Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 4-Carbamoylpyridin-1-ium 2,2,2-trichloroacetate–isonicotinamide (1/1)

**Franc Perdih**

Faculty of Chemistry and Chemical Technology, University of Ljubljana, Aškerčeva 5, PO Box 537, SI-1000 Ljubljana, Slovenia, and CO EN–FIST, Dunajska 156, SI-1000 Ljubljana, Slovenia
Correspondence e-mail: franc.perdih@fkkt.uni-lj.si

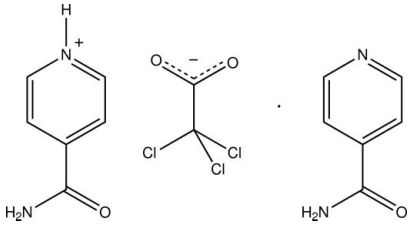
Received 9 August 2012; accepted 27 August 2012

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.040; $wR$ factor = 0.091; data-to-parameter ratio = 16.7.

In the crystal structure of the title 1:1 co-crystal, $C_6H_7N_2O^+\cdot C_2Cl_3O_2^-\cdot C_6H_6N_2O$, the amide groups of the 4-carbamoylpyridin-1-ium ion and the isonicotinamide molecule are twisted out of the plane of the aromatic ring with C—C—C—N torsion angles of 21.5 (4) and −33.5 (4)°, respectively. The 4-carbamoylpyridin-1-ium and isonicotinamide amide groups form $R_2^2(8)$ hydrogen-bonded dimers *via* N—H···O=C interactions. The two remaining amide H atoms (i) link dimers *via* the cation to an isonicotinamide and (ii) from the isonicotinamide to a trichloroacetate anion. The pyridinium H atom also forms an N—H···O hydrogen bond with the trichloroacetate anion. Due to the extended hydrogen bonding, including C—H···O and C—H···Cl interactions, all components in the structure aggregate into a three-dimensional supramolecular framework.

## Related literature

For applications of co-crystals, see: Karki *et al.* (2009); Friščić & Jones (2010). For related structures, see: Madeley *et al.* (2011).



## Experimental

### Crystal data

$C_6H_7N_2O^+\cdot C_2Cl_3O_2^-\cdot C_6H_6N_2O$
$M_r$ = 407.63
Orthorhombic, $Pna2_1$
$a$ = 13.7910 (3) Å
$b$ = 22.6680 (5) Å
$c$ = 5.6340 (1) Å
$V$ = 1761.27 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.55 mm$^{-1}$
$T$ = 293 K
0.4 × 0.1 × 0.1 mm

### Data collection

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min}$ = 0.811, $T_{max}$ = 0.947
16297 measured reflections
4017 independent reflections
3575 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.04
$wR(F^2)$ = 0.091
$S$ = 1.04
4017 reflections
241 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.57 e Å$^{-3}$
Absolute structure: Flack (1983), 1791 Friedel pairs
Flack parameter: 0.01 (6)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H15···O2$^{i}$ | 0.90 (3) | 1.78 (3) | 2.679 (3) | 175 (3) |
| N2—H16$A$···N3$^{ii}$ | 0.87 (3) | 2.11 (3) | 2.958 (3) | 164 (3) |
| N2—H16$B$···O3 | 0.90 (4) | 1.99 (4) | 2.887 (3) | 178 (3) |
| N4—H17$A$···O4 | 0.91 (4) | 2.08 (4) | 2.972 (3) | 167 (4) |
| N4—H17$B$···O1 | 0.89 (4) | 1.98 (4) | 2.839 (3) | 160 (3) |
| C1—H1···O1$^{i}$ | 0.93 | 2.58 | 3.211 (3) | 126 |
| C2—H2···O4$^{iii}$ | 0.93 | 2.55 | 3.358 (3) | 146 |
| C7—H7···O3$^{iv}$ | 0.93 | 2.58 | 3.489 (3) | 166 |
| C11—H11···Cl2$^{v}$ | 0.93 | 2.82 | 3.711 (3) | 162 |

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

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *publCIF* (Westrip, 2010).

