organic compounds

1,4-Diazoniabicyclo[2.2.2]octane terephthalate
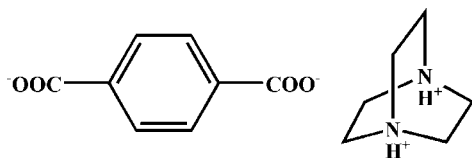
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In the title compound, $C_6H_{14}N_2^{2+} \cdot C_8H_4O_4^{2-}$, the protonated 1,4-diazoniabicyclo[2.2.2]octane cations and the deprotonated terephthalate anions are alternately linked by N—H···O hydrogen bonds into chains.

Experimental

Crystal data

$C_6H_{14}N_2^{2+} \cdot C_8H_4O_4^{2-}$
$M_r = 278.30$
Triclinic, $P\bar{1}$
$a = 7.8046\ (10)$ Å
$b = 9.5482\ (2)$ Å
$c = 10.8075\ (2)$ Å
$\alpha = 65.900\ (10)°$
$\beta = 78.360\ (10)°$

$\gamma = 66.800\ (10)°$
$V = 672.39\ (2)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 293\ (2)$ K
$0.23 \times 0.13 \times 0.08$ mm

Data collection

Siemens SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.900$, $T_{max} = 0.950$

7312 measured reflections
2377 independent reflections
1779 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.125$
$S = 1.04$
2377 reflections
188 parameters
2 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2	0.954 (15)	1.623 (15)	2.5757 (19)	176.8 (17)
N2—H2N···O4^i	0.959 (15)	1.600 (15)	2.5589 (19)	177.9 (18)

Symmetry code: (i) $x - 2, y + 1, z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Bergerhoff et al., 1996); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2092).

References

Bergerhoff, G., Berndt, M. & Brandenburg, K. (1996). *J. Res. Natl Inst. Stand. Technol.* **101**, 221–225.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supplementary materials

1,4-Diazoniabicyclo[2.2.2]octane terephthalate

E. Yang, X.-C. Song and J.-W. Zhu

Comment

The asymmetric unit of the title compound, (I), consists of a protonated 1,4-diazoniabicyclo[2.2.2]octane cation, $C_6H_{14}N_2^{2+}$, and a deprotonated terephthalate anion, $C_8H_4O_4^{2-}$, (Figure 1). Single N—H···O hydrogen bond was formed between the protonated N end of the cation and the deprotonated carboxylate group of the anion, which generates the hydrogen bonding chains (Table 1 & Figure 2).

Experimental

A mixture of 1,4-diazoniabicyclo[2.2.2]octane (0.072 g), terephthalic acid (0.08 g) and H_2O (10 ml) was sealed in a 25 ml stainless-steel reactor with a Teflon-lined stainless steel reactor and was heated at 373 K for 3 d. On completion of the reaction, the reactor was cooled slowly to room temperature and the mixture was filtered, giving collorless single crystals suitable for X-ray analysis.

Refinement

The H atoms bonded to C atoms were placed at calculated positions at C—H distances 0.93 and 0.97 Å for the aryl and methylene H-atoms, respectively, and $U_{iso}(H) = 1.2U_{eq}(C)$ using a riding model. The H atoms bonded to N atoms were refined freely.

Figures
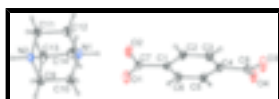

Fig. 1. A view of the title compound, showing 30% probability displacement ellipsoids.
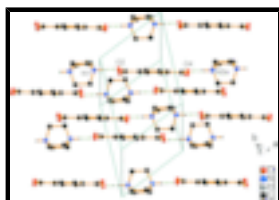

Fig. 2. The unit-cell packing of the title compound, showing the hydrogen bonding chains (symmetry code: a = 2 + x, -1 + y, z).

