

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

Bis[(4-cyanobenzyl)ammonium]  
bis(perchlorate) monohydrate

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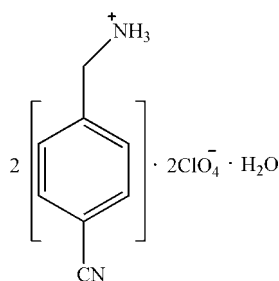
Received 14 October 2011; accepted 16 November 2011

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å; disorder in main residue;  $R$  factor = 0.066;  $wR$  factor = 0.179; data-to-parameter ratio = 16.8.

The asymmetric unit of the title compound,  $2\text{C}_8\text{H}_9\text{N}_2^+ \cdot 2\text{ClO}_4^- \cdot \text{H}_2\text{O}$ , consists of two (4-cyanobenzyl)ammonium cations, two disordered  $\text{ClO}_4^-$  anions and one water molecule. The differences in the two cations are reflected in the  $\text{N}-\text{C}-\text{C}-\text{C}$  torsion angles [ $-94.7$  (3) and  $-115.9$  (3)°]. In addition, the cations show different hydrogen-bonding patterns as one N atom bonds to two O atoms of  $\text{ClO}_4^-$  ions, while the other N atom is involved in hydrogen bonding with the O atoms of the  $\text{ClO}_4^-$  ions and water molecules. In the crystal,  $\text{N}-\text{H} \cdots \text{O}$  and  $\text{O}-\text{H} \cdots \text{O}$  hydrogen bonds result in a three-dimensional network. An O atom in each of the anions is disordered over two positions of equal occupancy.

## Related literature

For a related structure, see: Shahwar *et al.* (2009).



## Experimental

## Crystal data

$2\text{C}_8\text{H}_9\text{N}_2^+ \cdot 2\text{ClO}_4^- \cdot \text{H}_2\text{O}$   
 $M_r = 483.26$   
Triclinic,  $P\bar{1}$   
 $a = 5.0305$  (10) Å

$b = 13.616$  (3) Å  
 $c = 16.695$  (3) Å  
 $\alpha = 66.79$  (3)°  
 $\beta = 86.17$  (3)°

$\gamma = 88.97$  (3)°  
 $V = 1048.6$  (4) Å<sup>3</sup>  
 $Z = 2$   
Mo  $K\alpha$  radiation

$\mu = 0.37$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.29 \times 0.25 \times 0.21$  mm

## Data collection

Rigaku SCXmini diffractometer  
Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2005)  
 $T_{\min} = 0.8$ ,  $T_{\max} = 0.9$

10950 measured reflections  
4796 independent reflections  
3490 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.038$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$   
 $wR(F^2) = 0.179$   
 $S = 1.00$   
4796 reflections  
285 parameters  
62 restraints

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.70$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.70$  e Å<sup>-3</sup>

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

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N2}-\text{H2C} \cdots \text{O1}^i$	0.89	2.32	2.77 (3)	111
$\text{N2}-\text{H2C} \cdots \text{O2}^{ii}$	0.89	2.34	3.015 (4)	133
$\text{N2}-\text{H2C} \cdots \text{O1}^i$	0.89	2.42	2.96 (3)	120
$\text{N2}-\text{H2C} \cdots \text{O5}^{ii}$	0.89	2.46	2.905 (9)	112
$\text{N2}-\text{H2C} \cdots \text{O5}^i$	0.89	2.55	3.036 (9)	115
$\text{N2}-\text{H2E} \cdots \text{O7}^{iii}$	0.89	2.43	3.306 (5)	167
$\text{N2}-\text{H2E} \cdots \text{O5}^{iii}$	0.89	2.45	3.176 (8)	139
$\text{N4}-\text{H4C} \cdots \text{O1W}^{iv}$	0.89	1.96	2.845 (4)	177
$\text{N4}-\text{H4D} \cdots \text{O3}^{iv}$	0.89	2.15	3.002 (5)	159
$\text{O1W}-\text{H1WA} \cdots \text{O6}^v$	0.82 (1)	2.02 (1)	2.839 (5)	173 (5)
$\text{O1W}-\text{H1WB} \cdots \text{O7}^{ii}$	0.82 (1)	2.21 (2)	2.991 (4)	160 (4)
$\text{O1W}-\text{H1WB} \cdots \text{Cl2}^{ii}$	0.82 (1)	2.98 (3)	3.593 (3)	133 (4)
$\text{N2}-\text{H2D} \cdots \text{O2}$	0.89	2.25	3.096 (5)	158
$\text{N2}-\text{H2D} \cdots \text{O3}$	0.89	2.40	3.154 (5)	143
$\text{N4}-\text{H4B} \cdots \text{O1W}$	0.89	2.25	3.011 (4)	143
$\text{N4}-\text{H4B} \cdots \text{O1}^i$	0.89	2.50	3.04 (3)	119
$\text{N4}-\text{H4B} \cdots \text{O5}^i$	0.89	2.66	3.225 (8)	123
$\text{N4}-\text{H4D} \cdots \text{O4}$	0.89	2.63	3.202 (5)	123