The author thanks the Ministry of Education, Science, Culture and Sport of the Republic of Slovenia and the Slovenian Research Agency for financial support through grants P1–0230–0175 as well as the EN–FIST Centre of Excellence, Dunajska 156, 1000 Ljubljana, Slovenia for use of the Supernova diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2095).

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# supporting information

*Acta Cryst.* (2012). E**68**, o2818 [doi:10.1107/S1600536812037002]

## 4-Carbamoylpyridin-1-ium 2,2,2-trichloroacetate–isonicotinamide (1/1)

**Franc Perdih**

### S1. Comment

Co-crystals have attracted much attention in recent years owing to their contributions to crystal engineering and pharmaceutical chemistry. They were found to be useful in improving the stability, solubility, dissolution rate and mechanical properties (Karki *et al.*, 2009; Friščić & Jones, 2010). Here we present the structure obtained by reacting isonicotinamide and trichloroacetic acid in 2:1 molar ratio.

The asymmetric unit of (I) consists of one 4-carbamoylpyridin-1-ium cation, one trichloroacetate anion and one isonicotinamide molecule (Fig. 1). The amide groups of 4-carbamoylpyridin-1-ium ion and isonicotinamide molecule are twisted out of the plane of the aromatic ring with a C—C—C—N torsion angle of 21.5 (4)° and -33.5 (4)°, respectively. Similar twisting was observed for example in isonicotinamide–2-naphthoic acid (1/1) (Madeley *et al.*, 2011). Aromatic rings of 4-carbamoylpyridin-1-ium ion and isonicotinamide molecule are not coplanar, but are inclined by 35.05 (12)°. In the crystal, all the components of the structure are associated *via* the extended system of hydrogen bonds (N—H···O and N—H···N) and weak C—H···O and C—H···Cl interactions into extended three-dimensional supramolecular framework (Figs. 2, 3). The 4-carbamoylpyridin-1-ium ion is hydrogen bonded *via* N—H···O hydrogen bonding of the pyridinium unit to the trichloroacetate ion. The amide groups from 4-carbamoylpyridin-1-ium and isonicotinamide form a dimer *via* N—H···O hydrogen bonding, that is a typical supramolecular hydrogen-bonded synthon observed for amide-amide homodimers. Furthermore, the amide group of the cation is hydrogen bonded to the pyridine unit of isonicotinamide and the amide group of the isonicotinamide is hydrogen bonded to the trichloroacetate ion.

### S2. Experimental

Crystals of the title compound were obtained by slow evaporation of a 2:1 mol. mixture of isonicotinamide and trichloroacetic acid in methanol at room temperature.

### S3. Refinement

All H atoms were initially located in a difference Fourier maps. H atoms attached to N atoms were refined isotropically with $U_{iso}$(H) = 1.5$U_{eq}$(N). Other H atoms were treated as riding atoms in geometrically idealized positions, with C—H = 0.93 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(C).

