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# *tert*-Butyl 6-oxo-2,7-diazaspiro[4.4]-nonane-2-carboxylate

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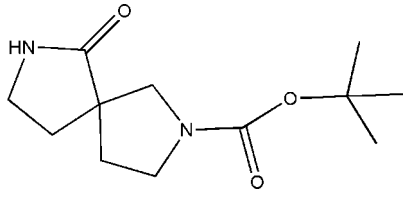
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.050; $wR$ factor = 0.105; data-to-parameter ratio = 9.9.

In the title molecule, $C_{12}H_{20}N_2O_3$, both five-membered rings are in envelope conformations. In the crystal, N—H···O hydrogen bonds link the molecules into chains along [010].

## Related literature

For applications of substituted pyrrolidines, see: Domagala *et al.* (1993); Pedder *et al.* (1976); Blanco & Sardina (1994); Husinec & Savic (2005). For standard bond lengths, see: Allen *et al.* (1987).




## Experimental

### *Crystal data*

$C_{12}H_{20}N_2O_3$
$M_r$ = 240.30
Monoclinic, $C2$
$a$ = 10.495 (5) Å
$b$ = 6.283 (3) Å
$c$ = 19.247 (10) Å
$\beta$ = 97.029 (8)°
$V$ = 1259.7 (11) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 173 K
0.21 × 0.15 × 0.06 mm

### *Data collection*

Rigaku Saturn 724+ diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)
$T_{min}$ = 0.981, $T_{max}$ = 0.995
3265 measured reflections
1557 independent reflections
1452 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.105
$S$ = 1.09
1557 reflections
157 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.88 | 1.97 | 2.848 (3) | 175 |

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

Data collection: *CrystalClear* (Rigaku, 2007); 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5363).

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Blanco, M. J. & Sardina, F. J. (1994). *Tetrahedron Lett.*, **35** , 8493–8396.
Domagala, J. M., Hagan, S. E., Joannides, T., Kiely, J. S., Laborde, E., Schroeder, M. C., Sesnie, J. A., Shapiro, M. A., Suto, M. J. S. & Vanderroest, S. (1993). *J. Med. Chem.* **36**, 871–882.
Husinec, S. & Savic, V. (2005). *Tetrahedron Asymmetry*, **16**, 2047–2061.
Pedder, D. J., Fales, H. M., Jaouni, T., Blum, M., MacConnell, J. & Crewe, R. M. (1976). *Tetrahedron*, **32**, 2275–227.
Rigaku (2007). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

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## *tert*-Butyl 6-oxo-2,7-diazaspiro[4.4]nonane-2-carboxylate

**Jie Yang**

### S1. Comment

Depending on the substitution pattern and functionalization, different substituted pyrrolidines have been shown to be effective antibacterials or fungicides agents and glycosidase inhibitors (Domagala *et al.*, 1993; Pedder *et al.*, 1976; Blanco *et al.*, 1994); Husinec *et al.*, 2005). The crystal structure of the title compound is reportede herein.

In the molecule (Fig. 1), all bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Both five-membered rings are in envelope conformations with C3 and C5 forming the flap. Atoms C6-C8/O2/O3/N2 are essentially planar, with a maximum deviation of 0.0082 (24) Å. In the crystal, N—H···O hydrogen bonds link molecules to form one dimensional chains along [010] (see Table 1).

### S2. Experimental

Tert-butyl 6-oxo-2,7-diazasiro[4.4]nonane-2-carboxylate was synthesized with methyl 1-tert-butyl 3-ethyl 3-(cyano-methyl)pyrrolidine-1,3-dicarboxylate (13.4g) and Raney Ni (3.4g) in methanol under H2(50 Psi) atmosphere at room temperature.

Single crystals of the compound suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature. In the absence of anomalous dispersion effects the Friedel pairs were merged.

### S3. Refinement

All H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances in the range 0.98–0.99 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(C) or $U_{iso}$(H) = 1.5$U_{eq}$($C_{methyl}$). The N—H distance is 0.88 Å, with $U_{iso}$(H) = 1.2$U_{eq}$(N).