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXL97*.

This work was supported by a start-up grant from the Yunnan Radio and TV University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2463).

## References

- Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.  
Shahwar, D., Tahir, M. N., Ahmad, N., Khan, M. A. & Yasmeen, A. (2009). *Acta Cryst.* **E65**, o1312.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

**supplementary materials**

*Acta Cryst.* (2011). E67, o3431 [ doi:10.1107/S1600536811048884 ]

## Bis[4-(4-cyanobenzyl)ammonium] bis(perchlorate) monohydrate

Y. Huang

### Comment

Recently, the crystal structure phenylmethanaminium chloroacetate (Shahwar *et al.*, 2009) has been reported. In our laboratory, a compound containing protonated 4-(aminomethyl)benzotrile,  $\text{ClO}_4^-$  anions and water molecule has been synthesized. In this paper, we report the crystal structure of the title compound.

The asymmetric unit of the title compound, consists of two independent 4-(aminomethyl)benzotrile cations, two disorder  $\text{ClO}_4^-$  anions and a water molecule. The dihedral angle between the 4-(aminomethyl)benzotrile planes in the two cations is  $7.25(9)^\circ$ . The atoms N2 and N4 are displaced out of these planes (C9–C16/N1 and C1–C8/N3) by 1.93 (3) and 1.325 (3) Å, respectively, with torsion angles C9/C12—C14/N2 and C6/C5—C2/N4 being  $-94.7(3)$  and  $-115.9(3)^\circ$ , respectively.

The cations show different hydrogen bonding patterns as N2 atom bonds to two O atoms of  $\text{ClO}_4^-$  ions, while N2 atom is involved in hydrogen bonding with the O atoms of the  $\text{ClO}_4^-$  ions and  $\text{H}_2\text{O}$  of hydration. In the crystal structure, intermolecular hydrogen bonds of the types N—H $\cdots$ O and O—H $\cdots$ O result in a three-dimensional network thus stabilizing the structure. (Tab. 1 & Fig. 2).

### Experimental

4-(Aminomethyl)benzotrile (20 mmol, 2.64 g) and 10% aqueous  $\text{HClO}_4$  in a molar ratio of 1:1 were mixed and dissolved in 50 ml water by heating to 363 K forming a clear solution. The reaction mixture was cooled slowly to room temperature, block crystals of the title compound were formed after five days.

### Refinement

The H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å and N—H = 0.89 Å, and refined using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{N})$ . The H-atoms of water molecule were located from a difference Fourier map and were allowed to refine with O—H = 0.82 (1) Å constrains. For the disordered  $\text{ClO}_4^-$ , SIMU and DELU commands in SHELXL-97 (Sheldrick, 2008) were used to restrict their  $U_{\text{eq}}$ .

## Figures

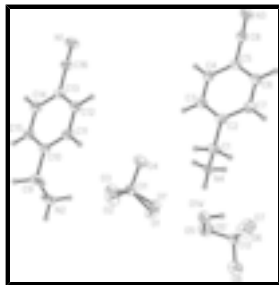


Fig. 1. The asymmetric unit of the title compound with atom labels. Displacement ellipsoids were drawn at the 30% probability level. The oxygen atoms of the anions O1/O1' and O5/O5' were disordered in a 1:1 ratio.