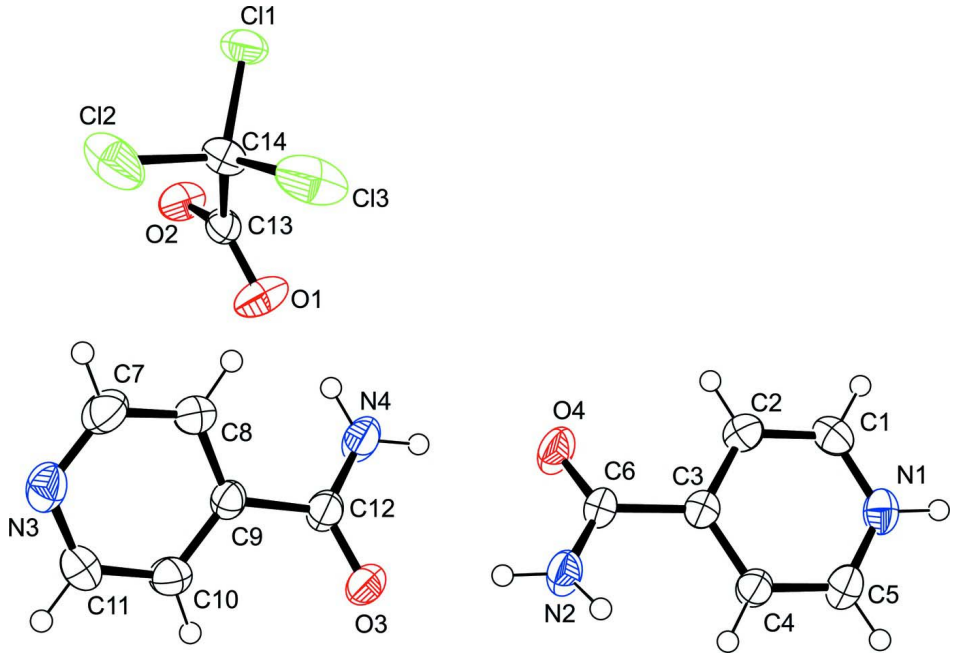


**Figure 1**

The asymmetric unit of the title compound with displacement ellipsoids drawn at the 50% probability level.

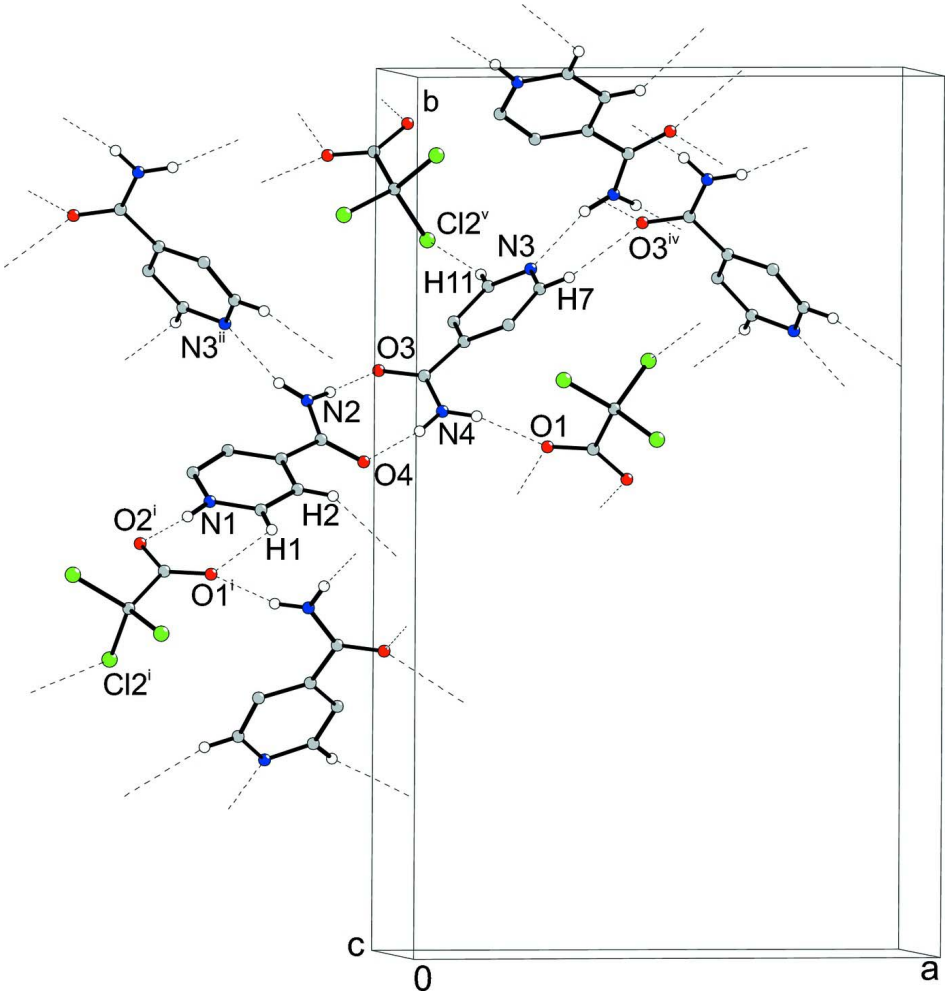


**Figure 2**

Hydrogen bonding diagram. Dashed lines indicate intermolecular N—H···O, N—H···N, C—H···O and C—H···Cl hydrogen bonding. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Symmetry codes: [i] –*x*, –*y* + 1, *z* + 3/2; [ii] *x* – 1/2, –*y* + 3/2, *z* + 1; [iv] *x* + 1/2, –*y* + 3/2, *z*; [v] *x* – 1/2, –*y* + 3/2, *z* – 1.

