wR(F^2)$ = 0.156

S = 1.07

3204 reflections

175 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

$w = 1/[\sigma^2(F_o^2) + (0.0939P)^2 + 0.0869P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\text{max}}$ = 0.32 e Å^{-3}

$\Delta\rho_{\text{min}}$ = −0.39 e Å^{-3}

Extinction correction: none

Special details

Experimental. Detector set at 30 mm from sample with different 2theta offsets 1 degree phi exposures for chi=0 degree settings 1 degree omega exposures for chi=90 degree settings

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 > 2sigma(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.

No disorder was detected during refinement, and no restraints were applied. All non-H atoms were refined anisotropically. All H atoms were located in the difference map, but several of them (notably the methyl groups) did not refine satisfactorily when their positions were allowed to refine freely. Hence, in the final model all H atoms were placed in calculated positions and refined using a riding model.

The pyridinium N1 and C6 sites were distinguished from their isotropic thermal parameters, and on the basis that N1 in the model donates a short hydrogen bond to Cl19 while C6 does not take part in hydrogen bonding.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	1.00995 (18)	1.23608 (18)	0.03259 (16)	0.0375 (4)
H1	0.9083	1.2271	0.0219	0.045*
C2	1.1167 (2)	1.3256 (2)	−0.0529 (2)	0.0440 (4)
H2	1.0822	1.3785	−0.1223	0.053*
C3	1.2756 (2)	1.3404 (2)	−0.0398 (2)	0.0456 (5)
H3	1.3517	1.4028	−0.1002	0.055*
C4	1.3232 (2)	1.2635 (2)	0.0623 (2)	0.0450 (5)
H4	1.4328	1.2726	0.0724	0.054*
C5	1.2120 (2)	1.1731 (2)	0.15019 (19)	0.0393 (4)
H5	1.2449	1.1207	0.2210	0.047*
C6	1.0510 (2)	1.1596 (2)	0.13364 (18)	0.0343 (4)
C7	0.9246 (2)	1.0642 (2)	0.22032 (17)	0.0345 (4)
N8	0.97461 (18)	1.02065 (19)	0.33875 (16)	0.0382 (4)
N9	0.83143 (18)	0.92777 (18)	0.39042 (16)	0.0373 (3)
H9	0.8268	0.8817	0.4685	0.045*
C10	0.6959 (2)	0.9144 (2)	0.30710 (18)	0.0353 (4)
C11	0.7501 (2)	1.0016 (2)	0.19638 (18)	0.0359 (4)
H11	0.6853	1.0166	0.1207	0.043*
N12	0.53424 (17)	0.82319 (18)	0.33567 (15)	0.0383 (4)
H12	0.4528	0.8062	0.2705	0.046*
C13	0.4932 (2)	0.7581 (2)	0.45821 (19)	0.0371 (4)
C14	0.3076 (2)	0.6580 (2)	0.46746 (19)	0.0392 (4)
C15	0.2148 (2)	0.7592 (2)	0.4264 (2)	0.0503 (5)
H15A	0.1000	0.7028	0.4473	0.075*
H15B	0.2150	0.7733	0.3276	0.075*
H15C	0.2701	0.8661	0.4782	0.075*
C16	0.2427 (2)	0.5028 (2)	0.3698 (2)	0.0449 (5)
H16A	0.3068	0.4423	0.3941	0.067*
H16B	0.2546	0.5325	0.2748	0.067*
H16C	0.1253	0.4346	0.3784	0.067*
C17	0.2872 (3)	0.6129 (3)	0.6162 (2)	0.0586 (6)
H17A	0.3307	0.7124	0.6780	0.088*
H17B	0.3485	0.5500	0.6413	0.088*
H17C	0.1692	0.5475	0.6246	0.088*
O18	0.59996 (16)	0.77827 (17)	0.55327 (14)	0.0465 (4)
Cl19	0.30815 (5)	0.74128 (6)	0.04736 (5)	0.04462 (19)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0274 (7)	0.0361 (8)	0.0447 (8)	0.0104 (6)	0.0038 (6)	0.0013 (6)
C2	0.0388 (10)	0.0383 (9)	0.0489 (11)	0.0110 (8)	0.0078 (8)	0.0043 (8)

