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4-Aza-1-azoniabicyclo[2.2.2]octane–2-aminobenzoate–2-aminobenzoic acid (1/1/1)

Hadi D. Arman,[a] Trupta Kaulgud[a] and Edward R. T. Tiekink[b]*

[a]Department of Chemistry, The University of Texas at San Antonio, One UTSA Circle, San Antonio, Texas 78249-0698, USA, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: edward.tiekink@gmail.com

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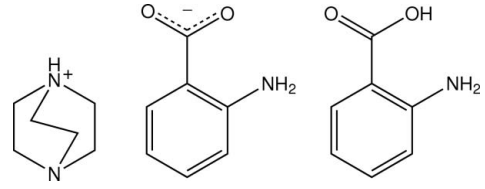
Key indicators: single-crystal X-ray study; T = 98 K; mean σ(C–C) = 0.002 Å; R factor = 0.056; wR factor = 0.128; data-to-parameter ratio = 16.6.

A 4-aza-1-azoniabicyclo[2.2.2]octane cation, a 2-aminobenzoate anion and a neutral 2-aminobenzoic acid molecule comprise the asymmetric unit of the title compound, $C_6H_{13}N_2^+\cdot C_7H_6NO_2^-\cdot C_7H_7NO_2$. An intramolecular N—H···O hydrogen bond occurs in the anion and in the neutral 2-aminobenzoic acid molecule. The cation provides a charge-assisted N—H···O hydrogen bond to the anion, and the 2-aminobenzoic acid molecule forms an O—H···N hydrogen bond to the unprotonated amino N atom in the cation. In this way, a three-component aggregate is formed. These are connected into a three-dimensional network by amino–carboxylate N—H···O hydrogen bonds. N—H···N hydrogen bonds are also observed.

Related literature

For related studies on co-crystal formation, see: Arman *et al.* (2010); Arman & Tiekink (2010); Wardell & Tiekink (2011). For examples of multi-component crystals containing the 2-aminobenzoate anion, see: Lynch *et al.* (1998); Chen & Peng (2011). For a description of the Cambridge Structural Database, see: Allen (2002).



Experimental

Crystal data

$C_6H_{13}N_2^+\cdot C_7H_6NO_2^-\cdot C_7H_7NO_2$ M_r = 386.45
Monoclinic, $P2_1/c$
a = 9.285 (3) Å
b = 16.843 (5) Å
c = 12.660 (4) Å
β = 102.127 (6)°
V = 1935.7 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 98 K
0.34 × 0.17 × 0.07 mm

Data collection

Rigaku AFC12/SATURN724 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.731, T_{max} = 1.000
16911 measured reflections
4440 independent reflections
3929 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.128
S = 1.13
4440 reflections
268 parameters
7 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···N3	0.84	1.77	2.597 (2)	168
N4—H5*n*···O3	0.93	1.64	2.546 (2)	166
N1—H2*n*···O2	0.88	2.03	2.725 (2)	135
N2—H3*n*···O3	0.88	2.04	2.696 (2)	131
N1—H1*n*···O4^{i}	0.88	2.08	2.941 (2)	165
N2—H4*n*···N1^{ii}	0.88	2.38	3.256 (2)	171

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

Data collection: *CrystalClear* (Molecular Structure Corporation & 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: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6440).

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supplementary materials

Acta Cryst. (2011). E**67**, o2933 [**doi:10.1107/S1600536811041559**]

4-Aza-1-azoniabicyclo[2.2.2]octane-2-aminobenzoate-2-aminobenzoic acid (1/1/1)

H. D. Arman, T. Kaulgud and E. R. T. Tiekink

Comment

As a part of on-going studies into co-crystallization experiments of carboxylic acids with N-containing molecules (Arman *et al.* 2010; Arman & Tiekink, 2010; Wardell & Tiekink, 2011), the 1:2 co-crystallization of 1,4-diazabicyclo[2.2.2]octane (DABCO) and 2-aminobenzoic was investigated, leading to the isolation of (I).

The crystallographic asymmetric unit of (I) comprises a 4-aza-1-azoniabicyclo(2.2.2)octane cation, Fig. 1, a 2-aminobenzoate anion, Fig. 2, and a neutral 2-aminobenzoic acid molecule, Fig. 3. While there are many examples of 4-aza-1-azoniabicyclo(2.2.2)octane cations and neutral 2-aminobenzoic acid molecules in the crystallographic literature (Allen, 2002), examples of 2-aminobenzoate anions are comparatively rare in all-organic molecules (Lynch *et al.*, 1998; Chen & Peng, 2011). The ions and neutral benzoic acid derivative associate into a three-molecule aggregate *via* N^+—H···O and O—H···N hydrogen bonds formed by and to the cation, Fig. 4 and Table 1; intramolecular N—H···O hydrogen bonds are also noted in the benzoate and benzoic acid species, Table 1.