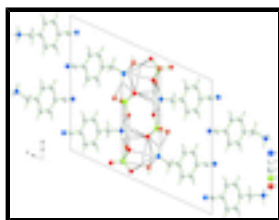
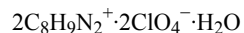


Fig. 2. The packing viewed along the *a*-axis. Hydrogen bonds are drawn as dashed lines

## Bis[(4-cyanobenzyl)ammonium] bis(perchlorate) monohydrate

### Crystal data



$M_r = 483.26$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 5.0305 (10) \text{ \AA}$

$b = 13.616 (3) \text{ \AA}$

$c = 16.695 (3) \text{ \AA}$

$\alpha = 66.79 (3)^\circ$

$\beta = 86.17 (3)^\circ$

$\gamma = 88.97 (3)^\circ$

$V = 1048.6 (4) \text{ \AA}^3$

$Z = 2$

$F(000) = 500$

$D_x = 1.531 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3490 reflections

$\theta = 3.3\text{--}27.5^\circ$

$\mu = 0.37 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colorless

$0.29 \times 0.25 \times 0.21 \text{ mm}$

### Data collection

Rigaku SCXmini  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

Detector resolution:  $13.6612 \text{ pixels mm}^{-1}$

$\omega$  scans

Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2005)

$T_{\min} = 0.8$ ,  $T_{\max} = 0.9$

10950 measured reflections

4796 independent reflections

3490 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 3.3^\circ$

$h = -6 \rightarrow 6$

$k = -17 \rightarrow 17$

$l = -21 \rightarrow 21$

Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.066$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.179$	$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 1.794P]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
4796 reflections	$(\Delta/\sigma)_{\max} = 0.001$
285 parameters	$\Delta\rho_{\max} = 0.70 \text{ e } \text{\AA}^{-3}$
62 restraints	$\Delta\rho_{\min} = -0.70 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kF_c[1+0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.047 (4)

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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.5034 (8)	0.8947 (3)	-0.0455 (3)	0.0622 (10)	
N2	1.2660 (6)	0.6834 (3)	0.36264 (19)	0.0446 (7)	
H2C	1.3892	0.6573	0.4013	0.067*	
H2D	1.1463	0.6327	0.3702	0.067*	
H2E	1.1855	0.7382	0.3702	0.067*	
C11	1.0956 (8)	0.6906 (3)	0.1705 (2)	0.0445 (9)	
H11A	1.1534	0.6202	0.1906	0.053*	
C15	1.1073 (8)	0.8638 (3)	0.1743 (2)	0.0431 (8)	
H15A	1.1737	0.9104	0.1966	0.052*	
C13	0.8291 (7)	0.8294 (3)	0.0775 (2)	0.0367 (7)	
C10	1.1925 (7)	0.7590 (3)	0.2048 (2)	0.0363 (7)	
C14	0.9249 (8)	0.8999 (3)	0.1109 (2)	0.0441 (8)	
H14A	0.8668	0.9702	0.0909	0.053*	
C9	1.3941 (7)	0.7202 (3)	0.2730 (2)	0.0474 (9)	
H9A	1.4938	0.6618	0.2666	0.057*	

