



Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

trans-K₃[TcO₂(CN)₄]Sayandev Chatterjee,^a Andrew S. Del Negro,^a Matthew K. Edwards,^a Brendan Twamley,^b Jeanette A. Krause^c and Samuel A. Bryan^{a*}^aRadiochemical Processing Laboratory, Pacific Northwest National Laboratory, Richland, WA 99357, USA, ^bDepartment of Chemistry, University of Idaho, Moscow, ID 83844, USA, and ^cDepartment of Chemistry, University of Cincinnati, Cincinnati, OH 45221-0172, USA

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Received 8 July 2010; accepted 14 July 2010

Key indicators: single-crystal X-ray study; *T* = 90 K; mean $\sigma(\text{N}-\text{C}) = 0.003 \text{ \AA}$; *R* factor = 0.020; *wR* factor = 0.049; data-to-parameter ratio = 16.1.

The structure of the title compound, tripotassium *trans*-tetracyanidodioxidotechnetate(V), is isotypic with its Re analogue. The [TcO₂(CN)₄]³⁻ *trans*-tetracyanidodioxidotechnetate anion has a slightly distorted octahedral configuration. The Tc atom is located on a center of inversion and is bound to two O atoms in axial and to four cyanide ligands in equatorial positions. The Tc—O distance is consistent with a double-bond character. The two potassium cations, one located on a center of inversion and one in a general position, reside in octahedral or tetrahedral environments, respectively. K⁺⋯O and K⁺⋯N interactions occur in the 2.7877 (19)–2.8598 (15) Å range.

Related literature

The isotypic rhenate(V) analogue was reported by Fenn *et al.* (1971) (neutron study) and Murmann & Schlemper (1971) (X-ray study). For further information on dioxidotetracyanido anions of Tc and Re, see: Fackler *et al.* (1985); Kastner *et al.* (1982, 1984); Kremer *et al.* (1997). Luminescence properties of Tc complexes were reported by Del Negro *et al.* (2005, 2006). For further information on hydroxidooxidotetracyanido or aquaoxidotetracyanido anions of Tc and Re, see: Baldas *et al.* (1990); Purcell *et al.* (1989, 1990). For general reviews on technetium structures, see: Bandoli *et al.* (2001, 2006); Bartholoma *et al.* (2010); Tisato *et al.* (1994). Synthetic details were given by Trop *et al.* (1980). For a description of the Cambridge Structural Database, see: Allen (2002).

Experimental

Crystal data

K ₃ [TcO ₂ (CN) ₄]	<i>b</i> = 6.9389 (6) Å
<i>M_r</i> = 351.38	<i>c</i> = 7.4347 (7) Å
Triclinic, <i>P</i> 1̄	α = 108.305 (1)°
<i>a</i> = 6.2539 (6) Å	β = 109.816 (2)°

γ = 104.143 (1)°
V = 265.01 (4) Å³
Z = 1
 Mo *K*α radiation

μ = 2.51 mm⁻¹
T = 90 K
 0.32 × 0.20 × 0.14 mm

Data collection

Bruker SMART APEX diffractometer	3949 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 2007)	1097 independent reflections
<i>T</i> _{min} = 0.490, <i>T</i> _{max} = 0.705	1064 reflections with $> \sigma(I)$
	<i>R</i> _{int} = 0.072

Refinement

<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.020	68 parameters
<i>wR</i> (<i>F</i> ²) = 0.049	$\Delta\rho_{\text{max}} = 0.77 \text{ e \AA}^{-3}$
<i>S</i> = 1.09	$\Delta\rho_{\text{min}} = -0.90 \text{ e \AA}^{-3}$
1097 reflections	

Table 1

Selected geometric parameters (Å, °).

Tc1—O1	1.7721 (12)	N1—C1	1.150 (3)
Tc1—C1	2.1423 (19)	N2—C2	1.151 (3)
Tc1—C2	2.145 (2)		
N1—C1—Tc1	177.71 (16)	N2—C2—Tc1	172.74 (15)

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *SHELXTL*.

We thank Dr Sean E. Hightower and Mr Chuck Z. Soderquist for helpful discussion during the synthesis and crystallization. Financial support was provided by DOE EMSP grant DE-FG02-07ER51629. The *SMART APEX* Diffraction Facility (University of Idaho) was funded by NSF-EPSCoR and the M. J. Murdock Charitable Trust, Vancouver, WA. The Radiochemical Processing and the Environmental Molecular Science Laboratories are national scientific user facilities sponsored by the DOE Office of Biological and Environmental Research and located at Pacific Northwest National Laboratory (PNNL). PNNL is operated by Battelle for the DOE under Contract DE-AC05-76RL01830.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2375).