1,4-Diazoniabicyclo[2.2.2]octane terephthalate

Crystal data

$C_6H_{14}N_2^{2+}\cdot C_8H_4O_4^{2-}$	$Z = 2$
$M_r = 278.30$	$F_{000} = 296$

supplementary materials

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.8046 (10)$ Å

$b = 9.5482 (2)$ Å

$c = 10.8075 (2)$ Å

$\alpha = 65.990 (10)°$

$\beta = 78.436 (10)°$

$\gamma = 66.180 (10)°$

$V = 672.39 (2)$ Å^3

$D_x = 1.375$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 98 reflections

$\theta = 2.1–25.0°$

$\mu = 0.10$ mm^{-1}

$T = 293 (2)$ K

Prism, colorless

$0.23 \times 0.13 \times 0.08$ mm

Data collection

Siemens SMART 1K CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{min} = 0.900$, $T_{max} = 0.950$

7312 measured reflections

2377 independent reflections

1779 reflections with $I > 2\sigma(I)$

$R_{int} = 0.026$

$\theta_{max} = 25.0°$

$\theta_{min} = 2.1°$

$h = -9 \rightarrow 9$

$k = -11 \rightarrow 11$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.125$

$S = 1.04$

2377 reflections

188 parameters

2 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.1475P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.21$ e Å^{-3}