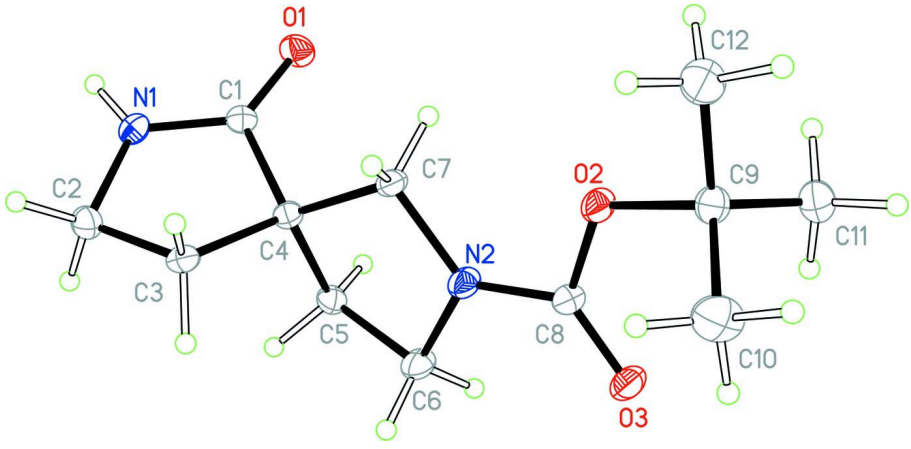

**Figure 1**

The molecular structure of the title compound with displacement ellipsoids are drawn at the 30% probability level.

## *tert*-Butyl 6-oxo-2,7-diazaspiro[4.4]nonane-2-carboxylate

*Crystal data*

$C_{12}H_{20}N_2O_3$
$M_r = 240.30$
Monoclinic, $C2$
Hall symbol: C 2y
$a = 10.495$ (5) Å
$b = 6.283$ (3) Å
$c = 19.247$ (10) Å
$\beta = 97.029$ (8)°
$V = 1259.7$ (11) Å$^3$
$Z = 4$
$F(000) = 520$
$D_x = 1.267$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2422 reflections
$\theta = 1.1$–$27.5°$
$\mu = 0.09$ mm$^{-1}$
$T = 173$ K
Platelet, colorless
0.21 × 0.15 × 0.06 mm