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supporting information

Acta Cryst. (2010). E66, i61–i62 [https://doi.org/10.1107/S1600536810028205]

***trans*-K₃[TcO₂(CN)₄]**

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S1. Comment

⁹⁹Tc is the most significant long-lived product of uranium fission. In addition to a long half-life (2.13×10⁵ yrs), it is readily water soluble, making it extremely mobile in the environment. This, coupled with its ability to form anionic species, causes major concern when considering long-term disposal of high-level radioactive waste. Thus, it is imperative to provide methods for chemical detection of ⁹⁹Tc. Under normal environmental conditions, ⁹⁹Tc composition is dominated by the pertechnetate anion (TcO₄⁻) which lacks a characteristic spectral signature. This prevents its rapid, sensitive and economic *in situ* detection. In order to address this problem, our research is focused on designing a suitable sensor for the detection of TcO₄⁻. The ultimate aim is to chemically convert the pertechnetate anion to an organic-ligated species that will have a readily characterizable spectral signature. As a first step to address this problem, our group has been able to identify several technetium complexes with long-lived excited states. Thus, the luminescence properties of technetium(V)-dioxidotetrapyridyl and technetium(II)-tris(1,2-bis(dimethylphosphino)ethane) were reported by Del Negro *et al.* (2005, 2006).

However, the significant dearth of Tc structures in general (Bandoli *et al.*, 2001, 2006; Bartholoma *et al.*, 2010; Tisato *et al.*, 1994) has resulted in a substantial knowledge gap in the structures and bonding of technetium complexes, thereby preventing the correlation of the electronic properties with structural parameters. As a representative comparison, the Cambridge Structural Database (CSD, version 5.31; Allen, 2002) currently contains 21 dioxidotechnetium complexes compared to 141 structures reported for dioxidorhenium and dioxidomanganese complexes. In an attempt to bridge this gap, we are focusing on the structural characterization of a series of dioxidotechnetium(V) complexes. Herein, we report the structure of K₃[TcO₂(CN)₄], (I), a tetracyanidodioxidotechnetium(V) salt.

The unit cell of (I) is comprised of three K⁺ cations and one discrete [TcO₂(CN)₄]³⁻ anion. The configuration of the anion is shown in Fig. 1. The Tc atom (located on an inversion center) resides in an octahedral environment defined by two oxido and four cyanido ligands. The *trans* basal C—Tc—C angles are linear resulting in a square planar arrangement of the tetracyanido groups about the Tc center.

The Tc=O distance (1.7721 (12) Å) is virtually identical to the Re=O distance in K₃[ReO₂(CN)₄] [neutron study: 1.773 (8) Å (Fenn *et al.*, 1971), X-ray study: 1.781 (3) Å (Murmman & Schlemper, 1971)]. An average Tc=O distance of 1.74 Å has been observed for an oxido ligand in cations of the type *trans*-[O₂R₄Tc]⁺ (R = 4-*tert*-butylpyridine, imidazole, 1-methylimidazole, trimethylenediamine) (Kastner *et al.*, 1984; Fackler *et al.*, 1985; Kremer *et al.*, 1997) or *trans*-[O₂en₂Tc]⁺ (en = ethylenediamine) (Kastner *et al.*, 1982) while a Tc—O distance of 2.559 (9) Å is observed in the [TcN(CN)₄(OH₂)]²⁻ dianion reported by Baldas *et al.* (1990).

The Tc—C distances (2.1423 (19) and 2.145 (2) Å) determined in this study are slightly longer than reported by Murmann & Schlemper (1971) and Fenn *et al.* (1971) for the [ReO₂(CN)₄]³⁻ anion (Re—C_{ave} = 2.13 Å). In addition, we

see a deviation from linearity for one Tc—C—N angle ($172.74(15)^\circ$ versus $177.71(16)^\circ$). Similar bending is common in the $[\text{ReO}_2(\text{CN})_4]^{3-}$, $[\text{ReO}(\text{OH})(\text{CN})_4]^{2-}$ and $[\text{ReO}(\text{OH}_2)(\text{CN})_4]^-$ anions (ave. 175° versus 178°) (Fenn *et al.*, 1971; Murmann & Schlemper, 1971; Purcell *et al.*, 1989, 1990) and may be a result of inequivalent interactions with the surrounding environment.