The three component aggregates are connected into the three-dimensional architecture by hydrogen bonds involving the amino-H atoms not participating in intramolecular N—H···O interactions, Fig. 5 and Table 1.

Experimental

Colourless crystals of (I) were isolated from the 1:2 co-crystallization of 1,4-diazabicyclo[2.2.2]octane (Sigma-Aldrich, 0.089 mmol) and anthranilic acid (Sigma-Aldrich, 0.19 mmol) in chloroform solution (6 ml); *M*.pt. 427–430 K.

Refinement

The C-bound H-atoms were placed in calculated positions (C—H 0.95–0.99 Å) and were included in the refinement in the riding model approximation with U_{iso}(H) set to 1.2U_{eq}(C). The O– and N-bound H-atoms were located in a difference Fourier map and were refined with distance restraints of O—H 0.840±0.001 Å and N—H = 0.088±0.001 Å, respectively, and with U_{iso}(H) = 1.5U_{eq}(O, N).

Figures

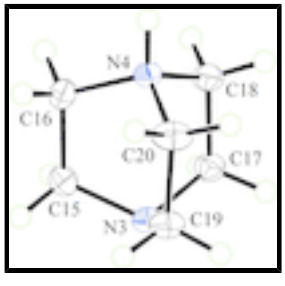

Fig. 1. Molecular structure of the 4-aza-1-azoniabicyclo(2.2.2)octane cation in (I) showing atom-labelling scheme and displacement ellipsoids at the 50% probability level.

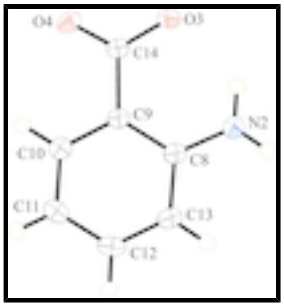

Fig. 2. Molecular structure of the 2-aminobenzoate anion in (I) showing atom-labelling scheme and displacement ellipsoids at the 50% probability level.

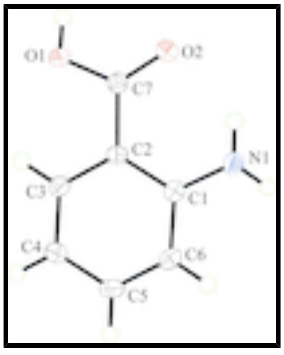

Fig. 3. Molecular structure of the neutral 2-aminobenzoic acid molecule in (I) showing atom-labelling scheme and displacement ellipsoids at the 50% probability level.

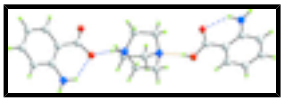

Fig. 4. Three component aggregate in (I) held together by O—H···N and N—H···O hydrogen bonds shown as orange and blue dashed lines, respectively.

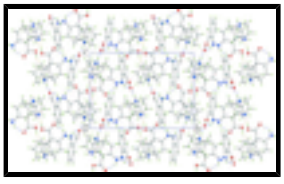

Fig. 5. View in projection down the *a* axis of the unit-cell contents of (I). The O—H···N and N—H···O hydrogen bonds are shown as orange and blue dashed lines, respectively.

4-Aza-1-azoniabicyclo[2.2.2]octane–2-aminobenzoate–2-aminobenzoic acid (1/1/1)

Crystal data

$C_6H_{13}N_2^+ \cdot C_7H_6NO_2^- \cdot C_7H_7NO_2$	$F(000) = 824$
$M_r = 386.45$	$D_x = 1.326$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 8162 reflections
$a = 9.285$ (3) Å	$\theta = 2.0$–$40.6°$
$b = 16.843$ (5) Å	$\mu = 0.09$ mm^{-1}
$c = 12.660$ (4) Å	$T = 98$ K
$\beta = 102.127$ (6)°	Block, colourless
$V = 1935.7$ (10) Å^3	0.34 × 0.17 × 0.07 mm
$Z = 4$	

