Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Benzimidazolium perchlorate

Lesław Sieroń

Institute of General and Ecological Chemistry, Technical University of Łódź, Żeromskiego 116, 90-924 Łódź, Poland
Correspondence e-mail: lsieron@p.lodz.pl

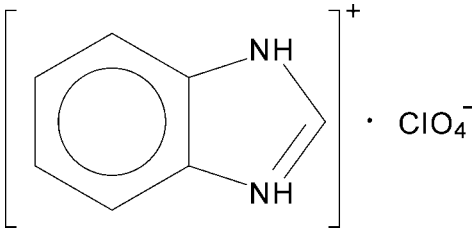
Received 12 July 2007; accepted 19 July 2007

Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.005 Å; R factor = 0.041; wR factor = 0.112; data-to-parameter ratio = 11.6.

The asymmetric unit of the title salt, $C_7H_7N_2^+\cdot ClO_4^-$, consists of two benzimidazolium cations and two perchlorate anions linked by N—H···O hydrogen bonds into sheets of alternating edge-fused $R_4^3(12)$ rings, which run parallel to the (102) plane.

Related literature

For related structures, see: Sieroń (2005*a*,*b*, 2007*a*,*b*,*c*). For related literature, see: Etter *et al.* (1990); Jeffrey & Saenger (1991).



Experimental

Crystal data

$C_7H_7N_2^+\cdot ClO_4^-$
M_r = 218.60
Orthorhombic, $Pca2_1$
a = 9.9885 (2) Å
b = 9.0509 (1) Å
c = 19.0182 (3) Å
V = 1719.34 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.43 mm^{-1}
T = 297 K
0.60 × 0.30 × 0.06 mm

Data collection

Kuma KM4 CCD area-detector diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.781, T_{max} = 0.974
16719 measured reflections
2923 independent reflections
2676 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.112
S = 1.06
2923 reflections
253 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.87 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Absolute structure: Flack (1983), with 1462 Friedel pairs
Flack parameter: 0.04 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	2.38	3.007 (5)	130
N1—H1···O12^{i}	0.86	2.26	3.022 (4)	148
N3—H3···O13	0.86	2.05	2.863 (4)	157
N11—H11···O11^{ii}	0.86	2.38	3.011 (5)	130
N11—H11···O2^{ii}	0.86	2.23	3.011 (4)	151
N13—H13···O3^{iii}	0.86	2.06	2.866 (4)	155
C2—H2···O2	0.93	2.47	3.349 (4)	159
C2—H2···O11	0.93	2.59	3.279 (4)	131
C7—H7···O13^{i}	0.93	2.51	3.413 (5)	164
C12—H12···O12^{ii}	0.93	2.49	3.370 (5)	158
C17—H17···O3^{ii}	0.93	2.48	3.393 (5)	166

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2032).

References

Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Jeffrey, G. A. & Saenger, W. (1991). *Hydrogen Bonding in Biological Structures*. New York: Springer-Verlag.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Versions 1.171. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Sheldrick, G. M. (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
Sieroń, L. (2005*a*). *Acta Cryst.* E**61**, o2091–o2092.
Sieroń, L. (2005*b*). *Anal. Sci. X-Ray Struct. Anal. Online*, **21**, x179–x180.
Sieroń, L. (2007*a*). *Acta Cryst.* E**63**, o1199–o1200.
Sieroń, L. (2007*b*). *Acta Cryst.* E**63**, o2089–o2090.
Sieroń, L. (2007*c*). *Acta Cryst.* E**63**, o2508.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

 doi:10.1107/S1600536807035428

supplementary materials

Acta Cryst. (2007). E**63**, o3585 [**doi:10.1107/S1600536807035428**]

Benzimidazolium perchlorate

L. Sieron

Comment

The title compound, (I), was investigated as part of a structural study on hydrogen bonding in *N*-heterocyclic perchlorate salts (Sieroń, 2005*a*, *b*, 2007*a*, *b*, *c*). In (I), the asymmetric unit is composed of two benzimidazolium cations and two perchlorate anions (Fig. 1). The cations and anions are linked together by intermolecular N–H···O hydrogen bonds, forming fused 12-membered rings, described by the graph-set notations as $R^3_4(12)$ (Etter *et al.*, 1990). The substructure based on these motif forms layers lying parallel to the (102) plane (Fig. 2). The distance between neighboring planes is *ca* 3.3 Å. One of the N-bonded H atoms of both benzotriazolium cations are engaged in a bifurcated unsymmetrical (strong and weak) hydrogen bonds. Each of these hydrogen bonds involve two perchlorate O atoms. A bifurcation is confirmed by the sums of angles about atoms H1 and H11, which are 355.5° and 357.9°, respectively (Jeffrey & Saenger, 1991). The crystal packing is stabilized by weak intermolecular C–H···O interactions to build up a three-dimensional network.