## supplementary materials

H9B	1.5188	0.7777	0.2641	0.057*	
C16	0.6445 (8)	0.8667 (3)	0.0090 (2)	0.0444 (8)	
C12	0.9149 (8)	0.7251 (3)	0.1071 (2)	0.0443 (8)	
H12A	0.8510	0.6785	0.0844	0.053*	
N3	-0.4792 (8)	0.3873 (3)	-0.0191 (2)	0.0595 (9)	
N4	0.3279 (6)	0.2507 (3)	0.37842 (19)	0.0423 (7)	
H4B	0.4520	0.2284	0.4171	0.063*	
H4C	0.1783	0.2134	0.4011	0.063*	
H4D	0.2968	0.3197	0.3653	0.063*	
C3	0.1437 (8)	0.3734 (3)	0.1900 (2)	0.0444 (8)	
H3A	0.2187	0.4246	0.2058	0.053*	
C1	0.4220 (7)	0.2352 (3)	0.2980 (2)	0.0459 (9)	
H1A	0.4646	0.1605	0.3133	0.055*	
H1B	0.5839	0.2767	0.2733	0.055*	
C8	-0.3365 (8)	0.3600 (3)	0.0352 (2)	0.0433 (8)	
C6	-0.0743 (8)	0.2218 (3)	0.1421 (3)	0.0458 (9)	
H6A	-0.1469	0.1706	0.1258	0.055*	
C2	0.2180 (7)	0.2681 (3)	0.2300 (2)	0.0371 (7)	
C7	0.1075 (8)	0.1923 (3)	0.2058 (3)	0.0464 (9)	
H7A	0.1560	0.1210	0.2326	0.056*	
C4	-0.0396 (8)	0.4042 (3)	0.1271 (2)	0.0438 (8)	
H4A	-0.0901	0.4753	0.1013	0.053*	
C5	-0.1485 (7)	0.3278 (3)	0.1025 (2)	0.0375 (7)	
C11	0.81260 (16)	0.45679 (7)	0.38569 (6)	0.0390 (2)	
O1	0.771 (5)	0.356 (3)	0.4610 (17)	0.068 (5)	0.50
O1'	0.709 (6)	0.363 (3)	0.4491 (18)	0.073 (5)	0.50
O2	0.7525 (6)	0.5448 (3)	0.4098 (2)	0.0673 (9)	
O3	1.0947 (6)	0.4613 (3)	0.3634 (2)	0.0665 (9)	
O4	0.6749 (6)	0.4664 (3)	0.3112 (2)	0.0647 (8)	
C12	0.23525 (17)	0.09462 (7)	0.62797 (6)	0.0409 (2)	
O6	0.3449 (6)	0.0479 (3)	0.5708 (2)	0.0705 (5)	
O7	-0.0484 (6)	0.0887 (3)	0.6317 (2)	0.0705 (5)	
O8	0.3295 (6)	0.0431 (3)	0.7120 (2)	0.0705 (5)	
O5	0.2648 (14)	0.2074 (7)	0.6001 (5)	0.0705 (5)	0.50
O5'	0.3568 (14)	0.1992 (7)	0.5835 (5)	0.0705 (5)	0.50
O1W	0.8375 (6)	0.1387 (3)	0.4461 (2)	0.0520 (7)	
H1WA	0.799 (10)	0.083 (2)	0.442 (3)	0.075 (17)*	
H1WB	0.827 (9)	0.128 (4)	0.4981 (8)	0.059 (14)*	

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.070 (2)	0.057 (2)	0.063 (2)	0.0166 (18)	-0.035 (2)	-0.0242 (18)
N2	0.0431 (17)	0.0548 (19)	0.0367 (16)	-0.0024 (14)	-0.0109 (13)	-0.0175 (14)
C11	0.053 (2)	0.0374 (19)	0.044 (2)	0.0122 (16)	-0.0147 (17)	-0.0160 (16)
C15	0.050 (2)	0.0426 (19)	0.0417 (19)	0.0012 (16)	-0.0097 (16)	-0.0209 (16)
C13	0.0365 (17)	0.0427 (19)	0.0316 (17)	0.0051 (14)	-0.0069 (13)	-0.0150 (14)
C10	0.0326 (17)	0.0454 (19)	0.0301 (16)	0.0022 (14)	-0.0032 (13)	-0.0138 (14)

