Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Poly[(μ_6-2-methyl-3,5-dinitrobenzoato)-potassium]

Muhammad Danish,[a]* Iram Saleem,[a] Nazir Ahmad,[a] Wojciech Starosta[b] and Janusz Leciejewicz[b]

[a]Department of Chemistry, University of Sargodha, Sargodha 40100, Pakistan, and [b]Institute of Nuclear Chemistry and Technology, ul. Dorodna 16, 03-195 Warszawa, Poland
Correspondence e-mail: drdanish62@gmail.com

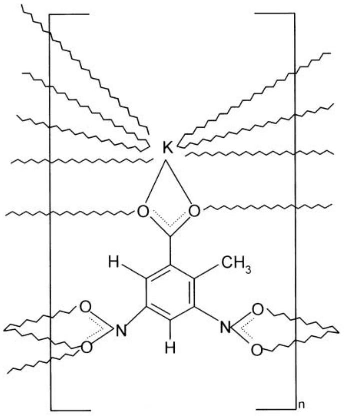
Received 31 March 2010; accepted 26 April 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.049; wR factor = 0.148; data-to-parameter ratio = 18.4.

In the structure of the title coordination polymer, $[K(C_8H_5N_2O_6)]_n$, each ligand bridges six K^+ cations. The carboxylate group coordinates both bidentately to one K^+ ion and monodentately to two K^+ ions, while one nitro group coordinates bidentately to a fourth K^+ ion. The last two K^+ ions are coordinated by the remaining nitro group, one in a bidentate fashion, the other monodentately through one O atom. This bridging mode results in a three-dimensional network. The coordination geometry of the K^+ ion is represented by an irregular KO_9 polyhedron. Very weak C—H⋯O interactions are observed in the crystal structure.

Related literature

Tin complexes with organic ligands have attracted considerable interest due to their biological activity, see, for example: Shahzadi *et al.* (2007). For the structure of a sodium(I) complex with the 2-methyl-3,5-dinitro-benzoate ligand, see: Danish *et al.* (2010).



Experimental

Crystal data

$[K(C_8H_5N_2O_6)]$
M_r = 264.24
Monoclinic, $P2_1/c$
a = 8.1632 (16) Å
b = 16.998 (3) Å
c = 7.0684 (14) Å
β = 90.49 (3)°
V = 980.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.56 mm^{-1}
T = 293 K
0.43 × 0.32 × 0.22 mm

Data collection

Kuma KM-4 four-circle diffractometer
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2008)
T_{min} = 0.889, T_{max} = 0.920
3035 measured reflections
2855 independent reflections
2200 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
3 standard reflections every 200 reflections
intensity decay: 0.7%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.148
S = 1.06
2855 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C6—H6⋯O4^{i}	0.93	2.59	3.518 (2)	174
C8—H81⋯O4^{ii}	0.96	2.84	3.576 (3)	134
C8—H82⋯O2^{iii}	0.96	2.78	3.610 (2)	146

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

Data collection: *KM-4 Software* (Kuma, 1996); cell refinement: *KM-4 Software*; data reduction: *DATAPROC* (Kuma, 2001); 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*.

MD is grateful to the Australian Government for the award of Endeavour Post Doctoral Fellowships for the years 2009–2010.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2207).

References

Danish, M., Saleem, I., Ahmad, N., Raza, A. R., Starosta, W. & Leciejewicz, J. (2010). *Acta Cryst.* E**66**, m137.
Kuma (1996). *KM-4 Software*. Kuma Diffraction Ltd, Wrocław, Poland.
Kuma (2001). *DATAPROC*. Kuma Diffraction Ltd, Wrocław, Poland.
Oxford Diffraction (2008). *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England
Shahzadi, S., Shahid, K. & Ali, S. (2007). *Russ. J. Coord. Chem.* **33**, 403–411.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, m616   [https://doi.org/10.1107/S1600536810015400]

Poly[(μ_6-2-methyl-3,5-dinitrobenzoato)potassium]

Muhammad Danish, Iram Saleem, Nazir Ahmad, Wojciech Starosta and Janusz Leciejewicz