Experimental

The title compound was prepared by reaction of stoichiometric amounts of benzimidazole and perchloric acid. The resulting solid was recrystallized from water at room temperature.

Refinement

All H atoms were initially located in a difference Fourier map. Afterwards they were placed in geometrically idealized positions and constrained to ride on their parent atoms, with N—H and C—H distances of 0.86 and 0.97 Å, respectively, and $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Figures

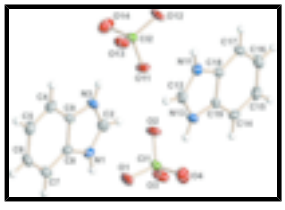


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

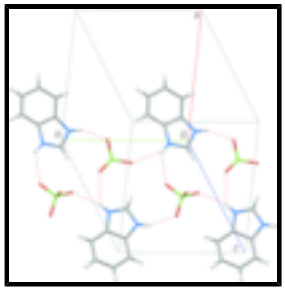


Fig. 2. The packing of (I), showing molecules connected by N—H···O hydrogen bonds (dashed lines) into sheets approximately parallel to the (102) plane.

Benzimidazolium perchlorate

Crystal data

$C_7H_7N_2^+·ClO_4^-$	F_{000} = 896
M_r = 218.60	D_x = 1.689 Mg m^{-3}
Orthorhombic, *Pca*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 11107 reflections
a = 9.9885 (2) Å	θ = 3.0–29.0º
b = 9.0509 (1) Å	μ = 0.43 mm^{-1}
c = 19.0182 (3) Å	*T* = 297 K
V = 1719.34 (5) Å^3	Plate, colourless
Z = 8	0.60 × 0.30 × 0.06 mm

Data collection

Kuma KM4 CCD area-detector diffractometer	2923 independent reflections
Monochromator: graphite	2676 reflections with $I > 2\sigma(I)$
Detector resolution: 8.2356 pixels mm^{-1}	R_{int} = 0.018
T = 297 K	θ_{max} = 25.0º
ω scans	θ_{min} = 3.0º
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)	*h* = −11→11
T_{min} = 0.781, T_{max} = 0.974	*k* = −10→10
16719 measured reflections	*l* = −22→22

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.041	$w = 1/[\sigma^2(F_o^2) + (0.0829P)^2 + 0.431P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.112	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.06	$\Delta\rho_{max}$ = 0.87 e Å^{-3}
2923 reflections	$\Delta\rho_{min}$ = −0.40 e Å^{-3}

253 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with 1462 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.04 (6)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating –*R*-factor-obs *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}
N1	0.1711 (3)	1.0228 (3)	0.83955 (19)	0.0372 (9)
N3	0.1090 (3)	0.8084 (3)	0.87538 (15)	0.0407 (9)
C2	0.1928 (3)	0.8817 (4)	0.83387 (18)	0.0429 (10)
C4	−0.0706 (4)	0.8899 (5)	0.96160 (19)	0.0443 (13)
C5	−0.1273 (4)	1.0173 (5)	0.9862 (3)	0.0467 (12)
C6	−0.0876 (4)	1.1586 (4)	0.9630 (2)	0.0432 (11)
C7	0.0100 (3)	1.1771 (4)	0.9134 (2)	0.0408 (11)
C8	0.0692 (4)	1.0482 (4)	0.88770 (18)	0.0339 (11)
C9	0.0286 (3)	0.9077 (3)	0.91156 (19)	0.0349 (10)
N11	0.0702 (3)	0.4754 (4)	0.69468 (19)	0.0373 (9)
N13	0.1382 (3)	0.6899 (3)	0.66146 (16)	0.0401 (9)
C12	0.0526 (3)	0.6176 (4)	0.70226 (18)	0.0419 (10)
C14	0.3180 (4)	0.6058 (4)	0.57613 (19)	0.0381 (11)
C15	0.3723 (4)	0.4758 (5)	0.5490 (2)	0.0457 (14)
C16	0.3277 (4)	0.3378 (5)	0.5725 (2)	0.0473 (12)
C17	0.2294 (4)	0.3210 (4)	0.62179 (18)	0.0397 (10)
C18	0.1730 (4)	0.4499 (4)	0.64646 (18)	0.0335 (11)
C19	0.2161 (3)	0.5872 (4)	0.62590 (17)	0.0321 (10)
Cl1	0.44447 (8)	0.96264 (9)	0.68820 (4)	0.0339 (3)
O1	0.3939 (3)	1.0716 (3)	0.73581 (17)	0.0557 (10)
O2	0.4461 (3)	0.8223 (3)	0.72205 (16)	0.0645 (10)
O3	0.5778 (3)	1.0008 (3)	0.6670 (2)	0.0581 (15)
O4	0.3618 (3)	0.9553 (4)	0.6270 (2)	0.0717 (12)
Cl2	0.29983 (8)	0.46265 (9)	0.84884 (4)	0.0350 (3)
O11	0.3484 (3)	0.5709 (3)	0.79958 (17)	0.0554 (9)
O12	0.2998 (4)	0.3209 (3)	0.81592 (16)	0.0672 (11)
O13	0.1659 (3)	0.4988 (3)	0.8680 (2)	0.0541 (13)
O14	0.3814 (3)	0.4610 (4)	0.9092 (2)	0.0743 (12)