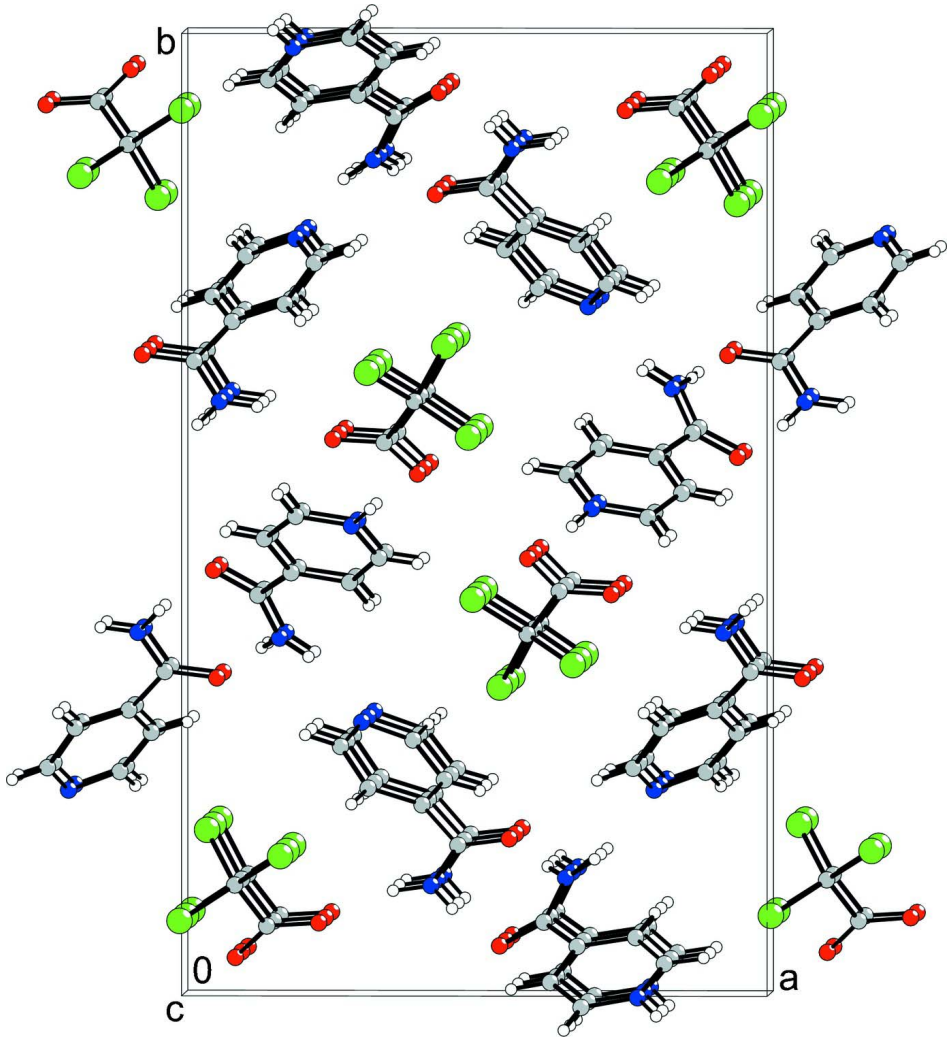


**Figure 3**

Crystal packing of the title compound. For the sake of clarity, hydrogen bonding is not presented.