S1. Comment

Methyl-benzoic acids have been studied as precursors in the synthesis of biologically active tin(IV) complexes (Shahzadi *et al.*, 2007). The structure of compound (1) is a three-dimensional polymeric network in which K^+ ions are bridged by carboxylate and nitro-group O atoms of the ligand (Fig. 1). The ligand's carboxylate group coordinates bidentately to K1. Its oxygen atoms also coordinate to $K1^{(i)}$ and $K1^{(ii)}$ [symmetry codes: (i) x,-y-3/2,z-1/2; (ii) x,-y+3/2,z+1/2]. The planes formed by atoms $K1/O1/K1^{(i)}/O2^{(i)}$ and $K1/O2/K1^{(ii)}/O1^{(ii)}$, each with s.u.s of 0.1326 (2) Å, make angles of 8.7 (1)° with the C7/O1/O2 plane forming a zig-zag molecular ribbon. A three-dimensional network (Fig. 2) composed of the ribbons inter-connected by nitro-groups represents the stucture of the title compound. The N1/O3/O4 nitro-group coordinates bidentately to $K1^{(vii)}$; N2/O5/O6 is chelated to the $K1^{(vi)}$, however, the O6 atom is also linked to $K1^{(iv)}$. The carboxylic group C7/O1/O2 makes an angle of 38.0 (1)° to the methylbenzene ring, while the nitro-groups N1/O3/O4 and N2/O5/O6 are oriented at angles of 6.7 (1)° and 35.5 (1)°, respectively. K1 is nine-coordinate with a complicated geometry, while the coordination environment of a Na(I) ion in the complex with the same ligand consists of seven O atoms (Danish *et al.*, 2010). Very weak interactions of the C—H···O type are also operating.

S2. Experimental

50 ml of aqueous solution containing 0.008 mol of 2-methyl-3,5-dinitro benzoic acid was added dropwise to 50 ml of an aqueous solution of potassium hydroxide (0.008 mol) with constant stirring at room temperature. The mixture was refluxed for 3 hours, then brought to room temperature and concentrated under reduced pressure. A brown solid was purified by repeated crystallization from ethanol-ethyl acetate (1:1) mixture to obtain brown single crystals.

S3. Refinement

H atoms attached to methyl and benzene-ring C atoms were positioned geometrically (C–H = 0.95–0.98 Å) and refined as riding with $U_{iso}(H) = 1.2–1.5U_{eq}(C)$.

