



Crystal structure of *trans*-cyclohexane-1,2-diammonium chromate(VI) from synchrotron X-ray diffraction data

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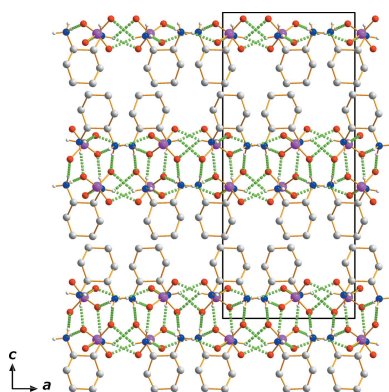
The structure of the title hybrid compound, $(C_6H_{16}N_2)[CrO_4]$, has been determined from synchrotron data. The organic cation adopts a chair conformation. The inorganic CrO_4^{2-} anion is slightly distorted owing to its involvement in $N-H\cdots O$ hydrogen-bonding interactions with neighbouring *trans*-cyclohexane-1,2-diammonium cations, whereby the two Cr—O bonds to the O atoms acting as acceptor atoms for two hydrogen bonds are slightly longer than the other two Cr—O bonds for which only one acceptor interaction per O atom is observed. In the crystal, cations and anions are packed into layers parallel to (001), held together through the aforementioned $N-H\cdots O$ hydrogen bonds.

1. Chemical context

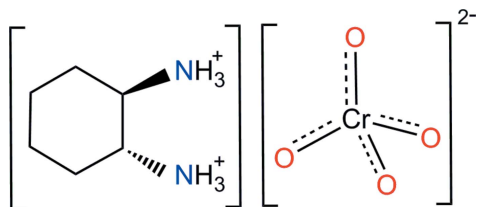
Organic–inorganic hybrid compounds are of interest because of the possibility of their forming extended networks through versatile hydrogen bonds (Mkaouar *et al.*, 2016). The amine *trans*-1,2-cyclohexanediamine (chxn), $C_6H_{14}N_2$, is strongly basic and readily captures two protons to form a dication, $(C_6H_{16}N_2)^{2+}$. Crystal structures of this amine or the dication have been determined for *trans*-1,2-cyclohexanediamine hydrobromide (Morse & Chesick, 1976), *trans*-cyclohexane-1,2-diammonium dichloride (Farrugia *et al.*, 2001) and *trans*-cyclohexane-1,2-diammonium bis(3'-nitro-*trans*-cinnamate) (Hosomi *et al.*, 2000). With respect to complex inorganic anions of the types $ZnCl_4^{2-}$, CrO_4^{2-} or $Cr_2O_7^{2-}$, the crystal structures of hybrid compounds with organic ammonium cations have been determined for propane-1,3-diammonium tetrachloridozincate (Kallel *et al.*, 1980), propane-1,3-diammonium dichromate(VI) (Trabelsi *et al.*, 2012) and propane-1,2-diammonium chromate(VI) (Trabelsi *et al.*, 2014). However, a combination of *trans*-cyclohexane-1,2-diammonium and CrO_4^{2-} has not been reported. In this communication, we present details on the preparation of the new organic chromate(VI), $(C_6H_{16}N_2)[CrO_4]$, (I) and its structural characterization by synchrotron single-crystal X-ray diffraction.

2. Structural commentary

Fig. 1 shows an ellipsoid plot of the molecular components of (I). The organic diammonium cation adopts a stable chair conformation with respect to the cyclohexane ring. The C—C and N—C distances range from 1.506 (5) to 1.525 (4) Å and from 1.492 (3) to 1.493 (3) Å, respectively; the range of N—



C—C and C—C—C angles is 108.3 (2) to 113.7 (2)° and 109.2 (2) to 112.0 (3)°, respectively.



The bond lengths and angles are very similar than in the structure of the bis(3'-nitro-*trans*-cinnamate) compound with the same cation (Hosomi *et al.*, 2000). The cyclohexane ring C—C bond lengths and angles and the torsion angles involving the C and N atoms are in essential agreement with the values obtained for [Cr(chxn)₃](ZnCl₄)Cl·3H₂O (Moon & Choi, 2016). The Cr^{VI} atom in the CrO₄²⁻ anion has the characteristic tetrahedral coordination environment of four O atoms, with Cr—O bond lengths ranging from 1.628 (2) to 1.6654 (19) Å and O—Cr—O angles ranging from 108.30 (10)–111.43 (11)° (Table 1). The distortion from ideal values is due to the influence of hydrogen bonding. For O atoms that are acceptor atoms of two hydrogen bonds (O1 and O4), the Cr—O bond lengths are slightly longer than those of the other two O atoms (O2 and O3) which are each involved in only one hydrogen-bonding interaction.