C14	0.052 (2)	0.0365 (19)	0.045 (2)	0.0070 (16)	-0.0091 (16)	-0.0166 (16)
C9	0.0368 (19)	0.067 (3)	0.0360 (19)	0.0068 (17)	-0.0091 (15)	-0.0168 (18)
C16	0.047 (2)	0.043 (2)	0.044 (2)	0.0073 (16)	-0.0117 (17)	-0.0164 (16)
C12	0.054 (2)	0.0408 (19)	0.043 (2)	0.0017 (16)	-0.0142 (17)	-0.0193 (16)
N3	0.069 (2)	0.060 (2)	0.057 (2)	0.0119 (18)	-0.0292 (19)	-0.0274 (18)
N4	0.0353 (15)	0.0513 (18)	0.0396 (16)	-0.0022 (13)	-0.0093 (12)	-0.0159 (14)
C3	0.050 (2)	0.0411 (19)	0.046 (2)	-0.0003 (16)	-0.0115 (16)	-0.0202 (16)
C1	0.0342 (18)	0.060 (2)	0.044 (2)	0.0085 (16)	-0.0076 (15)	-0.0213 (18)
C8	0.047 (2)	0.045 (2)	0.042 (2)	0.0020 (16)	-0.0067 (17)	-0.0199 (17)
C6	0.050 (2)	0.043 (2)	0.051 (2)	-0.0010 (16)	-0.0124 (17)	-0.0240 (17)
C2	0.0331 (17)	0.047 (2)	0.0338 (17)	0.0030 (14)	-0.0021 (13)	-0.0184 (15)
C7	0.053 (2)	0.0351 (19)	0.051 (2)	0.0074 (16)	-0.0103 (17)	-0.0154 (16)
C4	0.051 (2)	0.0361 (18)	0.044 (2)	0.0040 (16)	-0.0105 (16)	-0.0151 (16)
C5	0.0357 (18)	0.0453 (19)	0.0314 (16)	0.0024 (14)	-0.0042 (13)	-0.0149 (15)
C11	0.0360 (4)	0.0405 (5)	0.0415 (5)	0.0010 (3)	-0.0033 (3)	-0.0172 (4)
O1	0.090 (12)	0.056 (5)	0.042 (8)	-0.005 (7)	-0.005 (8)	-0.004 (6)
O1'	0.100 (12)	0.061 (8)	0.043 (6)	-0.033 (8)	-0.028 (6)	0.000 (5)
O2	0.0622 (19)	0.066 (2)	0.094 (2)	0.0170 (15)	-0.0153 (17)	-0.0525 (19)
O3	0.0370 (15)	0.077 (2)	0.095 (2)	0.0056 (14)	-0.0030 (15)	-0.0434 (19)
O4	0.0577 (18)	0.087 (2)	0.0553 (18)	-0.0067 (16)	-0.0151 (14)	-0.0324 (17)
C12	0.0403 (5)	0.0452 (5)	0.0388 (5)	-0.0029 (4)	-0.0029 (3)	-0.0181 (4)
O6	0.0604 (11)	0.0881 (12)	0.0651 (11)	0.0018 (9)	-0.0026 (8)	-0.0325 (9)
O7	0.0604 (11)	0.0881 (12)	0.0651 (11)	0.0018 (9)	-0.0026 (8)	-0.0325 (9)
O8	0.0604 (11)	0.0881 (12)	0.0651 (11)	0.0018 (9)	-0.0026 (8)	-0.0325 (9)
O5	0.0604 (11)	0.0881 (12)	0.0651 (11)	0.0018 (9)	-0.0026 (8)	-0.0325 (9)
O5'	0.0604 (11)	0.0881 (12)	0.0651 (11)	0.0018 (9)	-0.0026 (8)	-0.0325 (9)
O1W	0.0477 (16)	0.0612 (19)	0.0485 (17)	-0.0074 (14)	-0.0066 (13)	-0.0222 (15)

*Geometric parameters (Å, °)*

N1—C16	1.132 (5)	C3—H3A	0.9300
N2—C9	1.482 (5)	C1—C2	1.510 (5)
N2—H2C	0.8900	C1—H1A	0.9700
N2—H2D	0.8900	C1—H1B	0.9700
N2—H2E	0.8900	C8—C5	1.444 (5)
C11—C12	1.374 (5)	C6—C7	1.382 (5)
C11—C10	1.380 (5)	C6—C5	1.386 (5)
C11—H11A	0.9300	C6—H6A	0.9300
C15—C14	1.381 (5)	C2—C7	1.384 (5)
C15—C10	1.384 (5)	C7—H7A	0.9300
C15—H15A	0.9300	C4—C5	1.392 (5)
C13—C12	1.379 (5)	C4—H4A	0.9300
C13—C14	1.393 (5)	C11—O1'	1.38 (3)
C13—C16	1.447 (5)	C11—O4	1.423 (3)
C10—C9	1.506 (5)	C11—O2	1.428 (3)
C14—H14A	0.9300	C11—O3	1.439 (3)
C9—H9A	0.9700	C11—O1	1.45 (3)
C9—H9B	0.9700	C12—O8	1.410 (3)
C12—H12A	0.9300	C12—O6	1.420 (3)