*Data collection*

Rigaku Saturn 724+ diffractometer
Radiation source: rotating anode
Confocal monochromator
$\omega$ scans at fixed $\chi = 45°$
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)
$T_{min} = 0.981$, $T_{max} = 0.995$
3265 measured reflections
1557 independent reflections
1452 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$
$h = -13 \rightarrow 7$
$k = -8 \rightarrow 8$
$l = -23 \rightarrow 25$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.105$
$S = 1.09$
1557 reflections
157 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 0.9713P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ 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. Absolute configuration is unknown, there being no firm chemical evidence for its assignment to hand and it having not been established by anomalous dispersion effects in diffraction measurements on the crystal. An arbitrary choice of enantiomer has been made.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.2715 (2) | 0.4600 (3) | 0.43850 (10) | 0.0313 (5) |
| O2 | 0.33362 (17) | 0.2042 (3) | 0.18571 (9) | 0.0289 (5) |
| O3 | 0.55006 (19) | 0.1382 (4) | 0.19625 (10) | 0.0332 (5) |
| N1 | 0.3406 (2) | 0.8081 (4) | 0.44345 (11) | 0.0255 (5) |
| H1 | 0.3028 | 0.8474 | 0.4798 | 0.031* |
| N2 | 0.4618 (2) | 0.3546 (4) | 0.27139 (12) | 0.0275 (5) |
| C1 | 0.3315 (3) | 0.6117 (5) | 0.41741 (13) | 0.0219 (6) |
| C2 | 0.4181 (3) | 0.9527 (5) | 0.40704 (14) | 0.0279 (6) |
| H2B | 0.5037 | 0.9751 | 0.4339 | 0.033* |
| H2A | 0.3750 | 1.0920 | 0.3984 | 0.033* |
| C3 | 0.4286 (3) | 0.8347 (5) | 0.33812 (13) | 0.0234 (6) |
| H3B | 0.5134 | 0.8596 | 0.3220 | 0.028* |
| H3A | 0.3605 | 0.8813 | 0.3011 | 0.028* |
| C4 | 0.4118 (3) | 0.5993 (5) | 0.35651 (13) | 0.0209 (5) |
| C5 | 0.5419 (3) | 0.4915 (5) | 0.38093 (13) | 0.0241 (6) |
| H5B | 0.6051 | 0.5963 | 0.4027 | 0.029* |
| H5A | 0.5317 | 0.3766 | 0.4150 | 0.029* |
| C6 | 0.5835 (3) | 0.4018 (5) | 0.31360 (15) | 0.0303 (7) |
| H6B | 0.6336 | 0.5078 | 0.2902 | 0.036* |
| H6A | 0.6357 | 0.2714 | 0.3230 | 0.036* |
| C7 | 0.3515 (2) | 0.4573 (5) | 0.29695 (13) | 0.0233 (6) |
| H7B | 0.2936 | 0.3505 | 0.3143 | 0.028* |
| H7A | 0.3024 | 0.5428 | 0.2596 | 0.028* |
| C8 | 0.4568 (2) | 0.2247 (5) | 0.21527 (13) | 0.0244 (6) |
| C9 | 0.3028 (3) | 0.0912 (5) | 0.11865 (14) | 0.0290 (7) |
| C10 | 0.3776 (3) | 0.1875 (7) | 0.06389 (15) | 0.0461 (9) |
| H10A | 0.3444 | 0.1321 | 0.0176 | 0.069* |
| H10C | 0.4686 | 0.1501 | 0.0745 | 0.069* |
| H10B | 0.3683 | 0.3427 | 0.0641 | 0.069* |
| C11 | 0.3283 (3) | −0.1464 (6) | 0.12960 (17) | 0.0381 (8) |
| H11B | 0.2717 | −0.2031 | 0.1620 | 0.057* |
| H11C | 0.4180 | −0.1680 | 0.1492 | 0.057* |
| H11A | 0.3117 | −0.2205 | 0.0846 | 0.057* |
| C12 | 0.1607 (3) | 0.1350 (7) | 0.10196 (17) | 0.0419 (8) |
| H12A | 0.1281 | 0.0648 | 0.0579 | 0.063* |
| H12C | 0.1466 | 0.2888 | 0.0974 | 0.063* |
| H12B | 0.1154 | 0.0799 | 0.1398 | 0.063* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0348 (12) | 0.0317 (11) | 0.0293 (10) | −0.0073 (10) | 0.0115 (8) | 0.0027 (10) |
| O2 | 0.0228 (10) | 0.0378 (11) | 0.0253 (9) | 0.0021 (10) | 0.0002 (7) | −0.0106 (10) |
| O3 | 0.0261 (11) | 0.0410 (13) | 0.0335 (10) | 0.0050 (10) | 0.0074 (8) | −0.0098 (10) |
| N1 | 0.0263 (12) | 0.0276 (13) | 0.0234 (11) | 0.0030 (11) | 0.0066 (9) | −0.0023 (11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N2 | 0.0190 (11) | 0.0345 (13) | 0.0284 (11) | 0.0033 (11) | 0.0004 (9) | −0.0091 (11) |
| C1 | 0.0200 (13) | 0.0256 (13) | 0.0200 (11) | −0.0001 (12) | 0.0015 (10) | 0.0016 (11) |
| C2 | 0.0280 (15) | 0.0235 (13) | 0.0322 (14) | 0.0000 (13) | 0.0033 (11) | 0.0014 (13) |
| C3 | 0.0197 (13) | 0.0265 (14) | 0.0247 (12) | 0.0022 (12) | 0.0059 (10) | 0.0037 (12) |
| C4 | 0.0193 (12) | 0.0224 (13) | 0.0211 (11) | 0.0013 (12) | 0.0025 (10) | 0.0009 (11) |
| C5 | 0.0206 (13) | 0.0249 (14) | 0.0262 (13) | −0.0010 (12) | 0.0001 (10) | −0.0014 (12) |
| C6 | 0.0183 (13) | 0.0383 (18) | 0.0336 (14) | 0.0040 (13) | 0.0001 (11) | −0.0076 (13) |
| C7 | 0.0181 (13) | 0.0271 (13) | 0.0252 (12) | 0.0021 (12) | 0.0053 (10) | −0.0021 (12) |
| C8 | 0.0219 (13) | 0.0270 (14) | 0.0248 (13) | 0.0016 (12) | 0.0048 (10) | −0.0003 (12) |
| C9 | 0.0309 (15) | 0.0351 (16) | 0.0212 (13) | −0.0022 (14) | 0.0033 (11) | −0.0045 (13) |
| C10 | 0.048 (2) | 0.062 (3) | 0.0283 (15) | −0.009 (2) | 0.0058 (14) | 0.0031 (17) |
| C11 | 0.0388 (18) | 0.0359 (17) | 0.0394 (17) | −0.0022 (16) | 0.0030 (14) | −0.0081 (15) |
| C12 | 0.0337 (18) | 0.050 (2) | 0.0395 (17) | 0.0044 (17) | −0.0073 (14) | −0.0070 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.238 (3) | C5—C6 | 1.525 (4) |
| O2—C8 | 1.353 (3) | C5—H5B | 0.9900 |
| O2—C9 | 1.474 (3) | C5—H5A | 0.9900 |
| O3—C8 | 1.214 (3) | C6—H6B | 0.9900 |
| N1—C1 | 1.331 (4) | C6—H6A | 0.9900 |
| N1—C2 | 1.454 (4) | C7—H7B | 0.9900 |
| N1—H1 | 0.8800 | C7—H7A | 0.9900 |
| N2—C8 | 1.350 (3) | C9—C12 | 1.511 (4) |
| N2—C6 | 1.458 (4) | C9—C10 | 1.516 (4) |
| N2—C7 | 1.462 (3) | C9—C11 | 1.526 (5) |
| C1—C4 | 1.527 (4) | C10—H10A | 0.9800 |
| C2—C3 | 1.535 (4) | C10—H10C | 0.9800 |
| C2—H2B | 0.9900 | C10—H10B | 0.9800 |
| C2—H2A | 0.9900 | C11—H11B | 0.9800 |
| C3—C4 | 1.536 (4) | C11—H11C | 0.9800 |
| C3—H3B | 0.9900 | C11—H11A | 0.9800 |
| C3—H3A | 0.9900 | C12—H12A | 0.9800 |
| C4—C7 | 1.527 (4) | C12—H12C | 0.9800 |
| C4—C5 | 1.545 (4) | C12—H12B | 0.9800 |
| | | | |
| C8—O2—C9 | 120.7 (2) | N2—C6—H6A | 111.1 |
| C1—N1—C2 | 114.6 (2) | C5—C6—H6A | 111.1 |
| C1—N1—H1 | 122.7 | H6B—C6—H6A | 109.1 |
| C2—N1—H1 | 122.7 | N2—C7—C4 | 103.8 (2) |
| C8—N2—C6 | 121.0 (2) | N2—C7—H7B | 111.0 |
| C8—N2—C7 | 125.5 (2) | C4—C7—H7B | 111.0 |
| C6—N2—C7 | 113.5 (2) | N2—C7—H7A | 111.0 |
| O1—C1—N1 | 127.3 (3) | C4—C7—H7A | 111.0 |
| O1—C1—C4 | 124.3 (3) | H7B—C7—H7A | 109.0 |
| N1—C1—C4 | 108.4 (2) | O3—C8—N2 | 123.9 (3) |
| N1—C2—C3 | 102.6 (2) | O3—C8—O2 | 126.5 (3) |
| N1—C2—H2B | 111.2 | N2—C8—O2 | 109.6 (2) |