### 4-Carbamoylpyridin-1-ium 2,2,2-trichloroacetate–isonicotinamide (1/1)

*Crystal data*

$C_6H_7N_2O^+·C_2Cl_3O_2^-·C_6H_6N_2O$
$M_r$ = 407.63
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a$ = 13.7910 (3) Å
$b$ = 22.6680 (5) Å
$c$ = 5.6340 (1) Å
$V$ = 1761.27 (6) Å$^3$
$Z$ = 4
$F(000)$ = 832
$D_x$ = 1.537 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 7898 reflections
$\theta$ = 3.1–30.4°
$\mu$ = 0.55 mm$^{-1}$
$T$ = 293 K
Prism, colourless
0.4 × 0.1 × 0.1 mm

*Data collection*

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4933 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min}$ = 0.811, $T_{max}$ = 0.947
16297 measured reflections
4017 independent reflections
3575 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 3.1°
$h$ = −17→17
$k$ = −29→29
$l$ = −7→7

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.04
$wR(F^2)$ = 0.091
$S$ = 1.03
4017 reflections
241 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.8943P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.57 e Å$^{-3}$
Absolute structure: Flack (1983), 1791 Friedel pairs
Absolute structure parameter: 0.01 (6)

*Special details*

**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.49348 (5) | 0.58322 (3) | 0.45563 (14) | 0.04547 (17) |
| Cl2 | 0.44990 (9) | 0.67585 (4) | 0.11741 (18) | 0.0850 (4) |
| Cl3 | 0.32105 (7) | 0.65069 (5) | 0.50880 (15) | 0.0770 (3) |
| N1 | −0.29717 (16) | 0.50785 (9) | 1.1681 (4) | 0.0377 (5) |
| H15 | −0.330 (2) | 0.4892 (15) | 1.285 (6) | 0.057* |
| N2 | −0.16743 (16) | 0.62843 (10) | 0.4929 (5) | 0.0396 (5) |
| H16A | −0.217 (2) | 0.6478 (14) | 0.551 (7) | 0.059* |
| H16B | −0.135 (3) | 0.6394 (15) | 0.362 (6) | 0.059* |
| N3 | 0.19060 (17) | 0.78523 (10) | −0.3220 (5) | 0.0425 (5) |
| N4 | 0.06680 (19) | 0.61873 (11) | 0.1998 (5) | 0.0495 (7) |
| H17A | 0.030 (3) | 0.5952 (17) | 0.294 (8) | 0.074* |
| H17B | 0.131 (3) | 0.6133 (15) | 0.197 (7) | 0.074* |
| O1 | 0.25905 (13) | 0.57929 (9) | 0.1146 (4) | 0.0524 (5) |
| O2 | 0.40234 (13) | 0.54301 (8) | 0.0115 (4) | 0.0426 (4) |