3. Supramolecular features

In the crystal structure, the cations and anions are arranged in layers parallel to (001). The ammonium group is directed towards the anion, hence causing polar and non-polar sections in the crystal structure, alternating along [001]. As mentioned above, each of the O atoms is involved in N—H···O hydrogen bonds that hold the polar (001) sheets together (Fig. 2, Table 2).

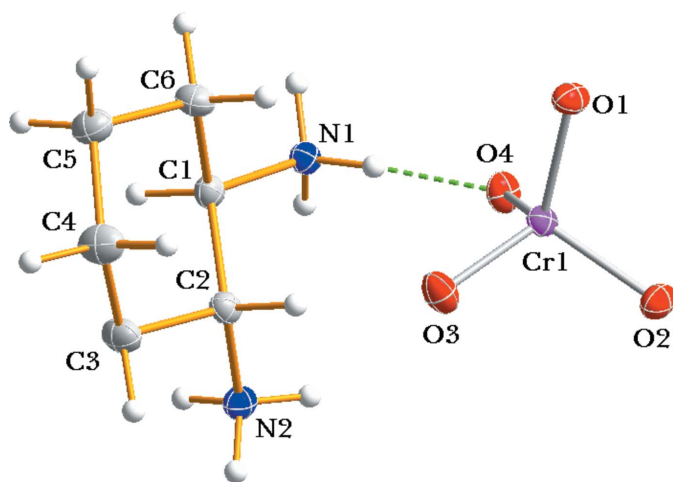


Figure 1
The molecular structures of the organic cation and the inorganic anion in (I), drawn with displacement ellipsoids at the 30% probability level. The dashed line represents a hydrogen-bonding interaction.

Table 1
Selected geometric parameters (Å, °).

Cr1—O3	1.628 (2)	Cr1—O1	1.6584 (19)
Cr1—O2	1.6394 (19)	Cr1—O4	1.6654 (19)
O3—Cr1—O2	108.60 (11)	O3—Cr1—O4	109.76 (10)
O3—Cr1—O1	111.43 (11)	O2—Cr1—O4	108.30 (10)
O2—Cr1—O1	109.72 (10)	O1—Cr1—O4	108.97 (10)

Table 2
Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N1···O2 ⁱ	0.91	1.99	2.896 (3)	172
N1—H3N1···O1 ⁱⁱ	0.91	2.00	2.884 (3)	164
N1—H2N1···O4	0.91	1.81	2.713 (3)	175
N2—H1N2···O4 ⁱ	0.91	1.87	2.771 (3)	169
N2—H3N2···O2 ⁱⁱⁱ	0.91	2.56	3.104 (3)	119
N2—H3N2···O3 ⁱⁱⁱ	0.91	2.04	2.927 (3)	166
N2—H2N2···O1 ^{iv}	0.91	1.86	2.748 (3)	165

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

4. Database survey

A search of the Cambridge Structural Database (Version 5.37, Feb 2016 with three updates; Groom *et al.*, 2016) indicates a total of 31 hits for compounds containing the cyclohexanediammonium cation (C₆H₁₆N₂)²⁺.

5. Synthesis and crystallization

Compound (I) was prepared by dissolving 5 mmol of chromium trioxide (0.50 g, Sigma–Aldrich) and 0.5 mmol of *trans*-