H1	0.21380	1.09040	0.81700	0.0450*
H2	0.25720	0.83860	0.80510	0.0520*
H3	0.10520	0.71380	0.87920	0.0490*
H4	−0.09700	0.79720	0.97750	0.0530*
H5	−0.19490	1.01040	1.01960	0.0560*
H6	−0.12900	1.24150	0.98200	0.0520*
H7	0.03560	1.27020	0.89760	0.0490*
H11	0.02560	0.40790	0.71620	0.0450*
H12	−0.01030	0.66140	0.73170	0.0500*
H13	0.14440	0.78440	0.65780	0.0480*
H14	0.34750	0.69870	0.56210	0.0460*
H15	0.43890	0.48090	0.51490	0.0550*
H16	0.36680	0.25340	0.55360	0.0570*
H17	0.20230	0.22860	0.63770	0.0480*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0363 (16)	0.0406 (15)	0.0346 (18)	−0.0029 (13)	0.0032 (15)	0.0083 (13)
N3	0.0473 (17)	0.0253 (14)	0.0495 (16)	0.0056 (12)	−0.0026 (14)	−0.0043 (12)
C2	0.0382 (16)	0.047 (2)	0.0436 (17)	0.0074 (17)	−0.0005 (15)	−0.0034 (16)
C4	0.045 (2)	0.054 (3)	0.0339 (18)	−0.0130 (18)	0.0038 (16)	0.0057 (18)
C5	0.038 (2)	0.065 (2)	0.037 (2)	−0.0036 (18)	0.0033 (18)	−0.0060 (17)
C6	0.0455 (19)	0.039 (2)	0.045 (2)	0.0018 (16)	−0.0006 (16)	−0.0139 (17)
C7	0.0388 (19)	0.0317 (17)	0.052 (2)	−0.0025 (14)	−0.0051 (16)	−0.0034 (16)
C8	0.0323 (18)	0.037 (2)	0.0324 (17)	−0.0023 (15)	−0.0012 (14)	0.0060 (14)
C9	0.0382 (18)	0.0243 (16)	0.0421 (18)	−0.0040 (14)	−0.0070 (15)	−0.0011 (15)
N11	0.0297 (15)	0.0434 (15)	0.0388 (18)	0.0000 (13)	0.0066 (15)	−0.0014 (14)
N13	0.0439 (16)	0.0274 (14)	0.0490 (15)	0.0046 (12)	−0.0033 (13)	−0.0019 (12)
C12	0.0366 (16)	0.050 (2)	0.0391 (16)	0.0068 (16)	0.0030 (15)	−0.0066 (15)
C14	0.0400 (19)	0.034 (2)	0.0402 (19)	−0.0049 (15)	−0.0035 (15)	0.0091 (16)
C15	0.038 (2)	0.068 (3)	0.031 (2)	0.0022 (18)	0.0044 (18)	−0.0073 (19)
C16	0.043 (2)	0.050 (2)	0.049 (2)	0.0070 (18)	−0.0005 (17)	−0.0135 (18)
C17	0.046 (2)	0.0269 (16)	0.0463 (18)	−0.0016 (14)	−0.0029 (16)	−0.0004 (14)
C18	0.0352 (18)	0.0297 (19)	0.0356 (18)	−0.0004 (14)	−0.0069 (15)	−0.0008 (14)
C19	0.0330 (18)	0.0338 (18)	0.0295 (15)	0.0078 (14)	−0.0050 (13)	−0.0033 (14)
Cl1	0.0330 (4)	0.0262 (4)	0.0425 (5)	−0.0005 (3)	0.0064 (4)	−0.0017 (4)
O1	0.0638 (18)	0.0384 (16)	0.0648 (18)	0.0065 (13)	0.0235 (15)	−0.0125 (13)
O2	0.094 (2)	0.0277 (15)	0.0718 (19)	−0.0002 (12)	0.0220 (16)	0.0107 (13)
O3	0.0322 (16)	0.0402 (17)	0.102 (4)	−0.0037 (11)	0.0316 (18)	−0.0026 (14)
O4	0.068 (2)	0.077 (2)	0.070 (2)	0.0044 (19)	−0.0172 (18)	−0.0132 (19)
Cl2	0.0340 (4)	0.0273 (4)	0.0436 (5)	0.0016 (3)	0.0077 (4)	0.0039 (4)
O11	0.0701 (18)	0.0360 (15)	0.0600 (16)	−0.0022 (13)	0.0253 (15)	0.0144 (13)
O12	0.099 (2)	0.0226 (14)	0.080 (2)	−0.0070 (13)	0.0311 (17)	−0.0102 (13)
O13	0.0418 (17)	0.0396 (17)	0.081 (3)	0.0061 (11)	0.0095 (18)	0.0030 (13)
O14	0.068 (2)	0.089 (2)	0.066 (2)	−0.0168 (19)	−0.0248 (18)	0.022 (2)