## supplementary materials

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N3—C8	1.135 (5)	C12—O5	1.426 (9)
N4—C1	1.486 (5)	C12—O7	1.426 (3)
N4—H4B	0.8900	C12—O5'	1.445 (9)
N4—H4C	0.8900	O5—O5'	0.557 (8)
N4—H4D	0.8900	O1W—H1WA	0.820 (2)
C3—C4	1.377 (5)	O1W—H1WB	0.820 (2)
C3—C2	1.379 (5)		
C9—N2—H2C	109.5	N4—C1—H1B	109.1
C9—N2—H2D	109.5	C2—C1—H1B	109.1
H2C—N2—H2D	109.5	H1A—C1—H1B	107.8
C9—N2—H2E	109.5	N3—C8—C5	178.1 (5)
H2C—N2—H2E	109.5	C7—C6—C5	119.9 (3)
H2D—N2—H2E	109.5	C7—C6—H6A	120.0
C12—C11—C10	120.8 (3)	C5—C6—H6A	120.0
C12—C11—H11A	119.6	C3—C2—C7	119.3 (3)
C10—C11—H11A	119.6	C3—C2—C1	120.8 (3)
C14—C15—C10	120.6 (3)	C7—C2—C1	119.9 (3)
C14—C15—H15A	119.7	C6—C7—C2	120.4 (3)
C10—C15—H15A	119.7	C6—C7—H7A	119.8
C12—C13—C14	120.5 (3)	C2—C7—H7A	119.8
C12—C13—C16	119.6 (3)	C3—C4—C5	119.4 (3)
C14—C13—C16	119.8 (3)	C3—C4—H4A	120.3
C11—C10—C15	119.4 (3)	C5—C4—H4A	120.3
C11—C10—C9	120.1 (3)	C6—C5—C4	119.8 (3)
C15—C10—C9	120.5 (3)	C6—C5—C8	120.5 (3)
C15—C14—C13	119.0 (3)	C4—C5—C8	119.6 (3)
C15—C14—H14A	120.5	O1'—C11—O4	102.7 (9)
C13—C14—H14A	120.5	O1'—C11—O2	110.4 (14)
N2—C9—C10	111.8 (3)	O4—C11—O2	110.4 (2)
N2—C9—H9A	109.3	O1'—C11—O3	117.5 (13)
C10—C9—H9A	109.3	O4—C11—O3	108.8 (2)
N2—C9—H9B	109.3	O2—C11—O3	106.82 (19)
C10—C9—H9B	109.3	O1'—C11—O1	14.8 (18)
H9A—C9—H9B	107.9	O4—C11—O1	115.4 (10)
N1—C16—C13	178.5 (4)	O2—C11—O1	110.3 (11)
C11—C12—C13	119.6 (3)	O3—C11—O1	104.6 (11)
C11—C12—H12A	120.2	O8—C12—O6	110.8 (2)
C13—C12—H12A	120.2	O8—C12—O5	109.5 (4)
C1—N4—H4B	109.5	O6—C12—O5	117.2 (3)
C1—N4—H4C	109.5	O8—C12—O7	110.7 (2)
H4B—N4—H4C	109.5	O6—C12—O7	109.4 (2)
C1—N4—H4D	109.5	O5—C12—O7	98.7 (3)
H4B—N4—H4D	109.5	O8—C12—O5'	110.7 (4)
H4C—N4—H4D	109.5	O6—C12—O5'	97.5 (3)
C4—C3—C2	121.1 (3)	O5—C12—O5'	22.3 (3)
C4—C3—H3A	119.4	O7—C12—O5'	117.1 (3)
C2—C3—H3A	119.4	O5'—O5—C12	80.8 (15)
N4—C1—C2	112.7 (3)	O5—O5'—C12	76.9 (15)
N4—C1—H1A	109.1	H1WA—O1W—H1WB	108 (5)