Data collection

Rigaku AFC12K/SATURN724 diffractometer	4440 independent reflections

Radiation source: fine-focus sealed tube	3929 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.054$
ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$h = -12 \rightarrow 12$
$T_{min} = 0.731$, $T_{max} = 1.000$	$k = -19 \rightarrow 21$
16911 measured reflections	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.056$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.128$	H-atom parameters constrained
$S = 1.13$	$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.9109P]$ where $P = (F_o^2 + 2F_c^2)/3$
4440 reflections	$(\Delta/\sigma)_{max} < 0.001$
268 parameters	$\Delta\rho_{max} = 0.30$ e Å^{-3}
7 restraints	$\Delta\rho_{min} = -0.23$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.73506 (15)	0.46965 (8)	1.01396 (10)	0.0286 (3)
H1o	0.7433	0.4752	0.9495	0.043*
O2	0.63182 (15)	0.35565 (8)	0.94734 (10)	0.0280 (3)
O3	0.87552 (13)	0.60731 (8)	0.46906 (9)	0.0255 (3)
O4	0.63909 (13)	0.62403 (7)	0.38987 (10)	0.0245 (3)
N1	0.58477 (16)	0.24385 (8)	1.09196 (12)	0.0225 (3)
H1n	0.5262	0.2075	1.1100	0.034*
H2n	0.5710	0.2596	1.0243	0.034*
N2	1.07213 (17)	0.69313 (11)	0.38788 (13)	0.0320 (4)
H3n	1.0547	0.6697	0.4460	0.048*
H4n	1.1631	0.7060	0.3850	0.048*

N3	0.76627 (15)	0.50739 (8)	0.82113 (11)	0.0203 (3)
N4	0.79912 (15)	0.55587 (8)	0.63832 (11)	0.0207 (3)
H5n	0.8114	0.5735	0.5712	0.025*
C1	0.61784 (17)	0.30546 (9)	1.16546 (13)	0.0184 (3)
C2	0.66288 (17)	0.38151 (9)	1.13704 (12)	0.0175 (3)
C3	0.69563 (17)	0.44053 (10)	1.21593 (13)	0.0200 (3)
H3	0.7229	0.4918	1.1959	0.024*
C4	0.68936 (19)	0.42613 (11)	1.32282 (13)	0.0232 (3)
H4	0.7110	0.4671	1.3754	0.028*
C5	0.65063 (18)	0.35033 (10)	1.35151 (13)	0.0228 (3)
H5	0.6493	0.3392	1.4249	0.027*
C6	0.61417 (17)	0.29115 (10)	1.27466 (13)	0.0209 (3)
H6	0.5863	0.2402	1.2957	0.025*
C7	0.67418 (17)	0.40032 (10)	1.02372 (13)	0.0192 (3)
C8	0.96688 (18)	0.68766 (10)	0.29348 (13)	0.0211 (3)
C9	0.82171 (17)	0.66015 (9)	0.29093 (13)	0.0185 (3)
C10	0.72053 (18)	0.65777 (10)	0.19229 (13)	0.0211 (3)
H10	0.6227	0.6407	0.1912	0.025*
C11	0.7582 (2)	0.67941 (10)	0.09614 (14)	0.0253 (4)
H11	0.6883	0.6759	0.0298	0.030*
C12	0.9008 (2)	0.70645 (10)	0.09858 (14)	0.0259 (4)
H12	0.9283	0.7215	0.0333	0.031*
C13	1.00247 (19)	0.71146 (10)	0.19520 (14)	0.0243 (4)
H13	1.0983	0.7313	0.1955	0.029*
C14	0.77248 (18)	0.62962 (9)	0.38957 (13)	0.0190 (3)
C15	0.92485 (19)	0.50551 (11)	0.81698 (14)	0.0254 (4)
H15A	0.9641	0.4512	0.8332	0.031*
H15B	0.9806	0.5420	0.8721	0.031*
C16	0.94441 (19)	0.53077 (12)	0.70394 (15)	0.0289 (4)
H16A	1.0154	0.5753	0.7103	0.035*
H16B	0.9837	0.4858	0.6682	0.035*
C17	0.71221 (19)	0.58987 (10)	0.80251 (14)	0.0241 (4)
H17A	0.7639	0.6242	0.8620	0.029*
H17B	0.6055	0.5916	0.8022	0.029*
C18	0.7387 (2)	0.62134 (10)	0.69428 (14)	0.0262 (4)
H18A	0.6450	0.6406	0.6491	0.031*
H18B	0.8091	0.6662	0.7070	0.031*
C19	0.6829 (2)	0.45573 (11)	0.73500 (13)	0.0255 (4)
H19A	0.5780	0.4541	0.7403	0.031*
H19B	0.7224	0.4010	0.7443	0.031*
C20	0.6952 (2)	0.48755 (11)	0.62315 (14)	0.0294 (4)
H20A	0.7318	0.4452	0.5813	0.035*
H20B	0.5972	0.5048	0.5826	0.035*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0416 (7)	0.0283 (7)	0.0170 (6)	−0.0134 (5)	0.0084 (5)	0.0005 (5)

