

(4-Chlorobenzohydrazidato- $\kappa^2 N', O$)-[2-(4-chlorobenzoylhydrazono- $\kappa^2 N, O$)-propionato(2-)- κO]oxidovanadium(V)

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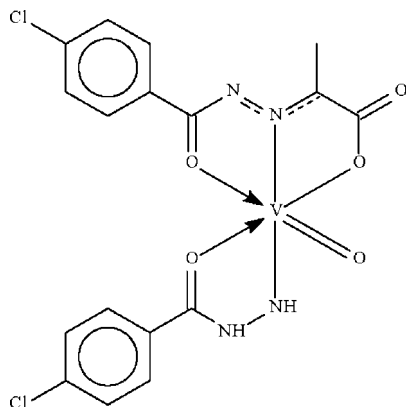
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 Key indicators: single-crystal X-ray study; $T = 119$ K; mean $\sigma(C-C) = 0.007$ Å;
 R factor = 0.059; wR factor = 0.175; data-to-parameter ratio = 17.5.

In the crystal structure of the title compound, $[VO(C_7H_6ClN_2O)(C_{10}H_7ClN_2O_3)]$, the V^V atom is N, O -chelated by the chlorobenzoylhydrazidate anion and O, N, O' -chelated by the (chlorobenzoylhydrazono)propionate dianion. The distorted octahedral $trans-N_2O_4$ coordination geometry is completed by the vanadyl O atom. In the crystal, molecules are linked by $N-H \cdots O$ hydrogen bonds into a linear chain parallel to $[010]$.

Related literature

For the analogous vanadyl complex without the chlorine substituent in the two ligands, see: Wong *et al.* (2009).



Experimental

Crystal data

 $[V(C_7H_6ClN_2O)(C_{10}H_7ClN_2O_3)O]$ $M_r = 475.15$

 Monoclinic, $C2/c$
 $a = 26.628$ (2) Å
 $b = 5.7109$ (3) Å
 $c = 24.772$ (1) Å
 $\beta = 100.396$ (3)°
 $V = 3705.2$ (4) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.86$ mm⁻¹
 $T = 119$ K
 $0.40 \times 0.04 \times 0.04$ mm

Data collection

 Bruker SMART APEX
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{min} = 0.724$, $T_{max} = 0.966$

 11303 measured reflections
 4189 independent reflections
 2154 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.174$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.059$
 $wR(F^2) = 0.175$
 $S = 0.97$
 4189 reflections

 239 parameters
 H-atom parameters constrained
 $\Delta\rho_{max} = 1.40$ e Å⁻³
 $\Delta\rho_{min} = -1.20$ e Å⁻³
Table 1

Selected bond lengths (Å).

V1—N1	2.064 (4)	V1—O3	1.989 (3)
V1—N4	1.888 (4)	V1—O4	2.207 (4)
V1—O1	1.988 (4)	V1—O5	1.593 (4)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N3-H3 \cdots O2^i$	0.88	1.92	2.744 (5)	156
$N4-H4 \cdots O1^i$	0.88	2.14	2.840 (5)	136

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

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2529).

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 Wong, H. W., Lo, K. M. & Ng, S. W. (2009). *Acta Cryst.* **E65**, m422.

supplementary materials

Acta Cryst. (2009). E65, m718 [doi:10.1107/S1600536809019928]

(4-Chlorobenzohydrazidato- κ^2N',O)[2-(4-chlorobenzoylhydrazono- κ^2N,O)propionato(2-)- κO]oxidovanadium(V)

H. W. Wong, K. M. Lo and S. W. Ng

Comment

(type here to add)

Experimental

2-[*p*-Chlorobenzoylhydrazono]propionic acid was prepared from the condensation reaction of *p*-chlorobenzhydrazide and pyruvic acid. The compound (0.70 g, 3 mmol) and vanadyl sulfate (0.25 g, 1.5 mmol) in 20 ml of distilled water were heated for 5 h. Slow evaporation of the filtrate gave orange crystals.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with $U(H)$ set to 1.2 to 1.5 $U(C)$. The nitrogen-bound H-atoms were similarly treated (N—H 0.88 Å).

All phenylene rings were refined as rigid hexagons of 1.39 Å sides.

The final difference Fourier map had a large peak/deep hole in the vicinity of the V1 atom.