$\Delta\rho_{min} = -0.26$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.023 (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, convention-al 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	0.2883 (2)	0.8953 (2)	0.60534 (16)	0.0691 (5)
O2	0.25555 (18)	0.84559 (17)	0.82616 (14)	0.0489 (4)
O3	1.1805 (2)	0.27977 (19)	0.89737 (15)	0.0629 (5)
O4	1.23265 (18)	0.40326 (18)	0.67751 (14)	0.0519 (4)
N1	−0.0871 (2)	1.03609 (18)	0.77218 (15)	0.0328 (4)
H1N	0.040 (2)	0.964 (2)	0.7898 (18)	0.039*
N2	−0.4239 (2)	1.21700 (18)	0.72154 (14)	0.0317 (4)
H2N	−0.553 (2)	1.285 (2)	0.7069 (18)	0.038*
C1	0.5526 (2)	0.7124 (2)	0.73811 (18)	0.0315 (4)
C2	0.6233 (2)	0.6175 (2)	0.86638 (18)	0.0345 (4)
H2A	0.5455	0.6256	0.9425	0.041*
C3	0.8080 (3)	0.5111 (2)	0.88253 (18)	0.0354 (4)
H3A	0.8524	0.4464	0.9692	0.042*
C4	0.9270 (2)	0.5006 (2)	0.77020 (17)	0.0303 (4)
C5	0.8571 (3)	0.5980 (2)	0.64181 (18)	0.0352 (4)
H5A	0.9362	0.5932	0.5657	0.042*
C6	0.6722 (3)	0.7017 (2)	0.62577 (19)	0.0376 (5)
H6A	0.6273	0.7649	0.5391	0.045*
C7	0.3509 (3)	0.8265 (2)	0.71843 (19)	0.0376 (5)
C8	1.1275 (3)	0.3846 (2)	0.78670 (19)	0.0356 (4)
C9	−0.3943 (3)	1.0480 (2)	0.7369 (2)	0.0389 (5)
H9A	−0.4315	1.0493	0.6559	0.047*
H9B	−0.4709	1.0034	0.8132	0.047*
C10	−0.1869 (3)	0.9399 (2)	0.7601 (2)	0.0407 (5)
H10A	−0.1752	0.8438	0.8422	0.049*
H10B	−0.1322	0.9032	0.6847	0.049*
C11	−0.3778 (3)	1.2165 (3)	0.84800 (19)	0.0419 (5)
H11A	−0.4643	1.1827	0.9213	0.050*
H11B	−0.3897	1.3264	0.8360	0.050*
C12	−0.1769 (3)	1.0977 (2)	0.88353 (18)	0.0377 (5)
H12A	−0.1062	1.1538	0.8965	0.045*
H12B	−0.1781	1.0064	0.9672	0.045*
C13	−0.3010 (3)	1.2820 (2)	0.60835 (19)	0.0404 (5)
H13A	−0.3256	1.3954	0.5939	0.048*
H13B	−0.3262	1.2783	0.5256	0.048*
C14	−0.0959 (3)	1.1773 (2)	0.64345 (19)	0.0411 (5)
H14A	−0.0299	1.1377	0.5711	0.049*
H14B	−0.0356	1.2435	0.6526	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0381 (9)	0.0919 (13)	0.0523 (10)	0.0164 (8)	−0.0141 (7)	−0.0364 (9)
O2	0.0265 (7)	0.0596 (10)	0.0459 (8)	0.0020 (7)	0.0000 (6)	−0.0230 (7)
O3	0.0341 (8)	0.0626 (10)	0.0484 (9)	0.0015 (7)	−0.0034 (7)	0.0032 (8)
O4	0.0259 (7)	0.0630 (10)	0.0413 (8)	0.0009 (7)	0.0016 (6)	−0.0126 (7)
N1	0.0195 (8)	0.0335 (9)	0.0379 (9)	−0.0030 (7)	−0.0017 (6)	−0.0122 (7)
N2	0.0203 (8)	0.0342 (9)	0.0353 (8)	−0.0016 (7)	−0.0034 (6)	−0.0145 (7)
C1	0.0242 (9)	0.0320 (10)	0.0394 (10)	−0.0070 (8)	−0.0002 (8)	−0.0177 (8)
C2	0.0277 (10)	0.0387 (11)	0.0348 (10)	−0.0085 (8)	0.0063 (8)	−0.0181 (8)
C3	0.0316 (10)	0.0363 (10)	0.0305 (9)	−0.0076 (8)	−0.0022 (8)	−0.0091 (8)
C4	0.0248 (9)	0.0272 (9)	0.0360 (10)	−0.0076 (8)	0.0007 (8)	−0.0116 (8)
C5	0.0270 (10)	0.0388 (11)	0.0329 (10)	−0.0059 (8)	0.0043 (8)	−0.0149 (8)
C6	0.0297 (10)	0.0403 (11)	0.0325 (10)	−0.0027 (8)	−0.0048 (8)	−0.0115 (8)
C7	0.0270 (10)	0.0403 (11)	0.0456 (11)	−0.0045 (8)	−0.0033 (9)	−0.0226 (9)
C8	0.0263 (10)	0.0344 (10)	0.0399 (11)	−0.0069 (8)	−0.0030 (8)	−0.0108 (9)
C9	0.0333 (11)	0.0375 (11)	0.0475 (11)	−0.0130 (9)	−0.0045 (9)	−0.0156 (9)
C10	0.0378 (11)	0.0316 (10)	0.0518 (12)	−0.0081 (9)	−0.0050 (9)	−0.0173 (9)
C11	0.0308 (11)	0.0532 (13)	0.0422 (11)	−0.0041 (9)	−0.0019 (8)	−0.0285 (10)
C12	0.0311 (10)	0.0438 (11)	0.0365 (10)	−0.0076 (9)	−0.0059 (8)	−0.0170 (9)
C13	0.0360 (11)	0.0348 (11)	0.0397 (10)	−0.0087 (9)	−0.0027 (8)	−0.0068 (9)
C14	0.0303 (10)	0.0420 (11)	0.0414 (11)	−0.0105 (9)	0.0028 (8)	−0.0104 (9)

Geometric parameters (Å, °)