| | | | |
|---|---|---|---|
| C3—C2—H2B | 111.2 | O2—C9—C12 | 101.8 (2) |
| N1—C2—H2A | 111.2 | O2—C9—C10 | 109.8 (3) |
| C3—C2—H2A | 111.2 | C12—C9—C10 | 111.2 (3) |
| H2B—C2—H2A | 109.2 | O2—C9—C11 | 109.5 (2) |
| C2—C3—C4 | 104.1 (2) | C12—C9—C11 | 111.1 (3) |
| C2—C3—H3B | 110.9 | C10—C9—C11 | 112.9 (3) |
| C4—C3—H3B | 110.9 | C9—C10—H10A | 109.5 |
| C2—C3—H3A | 110.9 | C9—C10—H10C | 109.5 |
| C4—C3—H3A | 110.9 | H10A—C10—H10C | 109.5 |
| H3B—C3—H3A | 109.0 | C9—C10—H10B | 109.5 |
| C1—C4—C7 | 112.9 (2) | H10A—C10—H10B | 109.5 |
| C1—C4—C3 | 102.6 (2) | H10C—C10—H10B | 109.5 |
| C7—C4—C3 | 116.0 (2) | C9—C11—H11B | 109.5 |
| C1—C4—C5 | 109.8 (2) | C9—C11—H11C | 109.5 |
| C7—C4—C5 | 104.0 (2) | H11B—C11—H11C | 109.5 |
| C3—C4—C5 | 111.7 (2) | C9—C11—H11A | 109.5 |
| C6—C5—C4 | 103.8 (2) | H11B—C11—H11A | 109.5 |
| C6—C5—H5B | 111.0 | H11C—C11—H11A | 109.5 |
| C4—C5—H5B | 111.0 | C9—C12—H12A | 109.5 |
| C6—C5—H5A | 111.0 | C9—C12—H12C | 109.5 |
| C4—C5—H5A | 111.0 | H12A—C12—H12C | 109.5 |
| H5B—C5—H5A | 109.0 | C9—C12—H12B | 109.5 |
| N2—C6—C5 | 103.1 (2) | H12A—C12—H12B | 109.5 |
| N2—C6—H6B | 111.1 | H12C—C12—H12B | 109.5 |
| C5—C6—H6B | 111.1 | | |