Figures

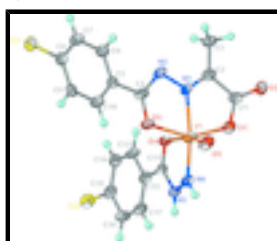


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $VO(C_7H_6ClN_2O)(C_{10}H_7ClN_2O_3)O$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

(4-Chlorobenzohydrazidato- κ^2N',O)[2-(4-chlorobenzoylhydrazono- κ^2N,O)propionato(2-)- κO]oxidovanadium(V)

Crystal data

$[V(C_7H_6ClN_2O)(C_{10}H_7ClN_2O_3)O]$

$M_r = 475.15$

Monoclinic, $C2/c$

$F_{000} = 1920$

$D_x = 1.704 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

supplementary materials

Hall symbol: -C 2yc
 $a = 26.628$ (2) Å
 $b = 5.7109$ (3) Å
 $c = 24.772$ (1) Å
 $\beta = 100.396$ (3)°
 $V = 3705.2$ (4) Å³
 $Z = 8$

$\lambda = 0.71073$ Å
Cell parameters from 957 reflections
 $\theta = 2.5$ – 23.1 °
 $\mu = 0.86$ mm⁻¹
 $T = 119$ K
Prism, orange
 $0.40 \times 0.04 \times 0.04$ mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
 $T = 123$ K
 ω scans
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.724$, $T_{\max} = 0.966$
11303 measured reflections

4189 independent reflections
2154 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.174$
 $\theta_{\max} = 27.5$ °
 $\theta_{\min} = 1.6$ °
 $h = -34 \rightarrow 34$
 $k = -7 \rightarrow 7$
 $l = -32 \rightarrow 30$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.059$
 $wR(F^2) = 0.175$
 $S = 0.97$
4189 reflections
239 parameters
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
 $w = 1/[\sigma^2(F_o^2) + (0.0714P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.40$ e Å⁻³
 $\Delta\rho_{\min} = -1.20$ e Å⁻³
Extinction correction: none

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
V1	0.66692 (3)	0.32434 (15)	0.27882 (4)	0.0216 (3)
Cl1	0.44158 (5)	0.7483 (2)	0.46938 (6)	0.0289 (4)
Cl2	0.84139 (5)	-0.6026 (2)	0.53131 (6)	0.0294 (4)
O1	0.67515 (13)	0.1122 (6)	0.21738 (17)	0.0236 (9)
O2	0.63441 (13)	-0.1245 (6)	0.15164 (17)	0.0281 (9)
O3	0.62899 (12)	0.4195 (6)	0.33748 (16)	0.0237 (9)
O4	0.69433 (12)	0.0349 (6)	0.33560 (16)	0.0222 (9)
O5	0.65919 (13)	0.5607 (6)	0.24415 (18)	0.0296 (10)
N1	0.60013 (15)	0.1326 (7)	0.2650 (2)	0.0203 (10)