O2	0.0388 (7)	0.0268 (7)	0.0190 (6)	−0.0089 (5)	0.0072 (5)	−0.0038 (5)
O3	0.0244 (6)	0.0334 (7)	0.0195 (6)	−0.0001 (5)	0.0065 (5)	0.0052 (5)
O4	0.0217 (6)	0.0242 (6)	0.0305 (7)	0.0010 (5)	0.0116 (5)	0.0023 (5)
N1	0.0269 (7)	0.0176 (7)	0.0247 (7)	−0.0022 (5)	0.0093 (6)	−0.0011 (6)
N2	0.0226 (7)	0.0477 (10)	0.0253 (8)	−0.0078 (7)	0.0038 (6)	0.0046 (7)
N3	0.0226 (7)	0.0212 (7)	0.0176 (6)	−0.0018 (5)	0.0052 (5)	0.0014 (5)
N4	0.0224 (7)	0.0229 (7)	0.0181 (6)	0.0010 (5)	0.0072 (5)	0.0027 (5)
C1	0.0162 (7)	0.0179 (8)	0.0214 (8)	0.0017 (6)	0.0048 (6)	0.0006 (6)
C2	0.0168 (7)	0.0196 (8)	0.0164 (7)	0.0012 (6)	0.0044 (6)	0.0009 (6)
C3	0.0210 (7)	0.0192 (8)	0.0203 (8)	−0.0014 (6)	0.0057 (6)	0.0006 (6)
C4	0.0265 (8)	0.0257 (9)	0.0179 (8)	−0.0038 (6)	0.0055 (6)	−0.0018 (7)
C5	0.0230 (8)	0.0286 (9)	0.0173 (8)	0.0001 (6)	0.0051 (6)	0.0046 (7)
C6	0.0213 (8)	0.0185 (8)	0.0236 (8)	0.0013 (6)	0.0067 (7)	0.0049 (6)
C7	0.0195 (7)	0.0191 (8)	0.0193 (8)	0.0001 (6)	0.0046 (6)	0.0001 (6)
C8	0.0225 (8)	0.0192 (8)	0.0226 (8)	0.0023 (6)	0.0069 (6)	0.0009 (6)
C9	0.0220 (8)	0.0151 (7)	0.0199 (8)	0.0015 (6)	0.0074 (6)	0.0004 (6)
C10	0.0233 (8)	0.0164 (8)	0.0237 (8)	0.0013 (6)	0.0053 (6)	−0.0014 (6)
C11	0.0321 (9)	0.0232 (9)	0.0199 (8)	0.0047 (7)	0.0040 (7)	0.0001 (7)
C12	0.0362 (9)	0.0215 (8)	0.0229 (8)	0.0053 (7)	0.0127 (7)	0.0044 (7)
C13	0.0254 (8)	0.0220 (8)	0.0283 (9)	0.0011 (6)	0.0117 (7)	0.0035 (7)
C14	0.0226 (8)	0.0150 (7)	0.0206 (8)	0.0006 (6)	0.0073 (6)	−0.0009 (6)
C15	0.0221 (8)	0.0312 (9)	0.0215 (8)	0.0024 (7)	0.0011 (7)	0.0047 (7)
C16	0.0202 (8)	0.0394 (11)	0.0284 (9)	0.0058 (7)	0.0084 (7)	0.0089 (8)
C17	0.0294 (9)	0.0230 (8)	0.0222 (8)	0.0026 (7)	0.0106 (7)	0.0007 (7)
C18	0.0319 (9)	0.0216 (8)	0.0288 (9)	0.0050 (7)	0.0146 (8)	0.0047 (7)
C19	0.0305 (9)	0.0251 (9)	0.0203 (8)	−0.0082 (7)	0.0036 (7)	−0.0004 (7)
C20	0.0396 (10)	0.0300 (10)	0.0173 (8)	−0.0114 (8)	0.0028 (7)	−0.0022 (7)

Geometric parameters (Å, °)