C3	0.0360 (10)	0.0422 (10)	0.0504 (11)	0.0089 (8)	0.0113 (8)	−0.0002 (8)
C4	0.0281 (9)	0.0457 (10)	0.0546 (12)	0.0106 (8)	0.0068 (8)	−0.0074 (9)
C5	0.0302 (9)	0.0437 (10)	0.0408 (10)	0.0146 (7)	0.0006 (7)	−0.0044 (8)
C6	0.0293 (8)	0.0320 (8)	0.0370 (9)	0.0102 (7)	0.0018 (7)	−0.0036 (7)
C7	0.0286 (8)	0.0345 (8)	0.0380 (9)	0.0122 (7)	0.0029 (7)	−0.0011 (7)
N8	0.0278 (7)	0.0420 (8)	0.0409 (8)	0.0121 (6)	0.0037 (6)	0.0038 (6)
N9	0.0260 (7)	0.0418 (8)	0.0413 (8)	0.0126 (6)	0.0028 (6)	0.0066 (6)
C10	0.0273 (8)	0.0356 (8)	0.0406 (9)	0.0125 (7)	0.0014 (7)	−0.0021 (7)
C11	0.0281 (8)	0.0370 (9)	0.0393 (9)	0.0121 (7)	0.0017 (7)	0.0004 (7)
N12	0.0246 (7)	0.0429 (8)	0.0411 (8)	0.0099 (6)	0.0008 (6)	0.0043 (7)
C13	0.0329 (9)	0.0335 (8)	0.0425 (10)	0.0127 (7)	0.0045 (7)	0.0009 (7)
C14	0.0306 (9)	0.0368 (9)	0.0440 (10)	0.0091 (7)	0.0064 (7)	0.0021 (7)
C15	0.0330 (10)	0.0444 (11)	0.0724 (14)	0.0160 (8)	0.0094 (9)	−0.0037 (10)
C16	0.0375 (10)	0.0339 (9)	0.0554 (11)	0.0090 (8)	0.0051 (8)	0.0011 (8)
C17	0.0401 (11)	0.0693 (14)	0.0500 (12)	0.0078 (10)	0.0117 (9)	0.0073 (10)
O18	0.0332 (7)	0.0529 (8)	0.0448 (8)	0.0119 (6)	−0.0006 (6)	0.0082 (6)
Cl19	0.0327 (3)	0.0517 (3)	0.0478 (3)	0.0176 (2)	0.00161 (19)	0.0072 (2)

Geometric parameters (Å, °)

N1—C6	1.343 (2)	C10—N12	1.389 (2)
N1—C2	1.344 (2)	C11—H11	0.9500
N1—H1	0.8800	N12—C13	1.366 (2)
C2—C3	1.372 (3)	N12—H12	0.8800
C2—H2	0.9500	C13—O18	1.220 (2)
C3—C4	1.380 (3)	C13—C14	1.531 (2)
C3—H3	0.9500	C14—C17	1.532 (3)
C4—C5	1.383 (3)	C14—C15	1.535 (3)
C4—H4	0.9500	C14—C16	1.538 (3)
C5—C6	1.395 (2)	C15—H15A	0.9800
C5—H5	0.9500	C15—H15B	0.9800
C6—C7	1.464 (2)	C15—H15C	0.9800
C7—N8	1.338 (2)	C16—H16A	0.9800
C7—C11	1.412 (2)	C16—H16B	0.9800
N8—N9	1.356 (2)	C16—H16C	0.9800
N9—C10	1.358 (2)	C17—H17A	0.9800
N9—H9	0.8800	C17—H17B	0.9800
C10—C11	1.365 (3)	C17—H17C	0.9800
C6—N1—C2	123.14 (16)	C13—N12—C10	124.38 (15)
C6—N1—H1	118.4	C13—N12—H12	117.8
C2—N1—H1	118.4	C10—N12—H12	117.8
N1—C2—C3	119.82 (19)	O18—C13—N12	121.14 (16)
N1—C2—H2	120.1	O18—C13—C14	122.71 (17)
C3—C2—H2	120.1	N12—C13—C14	116.15 (15)
C2—C3—C4	119.05 (18)	C13—C14—C17	107.90 (16)
C2—C3—H3	120.5	C13—C14—C15	109.60 (15)
C4—C3—H3	120.5	C17—C14—C15	110.49 (17)
C3—C4—C5	120.29 (18)	C13—C14—C16	108.53 (14)
C3—C4—H4	119.9	C17—C14—C16	109.94 (17)

C5—C4—H4	119.9	C15—C14—C16	110.32 (16)
C4—C5—C6	119.26 (19)	C14—C15—H15A	109.5
C4—C5—H5	120.4	C14—C15—H15B	109.5
C6—C5—H5	120.4	H15A—C15—H15B	109.5
N1—C6—C5	118.43 (16)	C14—C15—H15C	109.5
N1—C6—C7	118.64 (15)	H15A—C15—H15C	109.5
C5—C6—C7	122.93 (17)	H15B—C15—H15C	109.5
N8—C7—C11	112.09 (16)	C14—C16—H16A	109.5
N8—C7—C6	118.10 (15)	C14—C16—H16B	109.5
C11—C7—C6	129.75 (17)	H16A—C16—H16B	109.5
C7—N8—N9	104.16 (14)	C14—C16—H16C	109.5
N8—N9—C10	111.87 (15)	H16A—C16—H16C	109.5
N8—N9—H9	124.1	H16B—C16—H16C	109.5
C10—N9—H9	124.1	C14—C17—H17A	109.5
N9—C10—C11	107.81 (15)	C14—C17—H17B	109.5
N9—C10—N12	123.13 (16)	H17A—C17—H17B	109.5
C11—C10—N12	129.05 (17)	C14—C17—H17C	109.5
C10—C11—C7	104.07 (16)	H17A—C17—H17C	109.5
C10—C11—H11	128.0	H17B—C17—H17C	109.5
C7—C11—H11	128.0		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl19^{i}	0.88	2.15	2.9978 (16)	163
C4—H4···Cl19^{ii}	0.95	2.76	3.6112 (18)	149
N9—H9···N8^{iii}	0.88	2.33	2.936 (2)	127
N9—H9···O18	0.88	2.11	2.655 (2)	119
C11—H11···Cl19^{i}	0.95	2.78	3.5620 (19)	140
N12—H12···Cl19	0.88	2.35	3.1799 (15)	157
C16—H16B···Cl19	0.98	2.91	3.837 (2)	157