N2	0.56469 (15)	0.1678 (7)	0.2986 (2)	0.0236 (11)
N3	0.76603 (15)	0.2341 (7)	0.34041 (19)	0.0206 (10)
H3	0.7990	0.2555	0.3516	0.025*
N4	0.73671 (15)	0.3820 (7)	0.3060 (2)	0.0215 (10)
H4	0.7506	0.5106	0.2958	0.026*
C1	0.63701 (18)	-0.0159 (9)	0.1950 (3)	0.0228 (12)
C2	0.59319 (18)	-0.0213 (9)	0.2266 (2)	0.0225 (12)
C3	0.55037 (18)	-0.1879 (9)	0.2136 (3)	0.0280 (14)
H3A	0.5521	-0.3016	0.2435	0.042*
H3B	0.5526	-0.2702	0.1794	0.042*
H3C	0.5180	-0.1025	0.2092	0.042*
C4	0.58356 (18)	0.3289 (8)	0.3341 (3)	0.0221 (12)
C5	0.55047 (11)	0.4234 (5)	0.37140 (15)	0.0196 (12)
C6	0.50488 (12)	0.3112 (5)	0.37506 (15)	0.0239 (13)
H6	0.4965	0.1671	0.3565	0.029*
C7	0.47159 (10)	0.4100 (5)	0.40592 (17)	0.0249 (13)
H7A	0.4404	0.3333	0.4084	0.030*
C8	0.48390 (11)	0.6209 (5)	0.43310 (15)	0.0227 (12)
C9	0.52949 (12)	0.7331 (5)	0.42944 (15)	0.0239 (12)
H9	0.5379	0.8772	0.4480	0.029*
C10	0.56278 (10)	0.6343 (5)	0.39859 (16)	0.0254 (13)
H10	0.5939	0.7110	0.3961	0.031*
C11	0.74052 (18)	0.0506 (8)	0.3562 (2)	0.0186 (12)
C12	0.76740 (11)	-0.1137 (5)	0.39758 (13)	0.0222 (13)
C13	0.73990 (8)	-0.3013 (5)	0.41306 (14)	0.0245 (13)
H13	0.7054	-0.3249	0.3958	0.029*
C14	0.76286 (11)	-0.4543 (5)	0.45377 (15)	0.0241 (12)
H14	0.7441	-0.5825	0.4644	0.029*
C15	0.81333 (11)	-0.4198 (5)	0.47899 (13)	0.0251 (13)
C16	0.84083 (9)	-0.2323 (6)	0.46350 (14)	0.0273 (13)
H16	0.8753	-0.2087	0.4807	0.033*
C17	0.81787 (10)	-0.0792 (5)	0.42280 (15)	0.0251 (13)
H17	0.8367	0.0489	0.4122	0.030*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.0156 (4)	0.0218 (5)	0.0292 (7)	0.0012 (4)	0.0087 (4)	0.0014 (4)
Cl1	0.0244 (6)	0.0351 (8)	0.0291 (9)	0.0057 (5)	0.0097 (6)	-0.0082 (6)
Cl2	0.0273 (7)	0.0282 (7)	0.0332 (10)	0.0029 (6)	0.0066 (6)	0.0046 (7)
O1	0.0189 (17)	0.0246 (19)	0.028 (2)	0.0046 (15)	0.0074 (17)	0.0015 (17)
O2	0.0217 (18)	0.040 (2)	0.024 (3)	0.0046 (17)	0.0079 (17)	-0.004 (2)
O3	0.0151 (16)	0.0256 (19)	0.031 (3)	-0.0022 (14)	0.0067 (17)	-0.0060 (18)
O4	0.0150 (16)	0.0225 (18)	0.029 (3)	-0.0032 (14)	0.0048 (17)	-0.0022 (17)
O5	0.0269 (19)	0.025 (2)	0.039 (3)	0.0037 (16)	0.013 (2)	0.0060 (19)
N1	0.018 (2)	0.019 (2)	0.027 (3)	0.0043 (17)	0.011 (2)	0.003 (2)
N2	0.018 (2)	0.024 (2)	0.032 (3)	0.0009 (18)	0.014 (2)	-0.004 (2)
N3	0.0137 (19)	0.025 (2)	0.023 (3)	0.0018 (17)	0.0037 (18)	0.005 (2)

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N4	0.020 (2)	0.018 (2)	0.028 (3)	-0.0018 (17)	0.009 (2)	0.000 (2)
C1	0.016 (2)	0.020 (3)	0.033 (4)	0.003 (2)	0.004 (2)	-0.001 (3)
C2	0.016 (2)	0.021 (3)	0.030 (4)	0.004 (2)	0.004 (2)	0.003 (3)
C3	0.018 (2)	0.032 (3)	0.036 (4)	-0.004 (2)	0.010 (3)	-0.007 (3)
C4	0.018 (2)	0.017 (2)	0.032 (4)	0.000 (2)	0.005 (2)	0.004 (3)
C5	0.019 (2)	0.020 (3)	0.021 (3)	0.002 (2)	0.006 (2)	-0.003 (2)
C6	0.022 (2)	0.021 (3)	0.029 (4)	-0.003 (2)	0.006 (2)	-0.007 (3)
C7	0.021 (3)	0.021 (3)	0.034 (4)	0.000 (2)	0.009 (3)	-0.001 (3)
C8	0.022 (2)	0.023 (3)	0.023 (3)	0.005 (2)	0.006 (2)	-0.004 (2)
C9	0.026 (3)	0.026 (3)	0.019 (3)	0.000 (2)	0.003 (2)	-0.005 (2)
C10	0.017 (2)	0.028 (3)	0.033 (4)	-0.001 (2)	0.006 (2)	-0.003 (3)
C11	0.017 (2)	0.022 (3)	0.017 (3)	-0.003 (2)	0.005 (2)	-0.004 (2)
C12	0.019 (2)	0.018 (3)	0.031 (4)	0.002 (2)	0.011 (2)	-0.008 (2)
C13	0.016 (2)	0.024 (3)	0.036 (4)	-0.003 (2)	0.011 (2)	-0.006 (3)
C14	0.026 (3)	0.024 (3)	0.023 (3)	-0.002 (2)	0.006 (2)	0.000 (3)
C15	0.023 (3)	0.027 (3)	0.027 (4)	0.007 (2)	0.009 (3)	0.002 (3)
C16	0.014 (2)	0.032 (3)	0.035 (4)	-0.001 (2)	0.003 (2)	0.003 (3)
C17	0.023 (3)	0.024 (3)	0.029 (4)	-0.003 (2)	0.007 (3)	0.005 (3)