O1—C7	1.315 (2)	C6—H6	0.9500
O1—H1O	0.8402	C8—C13	1.411 (2)
O2—C7	1.223 (2)	C8—C9	1.419 (2)
O3—C14	1.290 (2)	C9—C10	1.397 (2)
O4—C14	1.243 (2)	C9—C14	1.507 (2)
N1—C1	1.384 (2)	C10—C11	1.384 (2)
N1—H1N	0.8800	C10—H10	0.9500
N1—H2N	0.8802	C11—C12	1.394 (3)
N2—C8	1.379 (2)	C11—H11	0.9500
N2—H3N	0.8800	C12—C13	1.382 (3)
N2—H4N	0.8799	C12—H12	0.9500
N3—C17	1.479 (2)	C13—H13	0.9500
N3—C19	1.480 (2)	C15—C16	1.540 (2)
N3—C15	1.485 (2)	C15—H15A	0.9900
N4—C18	1.483 (2)	C15—H15B	0.9900
N4—C20	1.488 (2)	C16—H16A	0.9900
N4—C16	1.489 (2)	C16—H16B	0.9900
N4—H5N	0.9300	C17—C18	1.536 (2)
C1—C6	1.411 (2)	C17—H17A	0.9900

C1—C2	1.417 (2)	C17—H17B	0.9900
C2—C3	1.397 (2)	C18—H18A	0.9900
C2—C7	1.494 (2)	C18—H18B	0.9900
C3—C4	1.388 (2)	C19—C20	1.541 (2)
C3—H3	0.9500	C19—H19A	0.9900
C4—C5	1.395 (2)	C19—H19B	0.9900
C4—H4	0.9500	C20—H20A	0.9900
C5—C6	1.384 (2)	C20—H20B	0.9900
C5—H5	0.9500		
C7—O1—H1O	108.5	C10—C11—H11	120.6
C1—N1—H1N	114.1	C12—C11—H11	120.6
C1—N1—H2N	113.2	C13—C12—C11	120.57 (16)
H1N—N1—H2N	119.4	C13—C12—H12	119.7
C8—N2—H3N	118.3	C11—C12—H12	119.7
C8—N2—H4N	119.5	C12—C13—C8	121.35 (16)
H3N—N2—H4N	119.5	C12—C13—H13	119.3
C17—N3—C19	109.10 (14)	C8—C13—H13	119.3
C17—N3—C15	108.67 (13)	O4—C14—O3	123.47 (15)
C19—N3—C15	109.31 (14)	O4—C14—C9	120.27 (15)
C18—N4—C20	109.67 (14)	O3—C14—C9	116.19 (14)
C18—N4—C16	109.51 (14)	N3—C15—C16	109.79 (13)
C20—N4—C16	109.83 (14)	N3—C15—H15A	109.7
C18—N4—H5N	109.3	C16—C15—H15A	109.7
C20—N4—H5N	109.3	N3—C15—H15B	109.7
C16—N4—H5N	109.3	C16—C15—H15B	109.7
N1—C1—C6	118.89 (15)	H15A—C15—H15B	108.2
N1—C1—C2	122.85 (14)	N4—C16—C15	109.08 (13)
C6—C1—C2	118.18 (15)	N4—C16—H16A	109.9
C3—C2—C1	119.56 (14)	C15—C16—H16A	109.9
C3—C2—C7	119.14 (14)	N4—C16—H16B	109.9
C1—C2—C7	121.29 (14)	C15—C16—H16B	109.9
C4—C3—C2	121.70 (15)	H16A—C16—H16B	108.3
C4—C3—H3	119.2	N3—C17—C18	110.71 (13)
C2—C3—H3	119.2	N3—C17—H17A	109.5
C3—C4—C5	118.66 (16)	C18—C17—H17A	109.5
C3—C4—H4	120.7	N3—C17—H17B	109.5
C5—C4—H4	120.7	C18—C17—H17B	109.5
C6—C5—C4	120.95 (15)	H17A—C17—H17B	108.1
C6—C5—H5	119.5	N4—C18—C17	108.48 (13)
C4—C5—H5	119.5	N4—C18—H18A	110.0
C5—C6—C1	120.87 (15)	C17—C18—H18A	110.0
C5—C6—H6	119.6	N4—C18—H18B	110.0
C1—C6—H6	119.6	C17—C18—H18B	110.0
O2—C7—O1	123.04 (15)	H18A—C18—H18B	108.4
O2—C7—C2	123.56 (15)	N3—C19—C20	110.12 (14)
O1—C7—C2	113.39 (14)	N3—C19—H19A	109.6
N2—C8—C13	119.39 (16)	C20—C19—H19A	109.6
N2—C8—C9	122.57 (15)	N3—C19—H19B	109.6
C13—C8—C9	118.03 (15)	C20—C19—H19B	109.6