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*; (ii) −*x*+2, −*y*+2, −*z*; (iii) −*x*+2, −*y*+2, −*z*+1.

Fig. 1

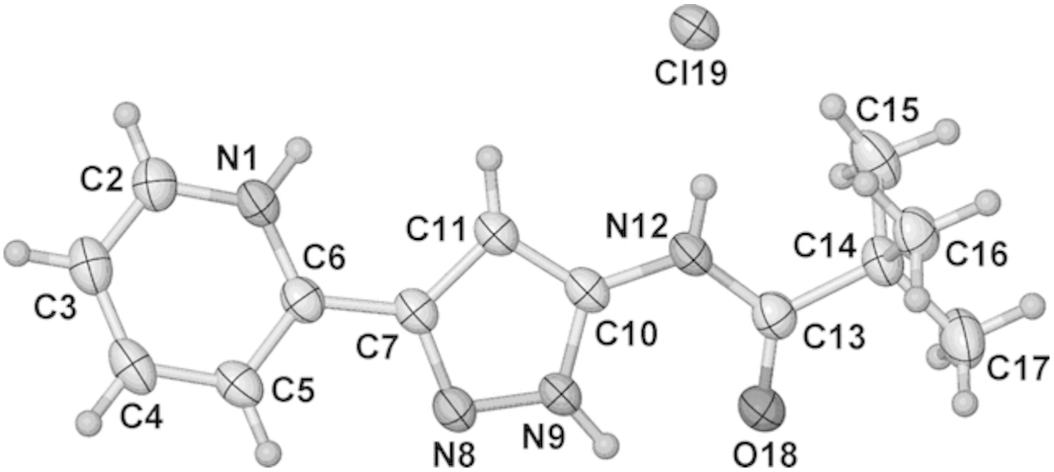
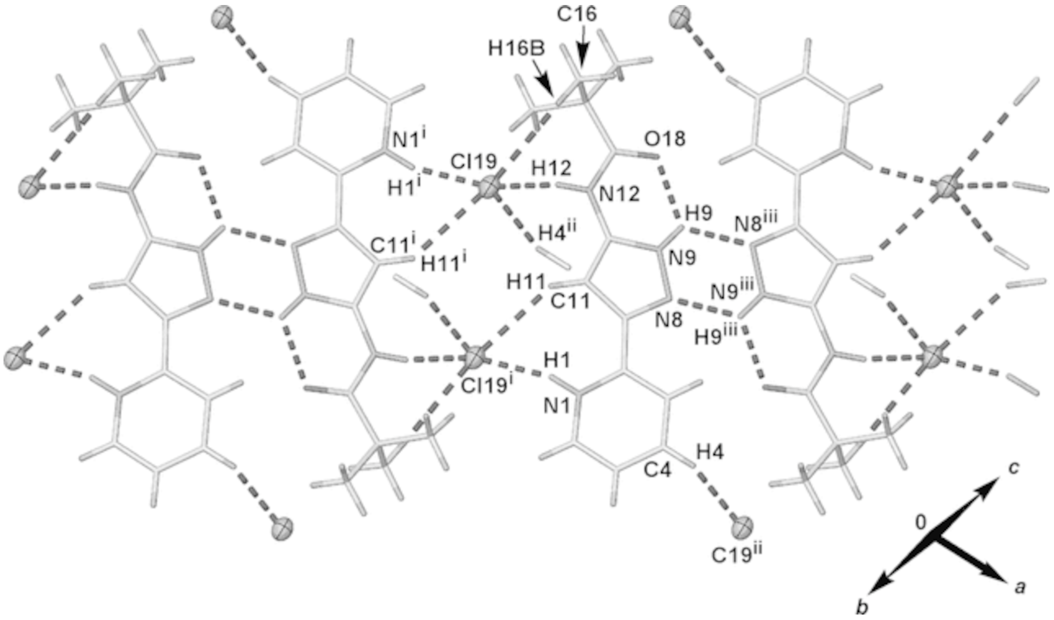


Fig. 2


C16
H16B
N1i
Cl19
H12
O18
H1i
N12
H9
N8iii
C11i
H4ii
N9
H11i
H11
C11
N9iii
N8
H9iii
H1
Cl19i
N1
H4
C4
C19ii
c
0
a
b