| | | | | |
|---|---|---|---|---|
| O3 | −0.06660 (12) | 0.66646 (9) | 0.0707 (4) | 0.0473 (5) |
| O4 | −0.05512 (13) | 0.55797 (8) | 0.5595 (4) | 0.0488 (5) |
| C1 | −0.20142 (19) | 0.49801 (11) | 1.1584 (5) | 0.0384 (6) |
| H1 | −0.1721 | 0.4742 | 1.2722 | 0.046* |
| C2 | −0.14636 (17) | 0.52287 (10) | 0.9812 (5) | 0.0356 (5) |
| H2 | −0.0801 | 0.5155 | 0.9729 | 0.043* |
| C3 | −0.19079 (17) | 0.55906 (10) | 0.8150 (4) | 0.0293 (5) |
| C4 | −0.29018 (18) | 0.56864 (11) | 0.8322 (5) | 0.0335 (5) |
| H4 | −0.3214 | 0.5929 | 0.7233 | 0.04* |
| C5 | −0.34193 (18) | 0.54211 (11) | 1.0107 (5) | 0.0387 (6) |
| H5 | −0.4085 | 0.5481 | 1.0217 | 0.046* |
| C6 | −0.13130 (17) | 0.58311 (10) | 0.6111 (5) | 0.0320 (5) |
| C7 | 0.2246 (2) | 0.76108 (12) | −0.1226 (6) | 0.0454 (7) |
| H7 | 0.2848 | 0.7735 | −0.0675 | 0.055* |
| C8 | 0.17586 (17) | 0.71890 (11) | 0.0064 (5) | 0.0396 (6) |
| H8 | 0.2034 | 0.7027 | 0.1424 | 0.048* |
| C9 | 0.08482 (16) | 0.70091 (10) | −0.0701 (5) | 0.0305 (5) |
| C10 | 0.0493 (2) | 0.72548 (12) | −0.2753 (5) | 0.0384 (6) |
| H10 | −0.0114 | 0.7145 | −0.3326 | 0.046* |
| C11 | 0.1043 (2) | 0.76648 (11) | −0.3955 (5) | 0.0433 (6) |
| H11 | 0.0797 | 0.7819 | −0.5361 | 0.052* |
| C12 | 0.02224 (18) | 0.65981 (11) | 0.0724 (5) | 0.0358 (6) |
| C13 | 0.34726 (17) | 0.57605 (10) | 0.1255 (4) | 0.0306 (5) |
| C14 | 0.40010 (19) | 0.61971 (11) | 0.2972 (5) | 0.0368 (6) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0384 (3) | 0.0586 (4) | 0.0395 (3) | 0.0069 (3) | −0.0118 (3) | 0.0020 (3) |
| Cl2 | 0.1224 (9) | 0.0644 (5) | 0.0681 (6) | −0.0526 (6) | −0.0447 (6) | 0.0302 (5) |
| Cl3 | 0.0848 (6) | 0.0950 (6) | 0.0510 (5) | 0.0508 (5) | −0.0198 (4) | −0.0362 (5) |
| N1 | 0.0414 (12) | 0.0353 (11) | 0.0365 (13) | −0.0078 (9) | 0.0084 (10) | 0.0028 (9) |
| N2 | 0.0375 (12) | 0.0379 (11) | 0.0435 (13) | 0.0082 (9) | 0.0147 (11) | 0.0077 (11) |
| N3 | 0.0423 (13) | 0.0351 (11) | 0.0503 (14) | −0.0051 (9) | 0.0080 (11) | 0.0057 (10) |
| N4 | 0.0317 (12) | 0.0490 (14) | 0.0678 (17) | 0.0069 (10) | 0.0141 (12) | 0.0256 (13) |
| O1 | 0.0277 (9) | 0.0589 (12) | 0.0706 (14) | 0.0023 (8) | −0.0027 (10) | −0.0192 (12) |
| O2 | 0.0346 (9) | 0.0506 (10) | 0.0426 (10) | 0.0042 (8) | −0.0017 (8) | −0.0179 (9) |
| O3 | 0.0278 (9) | 0.0557 (11) | 0.0585 (13) | 0.0039 (8) | 0.0068 (9) | 0.0201 (10) |
| O4 | 0.0367 (10) | 0.0514 (11) | 0.0585 (14) | 0.0153 (8) | 0.0200 (9) | 0.0148 (10) |
| C1 | 0.0444 (15) | 0.0377 (13) | 0.0332 (15) | 0.0002 (11) | −0.0051 (11) | 0.0055 (11) |
| C2 | 0.0307 (11) | 0.0348 (12) | 0.0413 (14) | 0.0014 (9) | 0.0001 (12) | 0.0002 (12) |
| C3 | 0.0297 (12) | 0.0279 (11) | 0.0304 (12) | −0.0023 (9) | 0.0021 (10) | −0.0034 (9) |
| C4 | 0.0300 (13) | 0.0340 (13) | 0.0364 (13) | 0.0012 (10) | 0.0031 (11) | 0.0044 (10) |