C10—C9—C8	119.14 (15)	H19A—C19—H19B	108.2
C10—C9—C14	117.83 (15)	N4—C20—C19	108.78 (14)
C8—C9—C14	122.98 (15)	N4—C20—H20A	109.9
C11—C10—C9	122.13 (16)	C19—C20—H20A	109.9
C11—C10—H10	118.9	N4—C20—H20B	109.9
C9—C10—H10	118.9	C19—C20—H20B	109.9
C10—C11—C12	118.74 (16)	H20A—C20—H20B	108.3
N1—C1—C2—C3	−179.44 (14)	C11—C12—C13—C8	1.6 (3)
C6—C1—C2—C3	−2.8 (2)	N2—C8—C13—C12	179.51 (17)
N1—C1—C2—C7	1.4 (2)	C9—C8—C13—C12	−1.6 (2)
C6—C1—C2—C7	178.02 (14)	C10—C9—C14—O4	−19.9 (2)
C1—C2—C3—C4	1.9 (2)	C8—C9—C14—O4	162.91 (15)
C7—C2—C3—C4	−178.95 (15)	C10—C9—C14—O3	157.33 (15)
C2—C3—C4—C5	0.6 (2)	C8—C9—C14—O3	−19.9 (2)
C3—C4—C5—C6	−2.2 (3)	C17—N3—C15—C16	−62.35 (18)
C4—C5—C6—C1	1.2 (3)	C19—N3—C15—C16	56.62 (19)
N1—C1—C6—C5	178.09 (15)	C18—N4—C16—C15	57.55 (19)
C2—C1—C6—C5	1.3 (2)	C20—N4—C16—C15	−62.94 (19)
C3—C2—C7—O2	−171.35 (16)	N3—C15—C16—N4	5.0 (2)
C1—C2—C7—O2	7.8 (2)	C19—N3—C17—C18	−62.15 (18)
C3—C2—C7—O1	9.0 (2)	C15—N3—C17—C18	56.95 (18)
C1—C2—C7—O1	−171.82 (15)	C20—N4—C18—C17	57.54 (18)
N2—C8—C9—C10	178.78 (16)	C16—N4—C18—C17	−63.04 (18)
C13—C8—C9—C10	−0.1 (2)	N3—C17—C18—N4	4.8 (2)
N2—C8—C9—C14	−4.0 (3)	C17—N3—C19—C20	55.90 (18)
C13—C8—C9—C14	177.12 (15)	C15—N3—C19—C20	−62.81 (18)
C8—C9—C10—C11	1.8 (2)	C18—N4—C20—C19	−63.53 (19)
C14—C9—C10—C11	−175.57 (15)	C16—N4—C20—C19	56.86 (19)
C9—C10—C11—C12	−1.8 (3)	N3—C19—C20—N4	5.3 (2)
C10—C11—C12—C13	0.0 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···N3	0.84	1.77	2.597 (2)	168
N4—H5n···O3	0.93	1.64	2.546 (2)	166
N1—H2n···O2	0.88	2.03	2.725 (2)	135
N2—H3n···O3	0.88	2.04	2.696 (2)	131
N1—H1n···O4^{i}	0.88	2.08	2.941 (2)	165
N2—H4n···N1^{ii}	0.88	2.38	3.256 (2)	171