| | | | |
|---|---|---|---|
| C2—N1—C1—O1 | −179.7 (3) | C7—N2—C6—C5 | 15.7 (3) |
| C2—N1—C1—C4 | 1.7 (3) | C4—C5—C6—N2 | −30.5 (3) |
| C1—N1—C2—C3 | 15.7 (3) | C8—N2—C7—C4 | −175.1 (3) |
| N1—C2—C3—C4 | −25.8 (3) | C6—N2—C7—C4 | 5.9 (3) |
| O1—C1—C4—C7 | 37.5 (4) | C1—C4—C7—N2 | −143.8 (2) |
| N1—C1—C4—C7 | −143.8 (2) | C3—C4—C7—N2 | 98.2 (3) |
| O1—C1—C4—C3 | 163.1 (3) | C5—C4—C7—N2 | −24.9 (3) |
| N1—C1—C4—C3 | −18.2 (3) | C6—N2—C8—O3 | 0.5 (4) |
| O1—C1—C4—C5 | −78.0 (3) | C7—N2—C8—O3 | −178.4 (3) |
| N1—C1—C4—C5 | 100.7 (3) | C6—N2—C8—O2 | 179.2 (3) |
| C2—C3—C4—C1 | 26.7 (3) | C7—N2—C8—O2 | 0.3 (4) |
| C2—C3—C4—C7 | 150.2 (2) | C9—O2—C8—O3 | −8.6 (4) |
| C2—C3—C4—C5 | −90.8 (2) | C9—O2—C8—N2 | 172.7 (2) |
| C1—C4—C5—C6 | 155.7 (2) | C8—O2—C9—C12 | −172.4 (3) |
| C7—C4—C5—C6 | 34.6 (3) | C8—O2—C9—C10 | −54.6 (4) |
| C3—C4—C5—C6 | −91.2 (3) | C8—O2—C9—C11 | 69.9 (3) |
| C8—N2—C6—C5 | −163.3 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.88 | 1.97 | 2.848 (3) | 175 |

Symmetry code: (i) −*x*+1/2, *y*+1/2, −*z*+1.