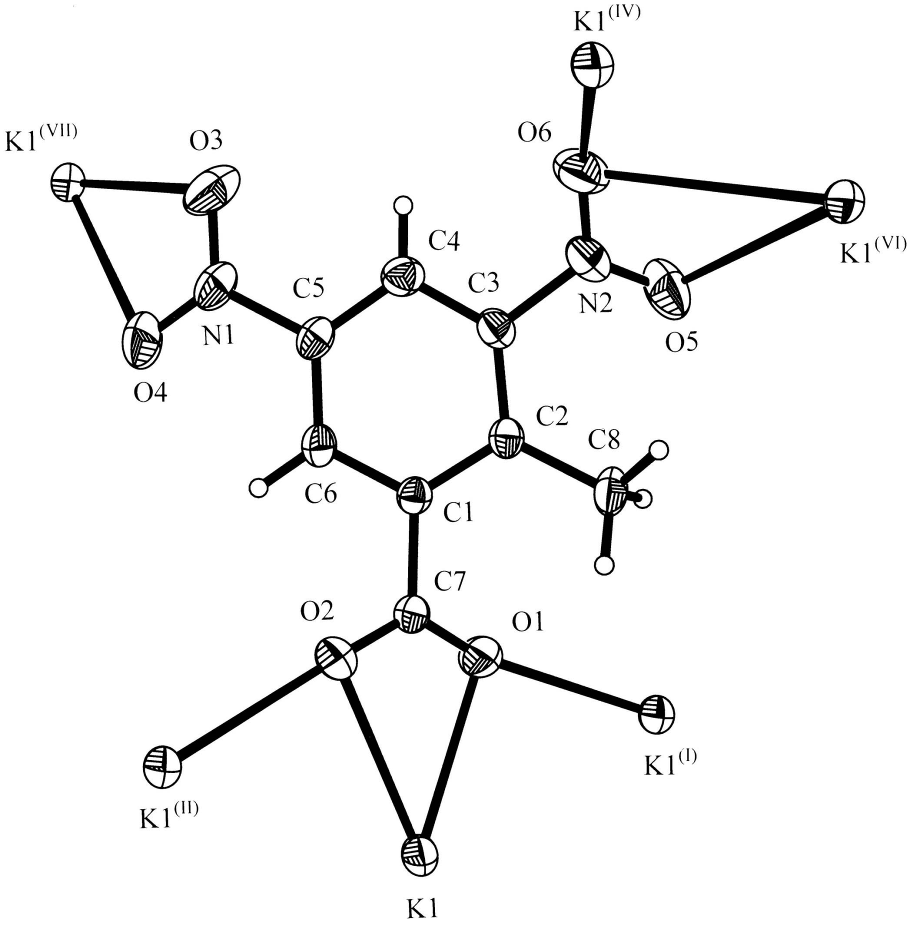


Figure 1

A structural unit of (1) with atom labelling scheme and 50% probability displacement ellipsoids. Symmetry codes: (i) x,-y-3/2,z-1/2; (ii) x,-y+3/2,z+1/2; (iii) -x,y-1/2,-z+3/2; (iv) -x+1,-y+2,-z+1; (v) -x+1,y-1/2,-z+1/2; (vi) -x+1,y+1/2;-z+1/2; (vii) -x,y+1/2,-z+3/2.

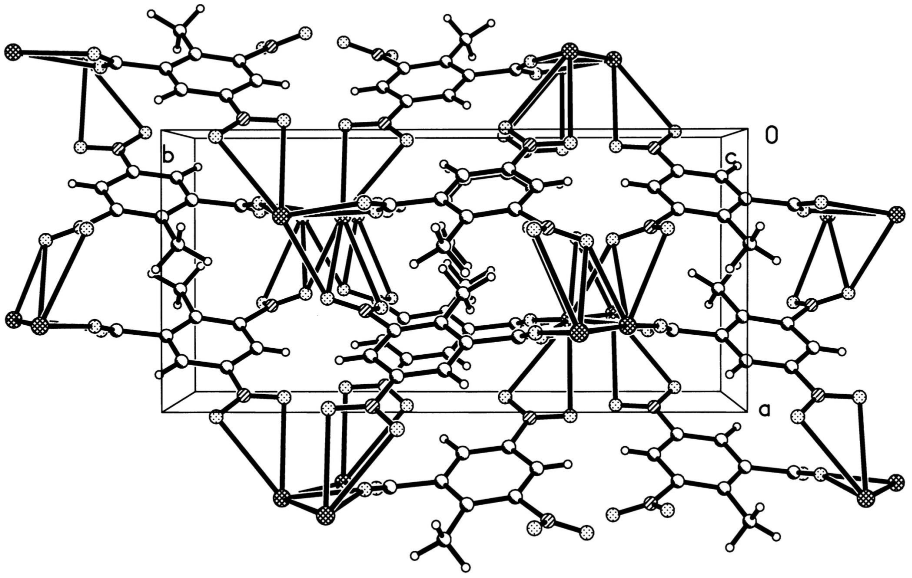


Figure 2

Packing diagram of the structure.

Poly[(μ_6-2-methyl-3,5-dinitrobenzoato)potassium]

Crystal data

$[K(C_8H_5N_2O_6)]$
$M_r = 264.24$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.1632\ (16)$ Å
$b = 16.998\ (3)$ Å
$c = 7.0684\ (14)$ Å
$\beta = 90.49\ (3)°$
$V = 980.7\ (3)$ Å^3
$Z = 4$

$F(000) = 536$
$D_x = 1.790$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 6$–$15°$
$\mu = 0.56$ mm^{-1}
$T = 293$ K
Block, brown
0.43 × 0.32 × 0.22 mm

Data collection

Kuma KM-4 four-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
profile data from $\omega/2\theta$ scans
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2008)
$T_{\min} = 0.889$, $T_{\max} = 0.920$
3035 measured reflections

2855 independent reflections
2200 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 30.1°$, $\theta_{\min} = 2.4°$
$h = -11 \rightarrow 0$
$k = 0 \rightarrow 23$
$l = -9 \rightarrow 9$
3 standard reflections every 200 reflections
intensity decay: 0.7%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.148$
$S = 1.06$
2855 reflections
155 parameters

0 restraints
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	$(\Delta/\sigma)_{max} = 0.001$
$w = 1/[\sigma^2(F_o^2) + (0.1144P)^2 + 0.0549P]$ where $P = (F_o^2 + 2F_c^2)/3$	$\Delta\rho_{max} = 0.72$ e Å^{-3}
	$\Delta\rho_{min} = -0.72$ e Å^{-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.