C2—C1—H1A

109.1

*Hydrogen-bond geometry* (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2C...O1 <sup>i</sup>	0.89	2.32	2.77 (3)	111.
N2—H2C...O2 <sup>ii</sup>	0.89	2.34	3.015 (4)	133.
N2—H2C...O1 <sup>i</sup>	0.89	2.42	2.96 (3)	120.
N2—H2C...O5 <sup>i</sup>	0.89	2.46	2.905 (9)	112.
N2—H2C...O5 <sup>i</sup>	0.89	2.55	3.036 (9)	115.
N2—H2E...O7 <sup>iii</sup>	0.89	2.43	3.306 (5)	167.
N2—H2E...O5 <sup>iii</sup>	0.89	2.45	3.176 (8)	139.
N4—H4C...O1W <sup>iv</sup>	0.89	1.96	2.845 (4)	177.
N4—H4D...O3 <sup>iv</sup>	0.89	2.15	3.002 (5)	159.
O1W—H1WA...O6 <sup>v</sup>	0.82 (1)	2.02 (1)	2.839 (5)	173 (5)
O1W—H1WB...O7 <sup>ii</sup>	0.82 (1)	2.21 (2)	2.991 (4)	160 (4)
O1W—H1WB...Cl2 <sup>ii</sup>	0.82 (1)	2.98 (3)	3.593 (3)	133 (4)
N2—H2D...O2	0.89	2.25	3.096 (5)	158.
N2—H2D...O3	0.89	2.40	3.154 (5)	143.
N4—H4B...O1W	0.89	2.25	3.011 (4)	143.
N4—H4B...O1'	0.89	2.50	3.04 (3)	119.
N4—H4B...O5'	0.89	2.66	3.225 (8)	123.
N4—H4D...O4	0.89	2.63	3.202 (5)	123.

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

Fig. 1

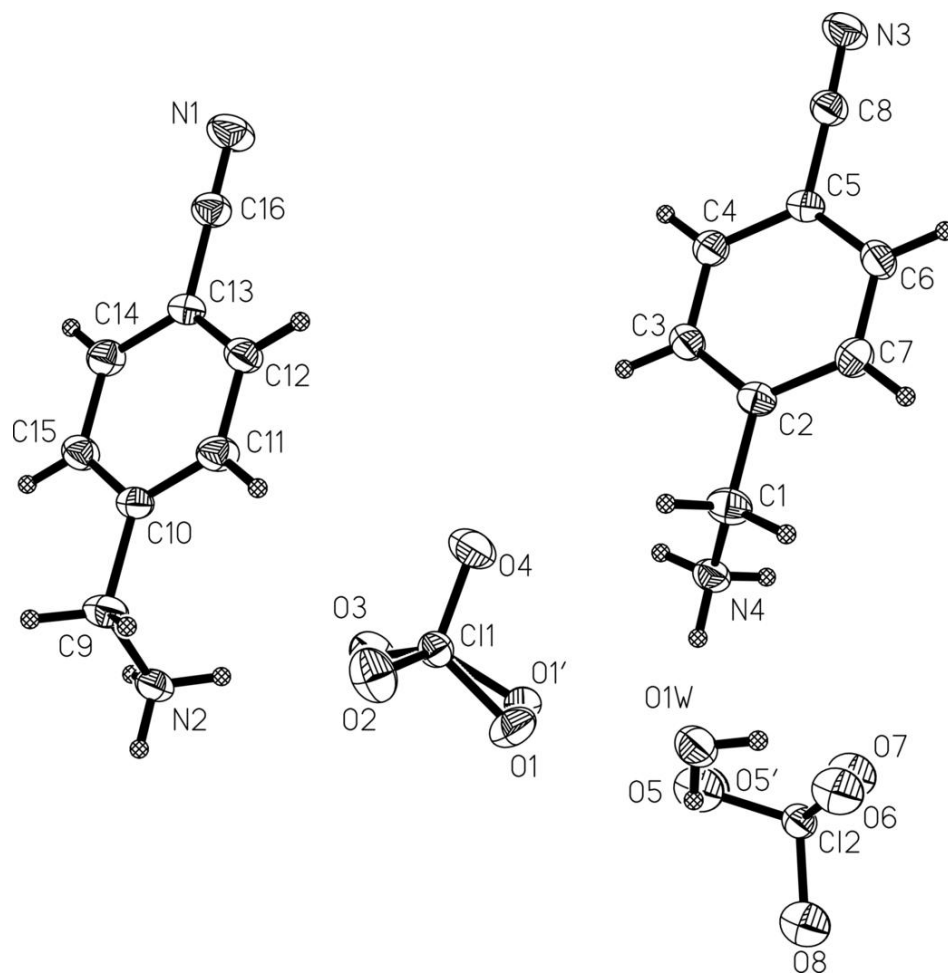


Fig. 2