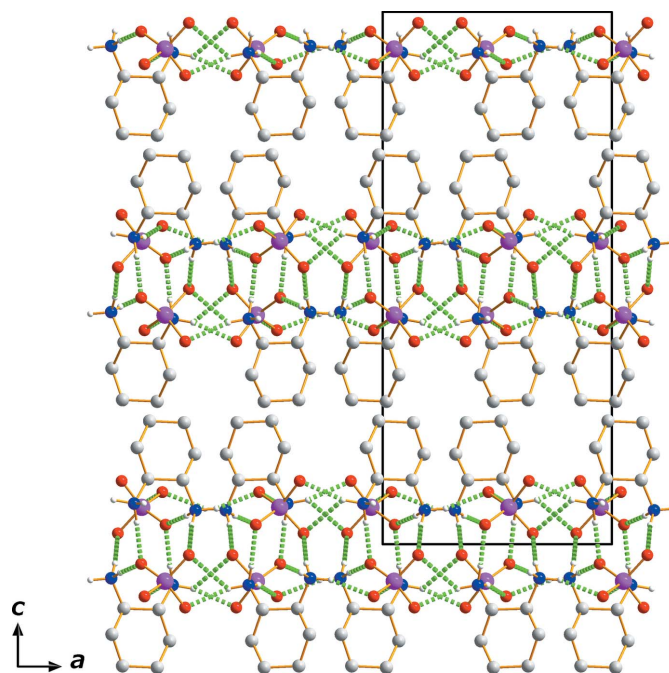


Figure 2
The crystal packing in (I), viewed along [010]. Hydrogen-bonding interactions are indicated by dashed lines.

1,2-cyclohexanediamine (0.6 mL, Sigma-Aldrich) in 40 mL of distilled water with a molar ratio of 1:1. The mixture was stirred for 30 minutes and the resulting solution was allowed to stand at room temperature for one day to give plate-like yellow crystals suitable for X-ray structural analysis.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H = 0.99–1.00 Å and N–H = 0.91 Å, and with $U_{\text{iso}}(\text{H})$ values of 1.2 or $1.5U_{\text{eq}}$ of the parent atoms.

Acknowledgements

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Table 3
Experimental details.

Crystal data	
Chemical formula	(C ₆ H ₁₆ N ₂)[CrO ₄]
M_r	232.21
Crystal system, space group	Orthorhombic, <i>Pbca</i>
Temperature (K)	173
a, b, c (Å)	9.910 (2), 8.3730 (17), 22.999 (5)
V (Å ³)	1908.4 (7)
Z	8
Radiation type	Synchrotron, $\lambda = 0.650$ Å
μ (mm ⁻¹)	0.92
Crystal size (mm)	0.10 × 0.09 × 0.01
Data collection	
Diffractometer	ADSC Q210 CCD area detector
Absorption correction	Empirical (using intensity measurements) (<i>HKL3000sm SCALEPACK</i> ; Otwinowski & Minor, 1997)
$T_{\text{min}}, T_{\text{max}}$	0.794, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16426, 2383, 1749
R_{int}	0.069
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.674
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.055, 0.160, 0.99
No. of reflections	2383
No. of parameters	121
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.95, -1.53

Computer programs: *PAL BL2D-SMDC* (Shin *et al.*, 2016), *HKL3000sm* (Otwinowski & Minor, 1997), *SHELXT2014* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *DIAMOND* (Putz & Brandenburg, 2014) and *publCIF* (Westrip, 2010).

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supporting information

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Crystal structure of *trans*-cyclohexane-1,2-diammonium chromate(VI) from synchrotron X-ray diffraction data

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Computing details

Data collection: *PAL BL2D-SMDC* (Shin *et al.*, 2016); cell refinement: *HKL3000sm* (Otwinowski & Minor, 1997); data reduction: *HKL3000sm* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXT2014* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Putz & Brandenburg, 2014); software used to prepare material for publication: *publCIF* (Westrip, 2010).

trans-Cyclohexane-1,2-diammonium chromate(VI)

Crystal data

(C₆H₁₆N₂)[CrO₄]
 $M_r = 232.21$
 Orthorhombic, *Pbca*
 $a = 9.910$ (2) Å
 $b = 8.3730$ (17) Å
 $c = 22.999$ (5) Å
 $V = 1908.4$ (7) Å³
 $Z = 8$
 $F(000) = 976$

$D_x = 1.616$ Mg m⁻³
 Synchrotron radiation, $\lambda = 0.650$ Å
 Cell parameters from 49521 reflections
 $\theta = 0.4$ – 33.4°
 $\mu = 0.92$ mm⁻¹
 $T = 173$ K
 Plate, yellow
 $0.10 \times 0.09 \times 0.01$ mm

Data collection

ADSC Q210 CCD area detector
 diffractometer
 Radiation source: PLSII 2D bending magnet
 ω scan
 Absorption correction: empirical (using
 intensity measurements)
 (*HKL3000sm Scalepack*; Otwinowski & Minor,
 1997)
 $T_{\min} = 0.794$, $T_{\max} = 1.000$

16426 measured reflections
 2383 independent reflections
 1749 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.069$
 $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 2.5^\circ$
 $h = -12 \rightarrow 12$
 $k = -11 \rightarrow 11$
 $l = -31 \rightarrow 31$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.160$
 $S = 0.99$
 2383 reflections
 121 parameters
 0 restraints
 Hydrogen site location: inferred from
 neighbouring sites