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

Fig. 1

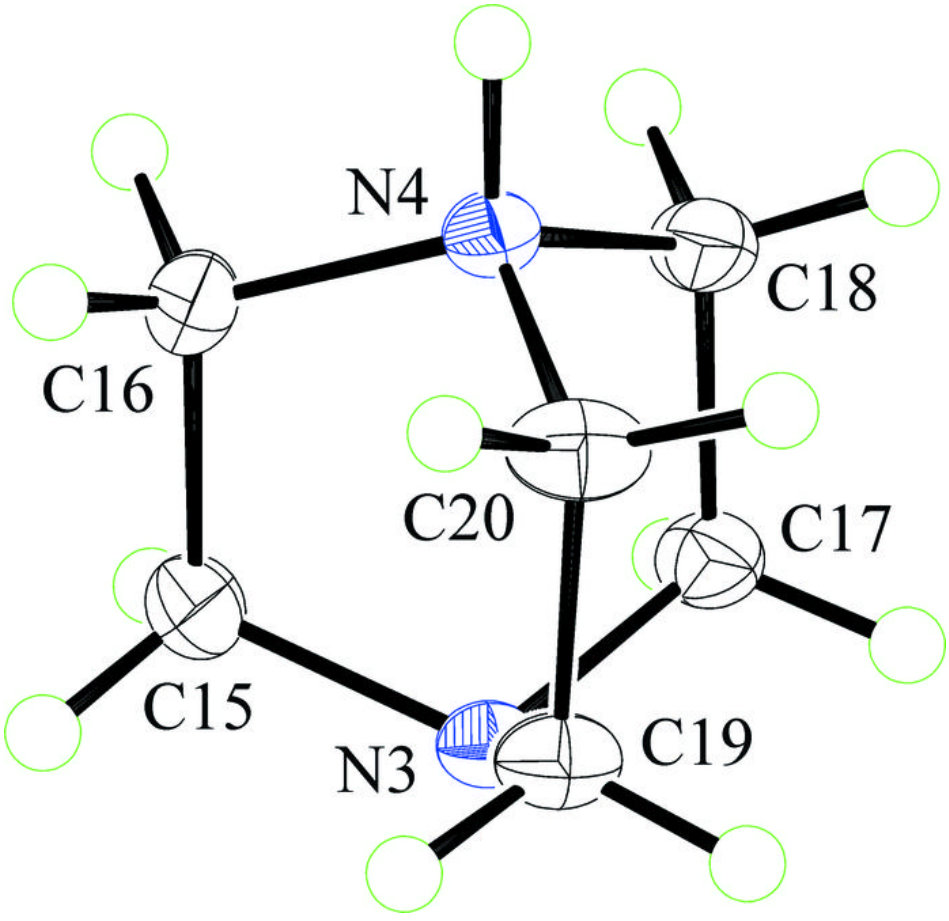

Fig. 2

