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# 1,4-Diazoniabicyclo[2.2.2]octane terephthalate

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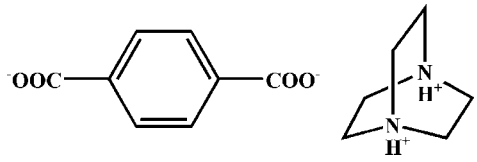
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.044; $wR$ factor = 0.126; data-to-parameter ratio = 12.6.

In the title compound, $C_6H_{14}N^{2+}\cdot C_8H_4O_4^{2-}$, 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, $P\overline{1}$
$a$ = 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 × 0.13 × 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—H1*N*···O2 | 0.954 (15) | 1.623 (15) | 2.5757 (19) | 176.8 (17) |
| N2—H2*N*···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).

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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.

# supporting information

*Acta Cryst.* (2008). E**64**, o1764 [doi:10.1107/S1600536808025312]

## 1,4-Diazoniabicyclo[2.2.2]octane terephthalate

**E Yang, Xu-Chun Song and Jian-Wei Zhu**

### S1. Comment

The asymmetric unit of the title compound, (I), consists of a protonated 1,4-diazoniabicyclo[2.2.2]octane cation, $C_6H_{14}N^{2+}$, 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).

### S2. 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.

### S3. 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) = 1.2$U_{eq}$(C) using a riding model. The H atoms bonded to N atoms were refined freely.

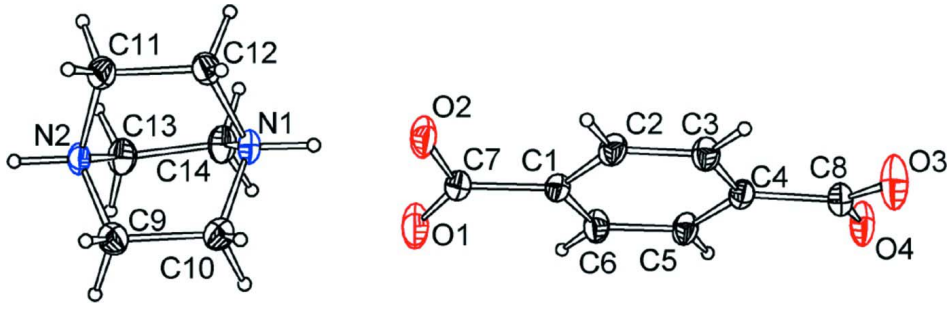


**Figure 1**

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

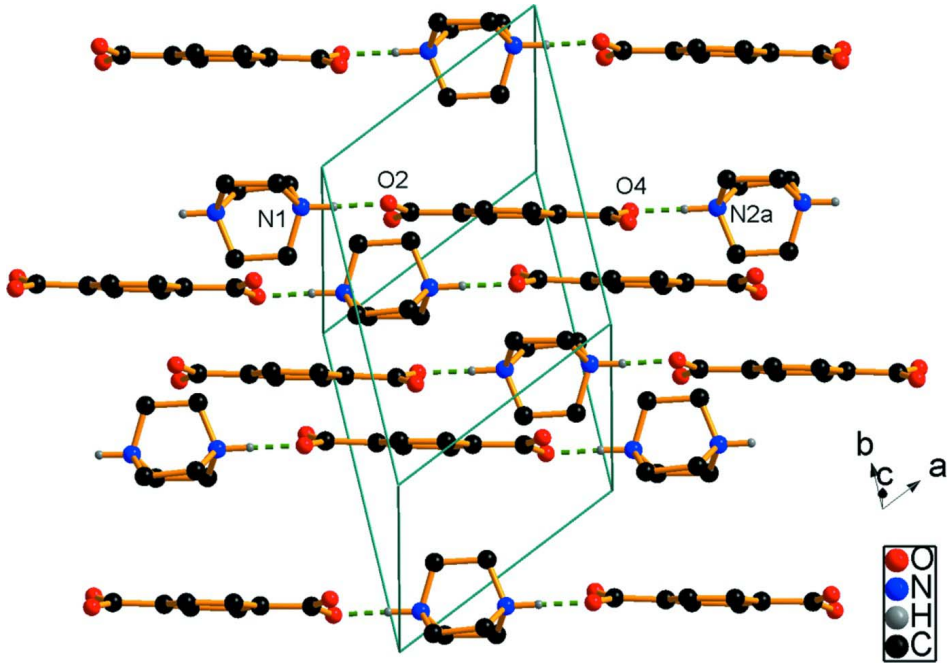


**Figure 2**

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*

$C_6H_{14}N_2^{2+}\cdot C_8H_4O_4^{2-}$
$M_r = 278.30$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.8046\ (10)$ Å
$b = 9.5482\ (2)$ Å
$c = 10.8075\ (2)$ Å
$\alpha = 65.99\ (1)°$
$\beta = 78.436\ (10)°$
$\gamma = 66.18\ (1)°$
$V = 672.39\ (2)$ Å$^3$
$Z = 2$
$F(000) = 296$
$D_x = 1.375$ 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$^{-1}$
$T = 293$ K
Prism, colorless
0.23 × 0.13 × 0.08 mm

*Data collection*

Siemens SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ 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 $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
$h = -9\rightarrow9$
$k = -11\rightarrow11$
$l = -12\rightarrow12$

*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$^{-3}$
$\Delta\rho_{min} = -0.27$ 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 $F^2$, 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 | 0.2883 (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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···O2 | 0.95 (2) | 1.62 (2) | 2.5757 (19) | 177 (2) |
| N2—H2*N*···O4$^{i}$ | 0.96 (2) | 1.60 (2) | 2.5589 (19) | 178 (2) |

Symmetry code: (i) *x*−2, *y*+1, *z*.