| C5 | 0.0347 (13) | 0.0381 (13) | 0.0435 (14) | −0.0010 (10) | 0.0099 (12) | 0.0013 (12) |
| C6 | 0.0295 (12) | 0.0343 (12) | 0.0321 (12) | −0.0013 (9) | 0.0071 (10) | −0.0003 (11) |
| C7 | 0.0327 (14) | 0.0460 (16) | 0.0577 (18) | −0.0081 (12) | −0.0007 (12) | 0.0027 (14) |
| C8 | 0.0317 (12) | 0.0450 (14) | 0.0422 (15) | 0.0006 (10) | −0.0029 (11) | 0.0061 (13) |
| C9 | 0.0288 (11) | 0.0289 (11) | 0.0337 (12) | 0.0018 (9) | 0.0050 (10) | 0.0020 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.0337 (13) | 0.0412 (14) | 0.0404 (14) | −0.0027 (11) | −0.0053 (11) | 0.0025 (12) |
| C11 | 0.0490 (15) | 0.0441 (14) | 0.0369 (14) | 0.0005 (12) | −0.0016 (13) | 0.0113 (13) |
| C12 | 0.0313 (13) | 0.0343 (12) | 0.0418 (15) | 0.0018 (10) | 0.0064 (11) | 0.0060 (11) |
| C13 | 0.0323 (12) | 0.0325 (11) | 0.0269 (11) | −0.0016 (9) | −0.0010 (10) | −0.0008 (10) |
| C14 | 0.0435 (15) | 0.0342 (14) | 0.0327 (12) | 0.0039 (11) | −0.0083 (11) | −0.0023 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C14 | 1.772 (3) | C1—H1 | 0.93 |
| Cl2—C14 | 1.765 (3) | C2—C3 | 1.387 (4) |
| Cl3—C14 | 1.762 (3) | C2—H2 | 0.93 |
| N1—C5 | 1.331 (4) | C3—C4 | 1.391 (3) |
| N1—C1 | 1.340 (3) | C3—C6 | 1.513 (3) |
| N1—H15 | 0.90 (3) | C4—C5 | 1.372 (4) |
| N2—C6 | 1.322 (3) | C4—H4 | 0.93 |
| N2—H16A | 0.87 (3) | C5—H5 | 0.93 |
| N2—H16B | 0.90 (4) | C7—C8 | 1.376 (4) |
| N3—C11 | 1.330 (4) | C7—H7 | 0.93 |
| N3—C7 | 1.335 (4) | C8—C9 | 1.389 (3) |
| N4—C12 | 1.326 (3) | C8—H8 | 0.93 |
| N4—H17A | 0.91 (4) | C9—C10 | 1.374 (4) |
| N4—H17B | 0.89 (4) | C9—C12 | 1.503 (3) |
| O1—C13 | 1.220 (3) | C10—C11 | 1.378 (4) |
| O2—C13 | 1.245 (3) | C10—H10 | 0.93 |
| O3—C12 | 1.234 (3) | C11—H11 | 0.93 |
| O4—C6 | 1.230 (3) | C13—C14 | 1.564 (3) |
| C1—C2 | 1.375 (4) | | |
| | | | |
| C5—N1—C1 | 121.8 (2) | N3—C7—C8 | 123.9 (3) |
| C5—N1—H15 | 122 (2) | N3—C7—H7 | 118 |
| C1—N1—H15 | 116 (2) | C8—C7—H7 | 118 |
| C6—N2—H16A | 120 (2) | C7—C8—C9 | 118.8 (3) |
| C6—N2—H16B | 116 (2) | C7—C8—H8 | 120.6 |
| H16A—N2—H16B | 124 (3) | C9—C8—H8 | 120.6 |
| C11—N3—C7 | 116.4 (2) | C10—C9—C8 | 117.7 (2) |
| C12—N4—H17A | 118 (2) | C10—C9—C12 | 119.7 (2) |
| C12—N4—H17B | 123 (2) | C8—C9—C12 | 122.4 (2) |
| H17A—N4—H17B | 119 (3) | C9—C10—C11 | 119.4 (2) |
| N1—C1—C2 | 120.3 (2) | C9—C10—H10 | 120.3 |
| N1—C1—H1 | 119.8 | C11—C10—H10 | 120.3 |
| C2—C1—H1 | 119.8 | N3—C11—C10 | 123.7 (3) |
| C1—C2—C3 | 119.2 (2) | N3—C11—H11 | 118.1 |
| C1—C2—H2 | 120.4 | C10—C11—H11 | 118.1 |
| C3—C2—H2 | 120.4 | O3—C12—N4 | 123.4 (2) |
| C2—C3—C4 | 118.7 (2) | O3—C12—C9 | 119.3 (2) |
| C2—C3—C6 | 119.1 (2) | N4—C12—C9 | 117.3 (2) |
| C4—C3—C6 | 122.1 (2) | O1—C13—O2 | 128.2 (2) |
| C5—C4—C3 | 119.7 (2) | O1—C13—C14 | 117.2 (2) |