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.116P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.95$ e Å⁻³
 $\Delta\rho_{\min} = -1.53$ e Å⁻³
 Extinction correction: SHELXL2014
 (Sheldrick, 2015b),
 $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.017 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.6842 (2)	0.3903 (3)	0.43614 (9)	0.0245 (5)
H1N1	0.6673	0.3156	0.4639	0.037*
H2N1	0.6444	0.4842	0.4463	0.037*
H3N1	0.7749	0.4049	0.4328	0.037*
N2	0.4159 (3)	0.2284 (3)	0.42353 (10)	0.0279 (5)
H1N2	0.4273	0.2758	0.4588	0.042*
H2N2	0.4575	0.1315	0.4235	0.042*
H3N2	0.3262	0.2151	0.4165	0.042*
C1	0.6285 (3)	0.3349 (3)	0.37930 (11)	0.0249 (6)
H1	0.6622	0.2241	0.3721	0.030*
C2	0.4758 (3)	0.3313 (3)	0.37730 (12)	0.0247 (5)
H2	0.4413	0.4427	0.3823	0.030*
C3	0.4292 (3)	0.2686 (4)	0.31840 (13)	0.0342 (7)
H3A	0.4600	0.1569	0.3137	0.041*
H3B	0.3293	0.2688	0.3171	0.041*
C4	0.4833 (3)	0.3687 (4)	0.26844 (14)	0.0410 (8)
H4A	0.4570	0.3192	0.2310	0.049*
H4B	0.4430	0.4768	0.2701	0.049*
C5	0.6347 (3)	0.3820 (4)	0.27138 (13)	0.0357 (7)
H5A	0.6665	0.4563	0.2408	0.043*
H5B	0.6754	0.2760	0.2638	0.043*
C6	0.6808 (3)	0.4421 (4)	0.33063 (12)	0.0318 (6)
H6A	0.6475	0.5523	0.3366	0.038*
H6B	0.7807	0.4447	0.3318	0.038*
Cr1	0.44912 (4)	0.79269 (5)	0.43056 (2)	0.0227 (2)
O1	0.53896 (18)	0.9403 (2)	0.40203 (9)	0.0308 (5)
O2	0.34508 (19)	0.8641 (2)	0.47950 (9)	0.0330 (5)
O3	0.3616 (2)	0.6998 (2)	0.38110 (10)	0.0362 (5)
O4	0.55315 (18)	0.6648 (2)	0.46324 (9)	0.0311 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0243 (12)	0.0215 (11)	0.0278 (11)	0.0015 (9)	-0.0012 (8)	0.0002 (8)
N2	0.0267 (13)	0.0252 (12)	0.0316 (13)	0.0024 (10)	0.0025 (9)	0.0012 (9)
C1	0.0249 (14)	0.0217 (12)	0.0279 (13)	0.0026 (10)	0.0002 (10)	-0.0011 (10)
C2	0.0257 (14)	0.0195 (12)	0.0289 (13)	0.0000 (10)	-0.0010 (10)	0.0009 (10)
C3	0.0314 (18)	0.0406 (16)	0.0307 (15)	-0.0072 (12)	-0.0047 (11)	-0.0009 (13)
C4	0.0376 (18)	0.055 (2)	0.0309 (15)	-0.0053 (16)	-0.0050 (12)	0.0073 (14)

C5	0.0317 (16)	0.0437 (17)	0.0315 (15)	-0.0018 (13)	0.0024 (11)	0.0029 (12)
C6	0.0298 (16)	0.0322 (14)	0.0334 (15)	-0.0062 (12)	0.0000 (11)	0.0044 (11)
Cr1	0.0223 (3)	0.0167 (3)	0.0290 (3)	0.00140 (14)	-0.00086 (15)	0.00134 (14)
O1	0.0285 (11)	0.0233 (10)	0.0405 (12)	-0.0002 (8)	0.0062 (8)	0.0049 (8)
O2	0.0305 (11)	0.0285 (10)	0.0402 (11)	0.0053 (8)	0.0078 (9)	0.0010 (8)
O3	0.0349 (12)	0.0293 (11)	0.0444 (13)	0.0026 (8)	-0.0129 (10)	-0.0053 (8)
O4	0.0356 (12)	0.0224 (9)	0.0354 (11)	0.0087 (8)	-0.0053 (8)	0.0010 (8)