Geometric parameters (\AA , $^\circ$)

V1—N1	2.064 (4)	C3—H3C	0.9800
V1—N4	1.888 (4)	C4—C5	1.489 (5)
V1—O1	1.988 (4)	C5—C6	1.3900
V1—O3	1.989 (3)	C5—C10	1.3900
V1—O4	2.207 (4)	C6—C7	1.3900
V1—O5	1.593 (4)	C6—H6	0.9500
C11—C8	1.724 (2)	C7—C8	1.3900
C12—C15	1.726 (3)	C7—H7A	0.9500
O1—C1	1.293 (6)	C8—C9	1.3900
O2—C1	1.231 (6)	C9—C10	1.3900
O3—C4	1.305 (5)	C9—H9	0.9500
O4—C11	1.247 (6)	C10—H10	0.9500
N1—C2	1.284 (7)	C11—C12	1.476 (6)
N1—N2	1.381 (5)	C12—C13	1.3900
N2—C4	1.308 (7)	C12—C17	1.3900
N3—N4	1.344 (6)	C13—C14	1.3900
N3—C11	1.345 (6)	C13—H13	0.9500
N3—H3	0.8800	C14—C15	1.3900
N4—H4	0.8800	C14—H14	0.9500
C1—C2	1.518 (7)	C15—C16	1.3900
C2—C3	1.475 (7)	C16—C17	1.3900
C3—H3A	0.9800	C16—H16	0.9500
C3—H3B	0.9800	C17—H17	0.9500
O5—V1—N4	93.89 (19)	O3—C4—C5	117.5 (4)
O5—V1—O1	97.17 (18)	N2—C4—C5	118.3 (4)
N4—V1—O1	98.17 (16)	C6—C5—C10	120.0
O5—V1—O3	97.41 (17)	C6—C5—C4	119.7 (3)
N4—V1—O3	106.77 (17)	C10—C5—C4	120.1 (3)

O1—V1—O3	150.05 (14)	C5—C6—C7	120.0
O5—V1—N1	109.67 (19)	C5—C6—H6	120.0
N4—V1—N1	156.21 (18)	C7—C6—H6	120.0
O1—V1—N1	76.15 (15)	C8—C7—C6	120.0
O3—V1—N1	74.34 (15)	C8—C7—H7A	120.0
O5—V1—O4	166.80 (18)	C6—C7—H7A	120.0
N4—V1—O4	73.27 (16)	C9—C8—C7	120.0
O1—V1—O4	87.83 (14)	C9—C8—C11	120.02 (18)
O3—V1—O4	83.82 (14)	C7—C8—C11	119.94 (18)
N1—V1—O4	83.36 (15)	C10—C9—C8	120.0
C1—O1—V1	119.1 (3)	C10—C9—H9	120.0
C4—O3—V1	115.6 (3)	C8—C9—H9	120.0
C11—O4—V1	113.3 (3)	C9—C10—C5	120.0
C2—N1—N2	121.6 (4)	C9—C10—H10	120.0
C2—N1—V1	119.4 (3)	C5—C10—H10	120.0
N2—N1—V1	119.0 (3)	O4—C11—N3	116.7 (5)
C4—N2—N1	106.6 (4)	O4—C11—C12	123.9 (4)
N4—N3—C11	114.1 (4)	N3—C11—C12	119.4 (4)
N4—N3—H3	123.0	C13—C12—C17	120.0
C11—N3—H3	123.0	C13—C12—C11	117.9 (3)
N3—N4—V1	122.6 (3)	C17—C12—C11	122.0 (3)
N3—N4—H4	118.7	C12—C13—C14	120.0
V1—N4—H4	118.7	C12—C13—H13	120.0
O2—C1—O1	125.1 (5)	C14—C13—H13	120.0
O2—C1—C2	120.8 (5)	C13—C14—C15	120.0
O1—C1—C2	114.1 (5)	C13—C14—H14	120.0
N1—C2—C3	127.3 (5)	C15—C14—H14	120.0
N1—C2—C1	110.3 (4)	C16—C15—C14	120.0
C3—C2—C1	122.4 (5)	C16—C15—C12	119.67 (19)
C2—C3—H3A	109.5	C14—C15—C12	120.29 (19)
C2—C3—H3B	109.5	C15—C16—C17	120.0
H3A—C3—H3B	109.5	C15—C16—H16	120.0
C2—C3—H3C	109.5	C17—C16—H16	120.0
H3A—C3—H3C	109.5	C16—C17—C12	120.0
H3B—C3—H3C	109.5	C16—C17—H17	120.0
O3—C4—N2	124.2 (5)	C12—C17—H17	120.0
O5—V1—O1—C1	-101.5 (4)	O1—C1—C2—N1	9.8 (6)
N4—V1—O1—C1	163.5 (4)	O2—C1—C2—C3	12.0 (8)
O3—V1—O1—C1	17.1 (6)	O1—C1—C2—C3	-169.0 (5)
N1—V1—O1—C1	7.1 (4)	V1—O3—C4—N2	6.8 (7)
O4—V1—O1—C1	90.8 (4)	V1—O3—C4—C5	-170.4 (3)
O5—V1—O3—C4	104.1 (4)	N1—N2—C4—O3	-4.5 (7)
N4—V1—O3—C4	-159.6 (4)	N1—N2—C4—C5	172.6 (4)
O1—V1—O3—C4	-14.5 (5)	O3—C4—C5—C6	-170.8 (4)
N1—V1—O3—C4	-4.3 (4)	N2—C4—C5—C6	11.9 (6)
O4—V1—O3—C4	-89.1 (4)	O3—C4—C5—C10	15.1 (6)
O5—V1—O4—C11	-14.2 (9)	N2—C4—C5—C10	-162.2 (4)
N4—V1—O4—C11	-0.7 (3)	C10—C5—C6—C7	0.0
O1—V1—O4—C11	98.5 (3)	C4—C5—C6—C7	-174.2 (4)