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}
K1	0.30646 (5)	0.70455 (2)	0.53719 (5)	0.03281 (15)
C1	0.24609 (16)	0.97819 (8)	0.52276 (19)	0.0220 (3)
C7	0.26751 (18)	0.88921 (9)	0.5373 (2)	0.0246 (3)
C2	0.30932 (17)	1.02265 (9)	0.3727 (2)	0.0233 (3)
C6	0.16004 (17)	1.01430 (9)	0.6686 (2)	0.0255 (3)
H6	0.1179	0.9844	0.7670	0.031*
C3	0.28318 (19)	1.10416 (9)	0.3816 (2)	0.0266 (3)
O1	0.25937 (19)	0.85008 (8)	0.38860 (17)	0.0395 (3)
O6	0.39480 (19)	1.22199 (8)	0.2829 (2)	0.0471 (4)
O2	0.28968 (18)	0.86252 (8)	0.69897 (17)	0.0373 (3)
N1	0.04619 (18)	1.13215 (9)	0.8188 (2)	0.0346 (3)
N2	0.34771 (18)	1.15683 (9)	0.2343 (2)	0.0334 (3)
C5	0.13803 (18)	1.09462 (9)	0.6657 (2)	0.0270 (3)
O5	0.3523 (2)	1.13326 (10)	0.0711 (2)	0.0526 (4)
C8	0.4096 (2)	0.98522 (10)	0.2191 (2)	0.0319 (3)
H81	0.3405	0.9739	0.1120	0.048*
H83	0.4573	0.9373	0.2656	0.048*
H82	0.4951	1.0207	0.1819	0.048*
C4	0.20004 (19)	1.14170 (10)	0.5248 (2)	0.0295 (3)
H4	0.1866	1.1960	0.5261	0.035*
O3	0.0391 (2)	1.20350 (9)	0.8230 (3)	0.0503 (4)
O4	−0.0210 (2)	1.08967 (10)	0.9335 (2)	0.0533 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0473 (3)	0.0273 (2)	0.0239 (2)	−0.00228 (13)	0.00711 (15)	0.00005 (11)
C1	0.0202 (6)	0.0239 (6)	0.0219 (6)	0.0009 (5)	0.0005 (5)	−0.0003 (5)
C7	0.0229 (6)	0.0250 (7)	0.0259 (7)	0.0011 (5)	0.0041 (5)	0.0009 (5)
C2	0.0202 (6)	0.0269 (7)	0.0228 (6)	−0.0014 (5)	0.0016 (5)	−0.0004 (5)
C6	0.0217 (6)	0.0314 (8)	0.0234 (6)	0.0004 (5)	0.0042 (5)	0.0004 (5)
C3	0.0250 (7)	0.0270 (7)	0.0280 (7)	−0.0014 (5)	0.0026 (5)	0.0045 (5)

O1	0.0608 (9)	0.0291 (6)	0.0285 (6)	0.0017 (6)	0.0010 (5)	−0.0041 (5)
O6	0.0453 (8)	0.0310 (7)	0.0650 (10)	−0.0058 (5)	0.0100 (7)	0.0082 (6)
O2	0.0520 (8)	0.0329 (6)	0.0270 (6)	0.0053 (5)	0.0035 (5)	0.0063 (5)
N1	0.0266 (7)	0.0394 (8)	0.0381 (7)	0.0041 (5)	0.0069 (5)	−0.0106 (6)
N2	0.0282 (6)	0.0336 (7)	0.0385 (7)	−0.0012 (5)	0.0023 (6)	0.0121 (6)
C5	0.0212 (6)	0.0312 (7)	0.0288 (7)	0.0029 (5)	0.0038 (5)	−0.0050 (6)
O5	0.0650 (10)	0.0608 (10)	0.0321 (7)	−0.0112 (8)	0.0014 (7)	0.0126 (6)
C8	0.0306 (7)	0.0386 (9)	0.0268 (7)	−0.0033 (6)	0.0107 (6)	−0.0039 (6)
C4	0.0261 (7)	0.0262 (7)	0.0361 (8)	0.0022 (5)	0.0028 (6)	−0.0002 (6)
O3	0.0412 (8)	0.0410 (8)	0.0688 (10)	−0.0005 (5)	0.0146 (7)	−0.0228 (7)
O4	0.0585 (10)	0.0587 (9)	0.0432 (8)	0.0134 (7)	0.0271 (7)	0.0030 (7)