Geometric parameters (Å, °)

Cl1—O1	1.431 (3)	C4—C9	1.383 (5)
Cl1—O2	1.424 (3)	C5—C6	1.410 (6)
Cl1—O3	1.434 (3)	C6—C7	1.367 (5)
Cl1—O4	1.429 (4)	C7—C8	1.396 (5)
Cl2—O11	1.440 (3)	C8—C9	1.410 (5)
Cl2—O12	1.428 (3)	C2—H2	0.93
Cl2—O13	1.425 (3)	C4—H4	0.93
Cl2—O14	1.408 (4)	C5—H5	0.93
N1—C2	1.300 (5)	C6—H6	0.93
N1—C8	1.388 (5)	C7—H7	0.93
N3—C2	1.328 (4)	C14—C19	1.400 (5)
N3—C9	1.388 (4)	C14—C15	1.395 (6)
N1—H1	0.86	C15—C16	1.399 (6)
N3—H3	0.86	C16—C17	1.366 (5)
N11—C12	1.307 (5)	C17—C18	1.378 (5)
N11—C18	1.396 (5)	C18—C19	1.372 (5)
N13—C19	1.388 (4)	C12—H12	0.93
N13—C12	1.327 (4)	C14—H14	0.93
N11—H11	0.86	C15—H15	0.93
N13—H13	0.86	C16—H16	0.93
C4—C5	1.367 (6)	C17—H17	0.93
O1—Cl1—O2	109.43 (18)	N3—C9—C4	132.9 (3)
O1—Cl1—O3	109.87 (18)	N3—C9—C8	105.0 (3)
O1—Cl1—O4	110.09 (19)	N1—C2—H2	125
O2—Cl1—O3	109.35 (18)	N3—C2—H2	125
O2—Cl1—O4	109.5 (2)	C5—C4—H4	122
O3—Cl1—O4	108.6 (2)	C9—C4—H4	122
O11—Cl2—O12	109.04 (18)	C6—C5—H5	119
O11—Cl2—O13	109.06 (18)	C4—C5—H5	119
O11—Cl2—O14	110.05 (19)	C5—C6—H6	119
O12—Cl2—O13	108.6 (2)	C7—C6—H6	119
O12—Cl2—O14	110.4 (2)	C8—C7—H7	122
O13—Cl2—O14	109.7 (2)	C6—C7—H7	122
C2—N1—C8	109.9 (3)	N11—C12—N13	109.5 (3)
C2—N3—C9	109.6 (3)	C15—C14—C19	115.6 (3)
C2—N3—H3	125	C14—C15—C16	120.7 (4)
C2—N1—H1	125	C15—C16—C17	123.2 (4)
C8—N1—H1	125	C16—C17—C18	115.7 (4)
C9—N3—H3	125	N11—C18—C17	131.6 (3)
C12—N11—C18	109.5 (3)	C17—C18—C19	122.8 (3)
C12—N13—C19	108.4 (3)	N11—C18—C19	105.6 (3)
C12—N11—H11	125	N13—C19—C18	107.0 (3)
C18—N11—H11	125	N13—C19—C14	131.0 (3)
C12—N13—H13	126	C14—C19—C18	122.0 (3)
C19—N13—H13	126	N11—C12—H12	125
N1—C2—N3	109.7 (3)	N13—C12—H12	125