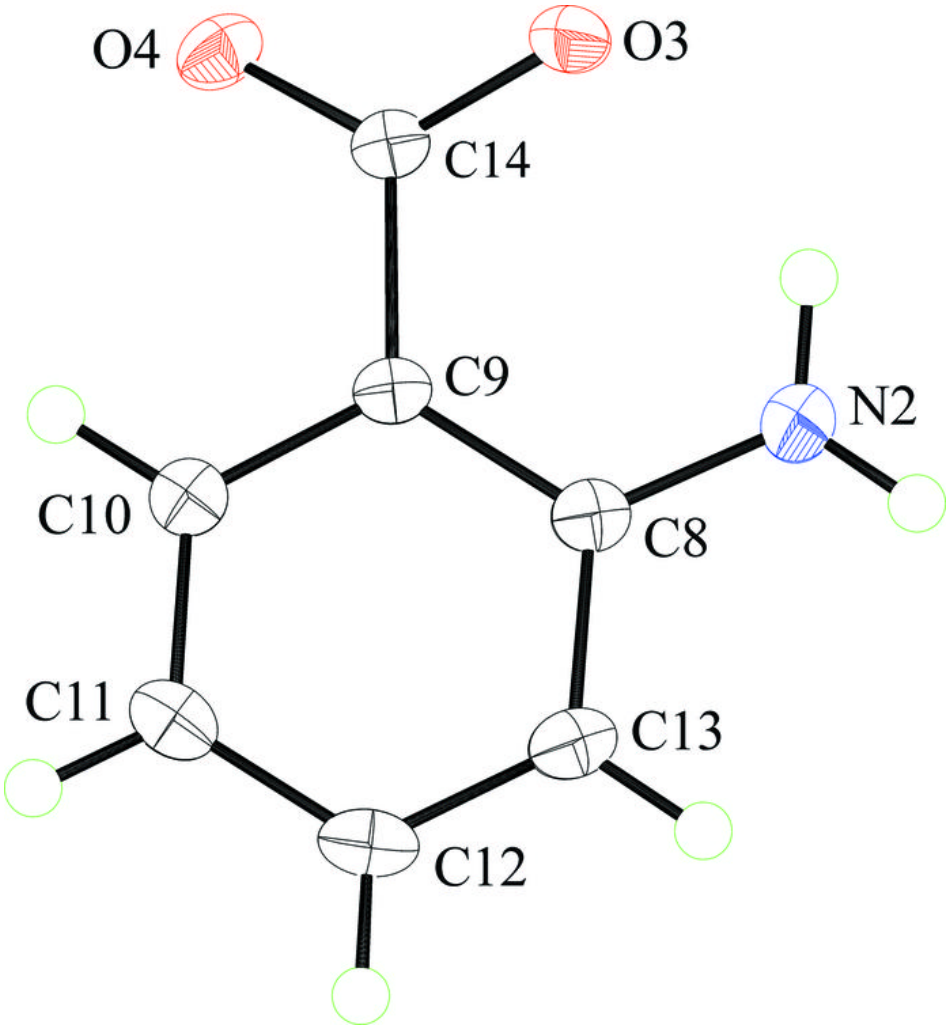

Fig. 3

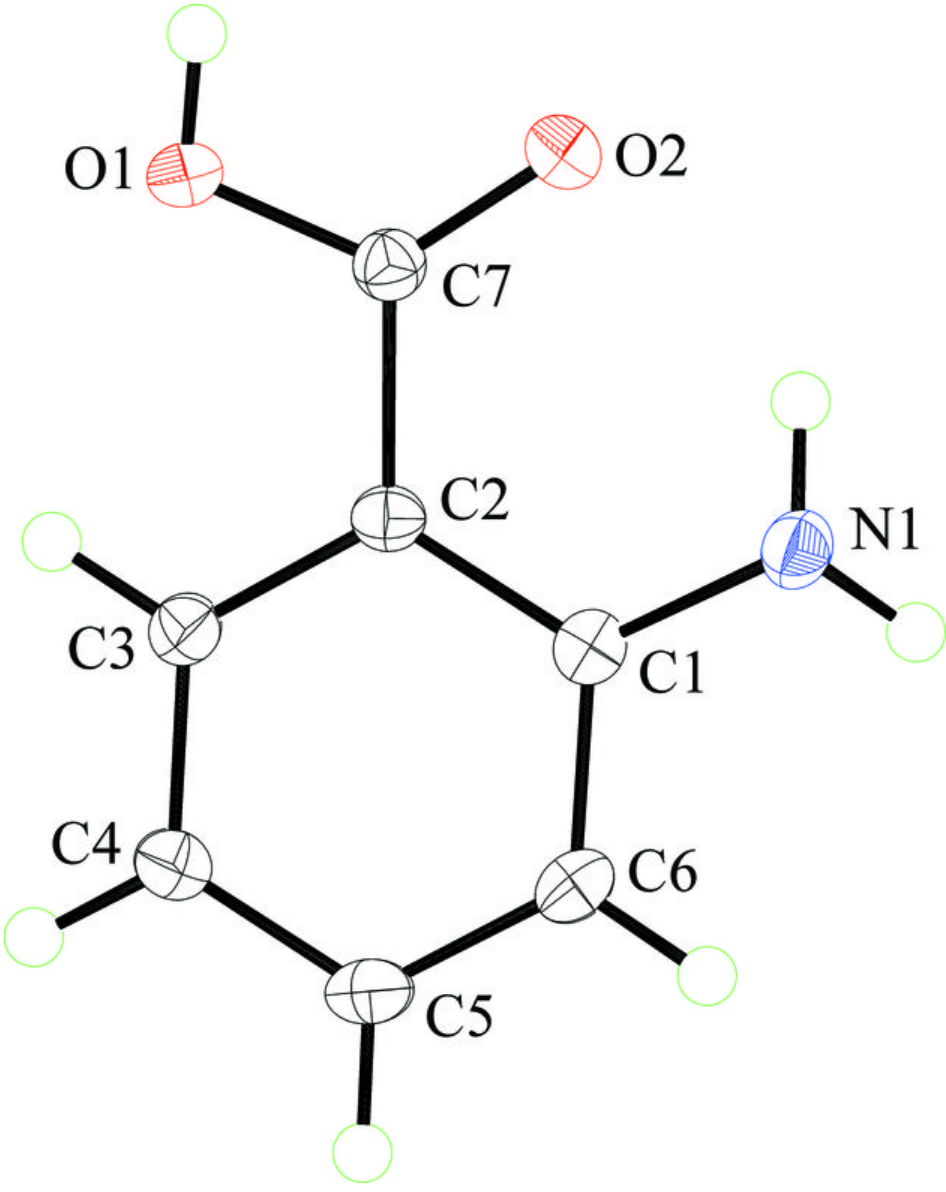

Fig. 4

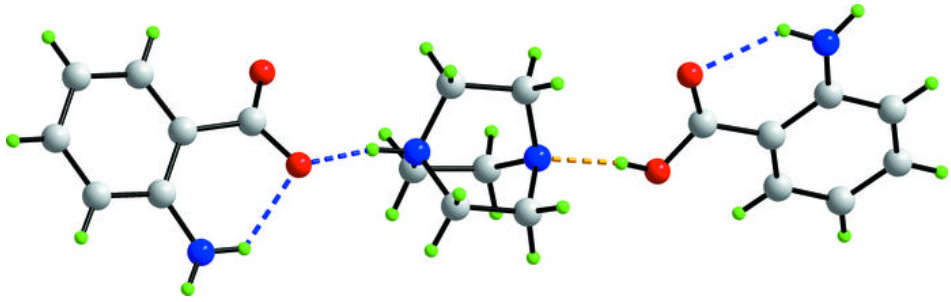

Fig. 5