Geometric parameters (Å, º)

K1—O2^{i}	2.6511 (13)	C3—C4	1.380 (2)
K1—O1^{ii}	2.6826 (13)	C3—N2	1.473 (2)
K1—O1	2.7133 (14)	O1—K1^{i}	2.6826 (13)
K1—O2	2.9221 (14)	O6—N2	1.221 (2)
K1—O3^{iii}	2.9974 (18)	O6—K1^{iv}	3.0116 (18)
K1—O6^{iv}	3.0115 (18)	O6—K1^{vi}	3.3541 (19)
K1—O4^{iii}	3.0485 (18)	O2—K1^{ii}	2.6511 (13)
K1—O5^{v}	3.1388 (19)	N1—O3	1.214 (2)
K1—C7	3.1548 (17)	N1—O4	1.220 (2)
K1—N1^{iii}	3.2998 (16)	N1—C5	1.468 (2)
K1—O6^{v}	3.3541 (19)	N1—K1^{vii}	3.2997 (16)
K1—K1^{i}	3.8572 (7)	N2—O5	1.222 (2)
C1—C6	1.395 (2)	C5—C4	1.377 (2)
C1—C2	1.4042 (19)	O5—K1^{vi}	3.1388 (19)
C1—C7	1.526 (2)	C8—H81	0.9600
C7—O2	1.2414 (19)	C8—H83	0.9600
C7—O1	1.2450 (19)	C8—H82	0.9600
C2—C3	1.403 (2)	C4—H4	0.9300
C2—C8	1.506 (2)	O3—K1^{vii}	2.9973 (18)
C6—C5	1.377 (2)	O4—K1^{vii}	3.0485 (17)
C6—H6	0.9300		
O2^{i}—K1—O1^{ii}	132.80 (4)	O3^{iii}—K1—K1^{i}	108.17 (4)
O2^{i}—K1—O1	92.12 (4)	O6^{iv}—K1—K1^{i}	102.38 (4)
O1^{ii}—K1—O1	130.77 (4)	O4^{iii}—K1—K1^{i}	108.96 (4)
O2^{i}—K1—O2	138.23 (4)	O5^{v}—K1—K1^{i}	85.65 (4)
O1^{ii}—K1—O2	87.03 (4)	C7—K1—K1^{i}	66.58 (3)
O1—K1—O2	46.12 (4)	N1^{iii}—K1—K1^{i}	116.03 (4)
O2^{i}—K1—O3^{iii}	104.67 (6)	O6^{v}—K1—K1^{i}	48.76 (3)
O1^{ii}—K1—O3^{iii}	63.25 (5)	C6—C1—C2	120.82 (14)
O1—K1—O3^{iii}	90.14 (4)	C6—C1—C7	116.39 (13)
O2—K1—O3^{iii}	80.16 (5)	C2—C1—C7	122.79 (12)
O2^{i}—K1—O6^{iv}	126.47 (5)	O2—C7—O1	126.02 (16)
O1^{ii}—K1—O6^{iv}	82.79 (5)	O2—C7—C1	116.09 (13)