Geometric parameters (Å, °)

N1—C1	1.493 (3)	C3—H3A	0.9900
N1—H1N1	0.9100	C3—H3B	0.9900
N1—H2N1	0.9100	C4—C5	1.506 (5)
N1—H3N1	0.9100	C4—H4A	0.9900
N2—C2	1.492 (3)	C4—H4B	0.9900
N2—H1N2	0.9100	C5—C6	1.523 (4)
N2—H2N2	0.9100	C5—H5A	0.9900
N2—H3N2	0.9100	C5—H5B	0.9900
C1—C2	1.514 (4)	C6—H6A	0.9900
C1—C6	1.525 (4)	C6—H6B	0.9900
C1—H1	1.0000	Cr1—O3	1.628 (2)
C2—C3	1.525 (4)	Cr1—O2	1.6394 (19)
C2—H2	1.0000	Cr1—O1	1.6584 (19)
C3—C4	1.520 (4)	Cr1—O4	1.6654 (19)
C1—N1—H1N1	109.5	C4—C3—H3B	109.2
C1—N1—H2N1	109.5	C2—C3—H3B	109.2
H1N1—N1—H2N1	109.5	H3A—C3—H3B	107.9
C1—N1—H3N1	109.5	C5—C4—C3	111.0 (3)
H1N1—N1—H3N1	109.5	C5—C4—H4A	109.4
H2N1—N1—H3N1	109.5	C3—C4—H4A	109.4
C2—N2—H1N2	109.5	C5—C4—H4B	109.4
C2—N2—H2N2	109.5	C3—C4—H4B	109.4
H1N2—N2—H2N2	109.5	H4A—C4—H4B	108.0
C2—N2—H3N2	109.5	C4—C5—C6	111.3 (2)
H1N2—N2—H3N2	109.5	C4—C5—H5A	109.4
H2N2—N2—H3N2	109.5	C6—C5—H5A	109.4
N1—C1—C2	113.7 (2)	C4—C5—H5B	109.4
N1—C1—C6	109.5 (2)	C6—C5—H5B	109.4
C2—C1—C6	109.2 (2)	H5A—C5—H5B	108.0
N1—C1—H1	108.1	C5—C6—C1	111.1 (2)
C2—C1—H1	108.1	C5—C6—H6A	109.4
C6—C1—H1	108.1	C1—C6—H6A	109.4
N2—C2—C1	112.8 (2)	C5—C6—H6B	109.4
N2—C2—C3	108.3 (2)	C1—C6—H6B	109.4
C1—C2—C3	109.7 (2)	H6A—C6—H6B	108.0
N2—C2—H2	108.7	O3—Cr1—O2	108.60 (11)
C1—C2—H2	108.7	O3—Cr1—O1	111.43 (11)

C3—C2—H2	108.7	O2—Cr1—O1	109.72 (10)
C4—C3—C2	112.0 (3)	O3—Cr1—O4	109.76 (10)
C4—C3—H3A	109.2	O2—Cr1—O4	108.30 (10)
C2—C3—H3A	109.2	O1—Cr1—O4	108.97 (10)
N1—C1—C2—N2	-57.5 (3)	C2—C3—C4—C5	54.6 (4)
C6—C1—C2—N2	179.8 (2)	C3—C4—C5—C6	-53.4 (4)
N1—C1—C2—C3	-178.3 (2)	C4—C5—C6—C1	56.5 (3)
C6—C1—C2—C3	59.0 (3)	N1—C1—C6—C5	175.7 (2)
N2—C2—C3—C4	178.8 (3)	C2—C1—C6—C5	-59.1 (3)
C1—C2—C3—C4	-57.6 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N1 \cdots O2 ⁱ	0.91	1.99	2.896 (3)	172
N1—H3N1 \cdots O1 ⁱⁱ	0.91	2.00	2.884 (3)	164
N1—H2N1 \cdots O4	0.91	1.81	2.713 (3)	175
N2—H1N2 \cdots O4 ⁱ	0.91	1.87	2.771 (3)	169
N2—H3N2 \cdots O2 ⁱⁱⁱ	0.91	2.56	3.104 (3)	119
N2—H3N2 \cdots O3 ⁱⁱⁱ	0.91	2.04	2.927 (3)	166
N2—H2N2 \cdots O1 ^{iv}	0.91	1.86	2.748 (3)	165

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+3/2, y-1/2, z$; (iii) $-x+1/2, y-1/2, z$; (iv) $x, y-1, z$.