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|---|---|---|---|
| C5—C4—H4 | 120.2 | O2—C13—C14 | 114.6 (2) |
| C3—C4—H4 | 120.2 | C13—C14—Cl3 | 112.49 (18) |
| N1—C5—C4 | 120.2 (2) | C13—C14—Cl2 | 106.43 (18) |
| N1—C5—H5 | 119.9 | Cl3—C14—Cl2 | 109.97 (15) |
| C4—C5—H5 | 119.9 | C13—C14—Cl1 | 110.78 (17) |
| O4—C6—N2 | 124.3 (2) | Cl3—C14—Cl1 | 107.15 (15) |
| O4—C6—C3 | 118.4 (2) | Cl2—C14—Cl1 | 110.04 (15) |
| N2—C6—C3 | 117.3 (2) | | |

| | | | |
|---|---|---|---|
| C5—N1—C1—C2 | −0.8 (4) | C7—C8—C9—C12 | −173.6 (2) |
| N1—C1—C2—C3 | 1.1 (4) | C8—C9—C10—C11 | −0.1 (4) |
| C1—C2—C3—C4 | −0.5 (4) | C12—C9—C10—C11 | 175.2 (2) |
| C1—C2—C3—C6 | −176.0 (2) | C7—N3—C11—C10 | 1.5 (4) |
| C2—C3—C4—C5 | −0.4 (4) | C9—C10—C11—N3 | −1.5 (4) |
| C6—C3—C4—C5 | 175.0 (2) | C10—C9—C12—O3 | −30.3 (4) |
| C1—N1—C5—C4 | −0.1 (4) | C8—C9—C12—O3 | 144.8 (3) |
| C3—C4—C5—N1 | 0.7 (4) | C10—C9—C12—N4 | 151.4 (3) |
| C2—C3—C6—O4 | 20.0 (4) | C8—C9—C12—N4 | −33.5 (4) |
| C4—C3—C6—O4 | −155.4 (3) | O1—C13—C14—Cl3 | 17.9 (3) |
| C2—C3—C6—N2 | −163.1 (2) | O2—C13—C14—Cl3 | −163.93 (19) |
| C4—C3—C6—N2 | 21.5 (4) | O1—C13—C14—Cl2 | −102.6 (3) |
| C11—N3—C7—C8 | 0.1 (4) | O2—C13—C14—Cl2 | 75.6 (2) |
| N3—C7—C8—C9 | −1.6 (4) | O1—C13—C14—Cl1 | 137.8 (2) |
| C7—C8—C9—C10 | 1.5 (4) | O2—C13—C14—Cl1 | −44.0 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H15···O2$^{i}$ | 0.90 (3) | 1.78 (3) | 2.679 (3) | 175 (3) |
| N2—H16*A*···N3$^{ii}$ | 0.87 (3) | 2.11 (3) | 2.958 (3) | 164 (3) |
| N2—H16*B*···O3 | 0.90 (4) | 1.99 (4) | 2.887 (3) | 178 (3) |
| N4—H17*A*···O4 | 0.91 (4) | 2.08 (4) | 2.972 (3) | 167 (4) |
| N4—H17*B*···O1 | 0.89 (4) | 1.98 (4) | 2.839 (3) | 160 (3) |
| C1—H1···O1$^{i}$ | 0.93 | 2.58 | 3.211 (3) | 126 |
| C2—H2···O4$^{iii}$ | 0.93 | 2.55 | 3.358 (3) | 146 |
| C7—H7···O3$^{iv}$ | 0.93 | 2.58 | 3.489 (3) | 166 |
| C11—H11···Cl2$^{v}$ | 0.93 | 2.82 | 3.711 (3) | 162 |

Symmetry codes: (i) −*x*, −*y*+1, *z*+3/2; (ii) *x*−1/2, −*y*+3/2, *z*+1; (iii) −*x*, −*y*+1, *z*+1/2; (iv) *x*+1/2, −*y*+3/2, *z*; (v) *x*−1/2, −*y*+3/2, *z*−1.