supplementary materials

O3—V1—O4—C11	-110.3 (3)	C5—C6—C7—C8	0.0
N1—V1—O4—C11	174.8 (4)	C6—C7—C8—C9	0.0
O5—V1—N1—C2	91.9 (4)	C6—C7—C8—C11	177.7 (3)
N4—V1—N1—C2	-79.5 (6)	C7—C8—C9—C10	0.0
O1—V1—N1—C2	-0.8 (4)	C11—C8—C9—C10	-177.7 (3)
O3—V1—N1—C2	-175.6 (4)	C8—C9—C10—C5	0.0
O4—V1—N1—C2	-90.2 (4)	C6—C5—C10—C9	0.0
O5—V1—N1—N2	-90.2 (4)	C4—C5—C10—C9	174.1 (4)
N4—V1—N1—N2	98.4 (5)	V1—O4—C11—N3	-1.1 (6)
O1—V1—N1—N2	177.0 (4)	V1—O4—C11—C12	176.7 (3)
O3—V1—N1—N2	2.2 (3)	N4—N3—C11—O4	3.1 (7)
O4—V1—N1—N2	87.6 (4)	N4—N3—C11—C12	-174.9 (4)
C2—N1—N2—C4	178.1 (5)	O4—C11—C12—C13	2.1 (6)
V1—N1—N2—C4	0.3 (5)	N3—C11—C12—C13	179.9 (3)
C11—N3—N4—V1	-4.0 (6)	O4—C11—C12—C17	-174.7 (4)
O5—V1—N4—N3	179.5 (4)	N3—C11—C12—C17	3.1 (5)
O1—V1—N4—N3	-82.7 (4)	C17—C12—C13—C14	0.0
O3—V1—N4—N3	80.5 (4)	C11—C12—C13—C14	-176.9 (3)
N1—V1—N4—N3	-8.6 (7)	C12—C13—C14—C15	0.0
O4—V1—N4—N3	2.5 (4)	C13—C14—C15—C16	0.0
V1—O1—C1—O2	167.7 (4)	C13—C14—C15—C12	177.6 (3)
V1—O1—C1—C2	-11.3 (6)	C14—C15—C16—C17	0.0
N2—N1—C2—C3	-3.4 (8)	C12—C15—C16—C17	-177.6 (3)
V1—N1—C2—C3	174.5 (4)	C15—C16—C17—C12	0.0
N2—N1—C2—C1	177.9 (4)	C13—C12—C17—C16	0.0
V1—N1—C2—C1	-4.3 (6)	C11—C12—C17—C16	176.8 (3)
O2—C1—C2—N1	-169.2 (5)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3 \cdots O2 ⁱ	0.88	1.92	2.744 (5)	156
N4—H4 \cdots O1 ⁱ	0.88	2.14	2.840 (5)	136

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

Fig. 1