O1—C7	1.221 (2)	C4—C8	1.503 (2)
O2—C7	1.288 (2)	C5—C6	1.378 (3)
O3—C8	1.222 (2)	C5—H5A	0.9300
O4—C8	1.287 (2)	C6—H6A	0.9300
N1—C10	1.478 (2)	C9—C10	1.533 (3)
N1—C12	1.480 (2)	C9—H9A	0.9700
N1—C14	1.482 (2)	C9—H9B	0.9700
N1—H1N	0.954 (15)	C10—H10A	0.9700
N2—C9	1.477 (2)	C10—H10B	0.9700
N2—C11	1.479 (2)	C11—C12	1.531 (3)
N2—C13	1.481 (2)	C11—H11A	0.9700
N2—H2N	0.959 (15)	C11—H11B	0.9700
C1—C6	1.388 (2)	C12—H12A	0.9700
C1—C2	1.388 (3)	C12—H12B	0.9700
C1—C7	1.507 (2)	C13—C14	1.532 (3)
C2—C3	1.383 (3)	C13—H13A	0.9700
C2—H2A	0.9300	C13—H13B	0.9700
C3—C4	1.385 (2)	C14—H14A	0.9700
C3—H3A	0.9300	C14—H14B	0.9700
C4—C5	1.390 (2)		
C10—N1—C12	109.46 (14)	N2—C9—C10	109.63 (14)

C10—N1—C14	109.60 (14)	N2—C9—H9A	109.7
C12—N1—C14	109.29 (14)	C10—C9—H9A	109.7
C10—N1—H1N	106.8 (11)	N2—C9—H9B	109.7
C12—N1—H1N	111.5 (11)	C10—C9—H9B	109.7
C14—N1—H1N	110.1 (11)	H9A—C9—H9B	108.2
C9—N2—C11	109.82 (14)	N1—C10—C9	109.09 (14)
C9—N2—C13	109.48 (14)	N1—C10—H10A	109.9
C11—N2—C13	109.17 (15)	C9—C10—H10A	109.9
C9—N2—H2N	109.9 (11)	N1—C10—H10B	109.9
C11—N2—H2N	106.9 (11)	C9—C10—H10B	109.9
C13—N2—H2N	111.5 (11)	H10A—C10—H10B	108.3
C6—C1—C2	118.69 (16)	N2—C11—C12	109.40 (14)
C6—C1—C7	119.60 (16)	N2—C11—H11A	109.8
C2—C1—C7	121.71 (16)	C12—C11—H11A	109.8
C3—C2—C1	120.90 (16)	N2—C11—H11B	109.8
C3—C2—H2A	119.6	C12—C11—H11B	109.8
C1—C2—H2A	119.6	H11A—C11—H11B	108.2
C2—C3—C4	120.26 (17)	N1—C12—C11	109.31 (14)
C2—C3—H3A	119.9	N1—C12—H12A	109.8
C4—C3—H3A	119.9	C11—C12—H12A	109.8
C3—C4—C5	118.80 (16)	N1—C12—H12B	109.8
C3—C4—C8	120.66 (16)	C11—C12—H12B	109.8
C5—C4—C8	120.54 (16)	H12A—C12—H12B	108.3
C6—C5—C4	120.92 (16)	N2—C13—C14	108.99 (15)
C6—C5—H5A	119.5	N2—C13—H13A	109.9
C4—C5—H5A	119.5	C14—C13—H13A	109.9
C5—C6—C1	120.40 (17)	N2—C13—H13B	109.9
C5—C6—H6A	119.8	C14—C13—H13B	109.9
C1—C6—H6A	119.8	H13A—C13—H13B	108.3
O1—C7—O2	124.21 (17)	N1—C14—C13	109.66 (14)
O1—C7—C1	120.01 (17)	N1—C14—H14A	109.7
O2—C7—C1	115.77 (16)	C13—C14—H14A	109.7
O3—C8—O4	124.30 (17)	N1—C14—H14B	109.7
O3—C8—C4	120.41 (17)	C13—C14—H14B	109.7
O4—C8—C4	115.28 (16)	H14A—C14—H14B	108.2

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2	0.954 (15)	1.623 (15)	2.5757 (19)	176.8 (17)
N2—H2N···O4^i	0.959 (15)	1.600 (15)	2.5589 (19)	177.9 (18)

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

Fig. 1

Fig. 2