O1—K1—O6^{iv}	84.09 (5)	O1—C7—C1	117.88 (14)
O2—K1—O6^{iv}	59.62 (4)	O2—C7—K1	67.84 (9)
O3^{iii}—K1—O6^{iv}	128.64 (5)	O1—C7—K1	58.19 (9)
O2^{i}—K1—O4^{iii}	75.65 (5)	C1—C7—K1	176.06 (10)
O1^{ii}—K1—O4^{iii}	66.40 (5)	C3—C2—C1	116.14 (13)
O1—K1—O4^{iii}	120.29 (5)	C3—C2—C8	122.20 (13)
O2—K1—O4^{iii}	121.57 (5)	C1—C2—C8	121.51 (14)
O3^{iii}—K1—O4^{iii}	41.55 (5)	C5—C6—C1	119.43 (14)
O6^{iv}—K1—O4^{iii}	148.60 (5)	C5—C6—H6	120.3
O2^{i}—K1—O5^{v}	69.70 (5)	C1—C6—H6	120.3
O1^{ii}—K1—O5^{v}	103.11 (5)	C4—C3—C2	124.41 (14)
O1—K1—O5^{v}	112.37 (5)	C4—C3—N2	114.73 (15)
O2—K1—O5^{v}	119.66 (5)	C2—C3—N2	120.86 (14)
O3^{iii}—K1—O5^{v}	156.64 (5)	C7—O1—K1^{i}	163.63 (12)
O6^{iv}—K1—O5^{v}	62.99 (5)	C7—O1—K1	98.86 (10)
O4^{iii}—K1—O5^{v}	116.73 (5)	K1^{i}—O1—K1	91.26 (4)
O2^{i}—K1—C7	115.06 (4)	N2—O6—K1^{iv}	138.32 (12)
O1^{ii}—K1—C7	109.20 (4)	N2—O6—K1^{vi}	87.65 (10)
O1—K1—C7	22.95 (4)	K1^{iv}—O6—K1^{vi}	74.37 (4)
O2—K1—C7	23.17 (4)	C7—O2—K1^{ii}	173.37 (12)
O3^{iii}—K1—C7	84.88 (4)	C7—O2—K1	89.00 (10)
O6^{iv}—K1—C7	70.65 (4)	K1^{ii}—O2—K1	87.45 (4)
O4^{iii}—K1—C7	124.07 (5)	O3—N1—O4	123.59 (16)
O5^{v}—K1—C7	118.28 (5)	O3—N1—C5	118.47 (16)
O2^{i}—K1—N1^{iii}	94.55 (5)	O4—N1—C5	117.93 (16)
O1^{ii}—K1—N1^{iii}	56.83 (5)	O3—N1—K1^{vii}	65.06 (9)
O1—K1—N1^{iii}	109.77 (5)	O4—N1—K1^{vii}	67.48 (10)
O2—K1—N1^{iii}	100.25 (4)	C5—N1—K1^{vii}	147.35 (11)
O3^{iii}—K1—N1^{iii}	21.56 (4)	O6—N2—O5	123.42 (15)
O6^{iv}—K1—N1^{iii}	136.87 (4)	O6—N2—C3	117.79 (15)
O4^{iii}—K1—N1^{iii}	21.69 (4)	O5—N2—C3	118.79 (15)
O5^{v}—K1—N1^{iii}	135.22 (5)	C6—C5—C4	122.57 (14)
C7—K1—N1^{iii}	106.43 (4)	C6—C5—N1	119.15 (14)
O2^{i}—K1—O6^{v}	57.45 (4)	C4—C5—N1	118.28 (15)
O1^{ii}—K1—O6^{v}	140.00 (5)	N2—O5—K1^{vi}	97.94 (12)
O1—K1—O6^{v}	76.10 (4)	C2—C8—H81	109.5
O2—K1—O6^{v}	102.69 (4)	C2—C8—H83	109.5
O3^{iii}—K1—O6^{v}	156.18 (5)	H81—C8—H83	109.5
O6^{iv}—K1—O6^{v}	69.96 (4)	C2—C8—H82	109.5
O4^{iii}—K1—O6^{v}	131.53 (4)	H81—C8—H82	109.5
O5^{v}—K1—O6^{v}	38.52 (4)	H83—C8—H82	109.5
C7—K1—O6^{v}	89.20 (4)	C5—C4—C3	116.61 (15)
N1^{iii}—K1—O6^{v}	151.93 (4)	C5—C4—H4	121.7
O2^{i}—K1—K1^{i}	49.19 (3)	C3—C4—H4	121.7
O1^{ii}—K1—K1^{i}	171.19 (3)	N1—O3—K1^{vii}	93.38 (10)

O1—K1—K1^{i}	44.05 (3)	N1—O4—K1^{vii}	90.82 (11)
O2—K1—K1^{i}	89.48 (3)		

Symmetry codes: (i) *x*, −*y*+3/2, *z*−1/2; (ii) *x*, −*y*+3/2, *z*+1/2; (iii) −*x*, *y*−1/2, −*z*+3/2; (iv) −*x*+1, −*y*+2, −*z*+1; (v) −*x*+1, *y*−1/2, −*z*+1/2; (vi) −*x*+1, *y*+1/2, −*z*+1/2; (vii) −*x*, *y*+1/2, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O4^{viii}	0.93	2.59	3.518 (2)	174
C8—H81···O4^{ix}	0.96	2.84	3.576 (3)	134
C8—H82···O2^{iv}	0.96	2.78	3.610 (2)	146

Symmetry codes: (iv) −*x*+1, −*y*+2, −*z*+1; (viii) −*x*, −*y*+2, −*z*+2; (ix) −*x*, −*y*+2, −*z*+1.