C5—C4—C9	115.7 (4)	C15—C14—H14	122
C4—C5—C6	122.8 (4)	C19—C14—H14	122
C5—C6—C7	121.9 (4)	C14—C15—H15	120
C6—C7—C8	116.2 (3)	C16—C15—H15	120
N1—C8—C7	132.8 (3)	C15—C16—H16	118
C7—C8—C9	121.3 (3)	C17—C16—H16	118
N1—C8—C9	105.9 (3)	C18—C17—H17	122
C4—C9—C8	122.2 (3)	C16—C17—H17	122

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.86	2.38	3.007 (5)	130
N1—H1···O12^{i}	0.86	2.26	3.022 (4)	148
N3—H3···O13	0.86	2.05	2.863 (4)	157
N11—H11···O11^{ii}	0.86	2.38	3.011 (5)	130
N11—H11···O2^{ii}	0.86	2.23	3.011 (4)	151
N13—H13···O3^{iii}	0.86	2.06	2.866 (4)	155
C2—H2···O2	0.93	2.47	3.349 (4)	159
C2—H2···O11	0.93	2.59	3.279 (4)	131
C7—H7···O13^{i}	0.93	2.51	3.413 (5)	164
C12—H12···O12^{ii}	0.93	2.49	3.370 (5)	158
C17—H17···O3^{ii}	0.93	2.48	3.393 (5)	166

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

Fig. 1

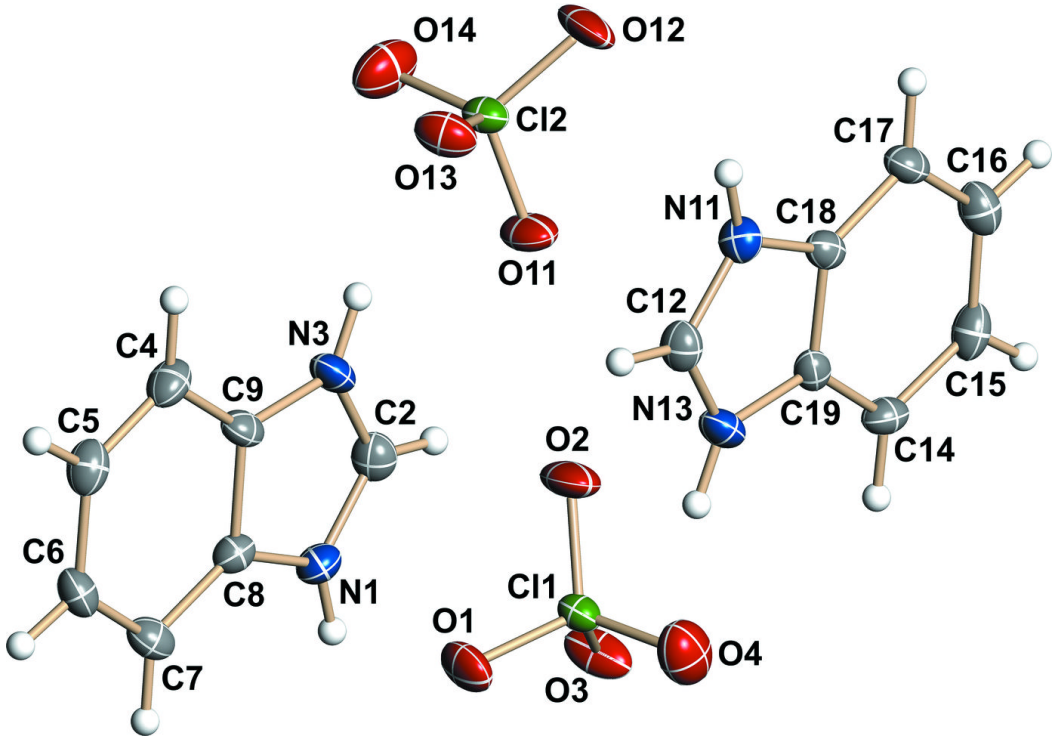

O14
O12
Cl2
O13
O11
C17
C16
N11
C18
C12
C15
N13
C19
C14
N3
C4
C9
C2
C5
O2
C6
C8
N1
Cl1
O1
O4
C7
O3

Fig. 2