(I) shows interionic interactions between the K^+ cations and the $[\text{TcO}_2(\text{CN})_4]^{3-}$ anion. The K^+ cations reside in two distinct environments [K1 located on a special position with inversion symmetry and K2 is located on a general position] (Fig 2). Each oxygen atom of the anion interacts with one K1 and two K2 atoms. The local environment about K1 is distorted octahedral, consisting of the following interactions: $\text{K1}\cdots\text{O1} = 2.8235(13) \text{ \AA}$, $\text{K1}\cdots\text{N1} = 2.8315(18) \text{ \AA}$, $\text{K1}\cdots\text{N2} = 2.8598(15) \text{ \AA}$ and the symmetry equivalents. On the other hand, the local environment about K2 is approximately tetrahedral with interactions of $\text{K2}\cdots\text{O1} = 2.7936(12)$ and $2.8262(14) \text{ \AA}$, $\text{K2}\cdots\text{N1} = 2.8152(16) \text{ \AA}$ and $\text{K2}\cdots\text{N2} = 2.7877(19) \text{ \AA}$. In addition, there are three significantly longer contacts between K2 and N1 ($3.1710(16)$ and $3.5715(16) \text{ \AA}$) and K2 and N2 ($3.1496(17) \text{ \AA}$).

S2. Experimental

(I) is prepared from $[\text{TcO}_2(\text{py})_4]\text{Cl}$ (py = pyridyl) by the method of Trop *et al.* (1980). Complete cyanide substitution of the pyridyl groups of the starting material is achieved by adding an excess of alkaline cyanide.

In a typical preparation, 0.5 g of $[\text{TcO}_2(\text{py})_4]\text{Cl}$ was dissolved in a minimum amount of methanol. Addition of 50 ml of 1.2M KCN in 5:1 (v/v) methanol/water to the above resulted in an immediate green solution which turned yellow in approx. 5 minutes. This was followed by a gradual appearance of a fine yellow precipitate. The mixture was stirred on a hot plate set to low heat for an additional hour to ensure complete conversion. The supernatant was drawn off and the yellow precipitate was washed with diethyl ether. The yellow precipitate was dissolved in a minimal amount of water, followed by slow diffusion of methanol vapors, to yield crystals of (I) suitable for X-ray diffraction.

CAUTION! All syntheses and characterizations were performed with ^{99}Tc , which is a β -emitting isotope with a half-life of 2.13×10^5 years. The handling of small quantities (generally <100 milligrams) of this material does not pose a serious health hazard since common laboratory materials provide adequate shielding. However, radiation safety procedures must be used to prevent contamination!

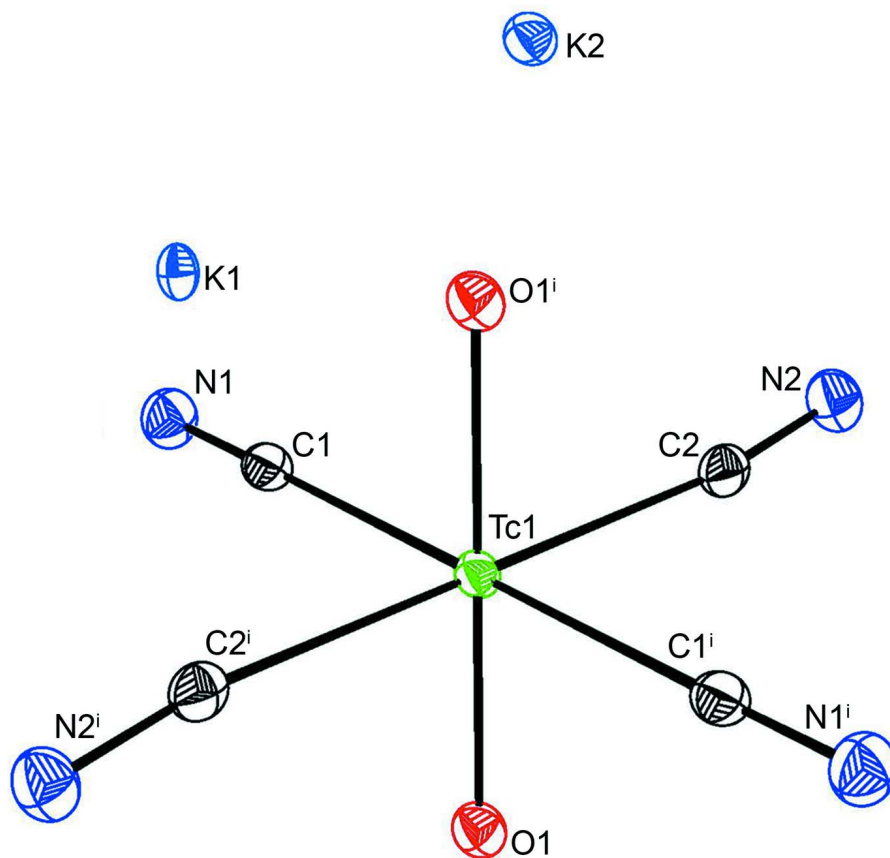


Figure 1

The structural moiety of (I) with the atomic labelling scheme and 50% probability ellipsoids. [Symmetry code: (i) $-x + 1, -y + 1, -z + 1$].

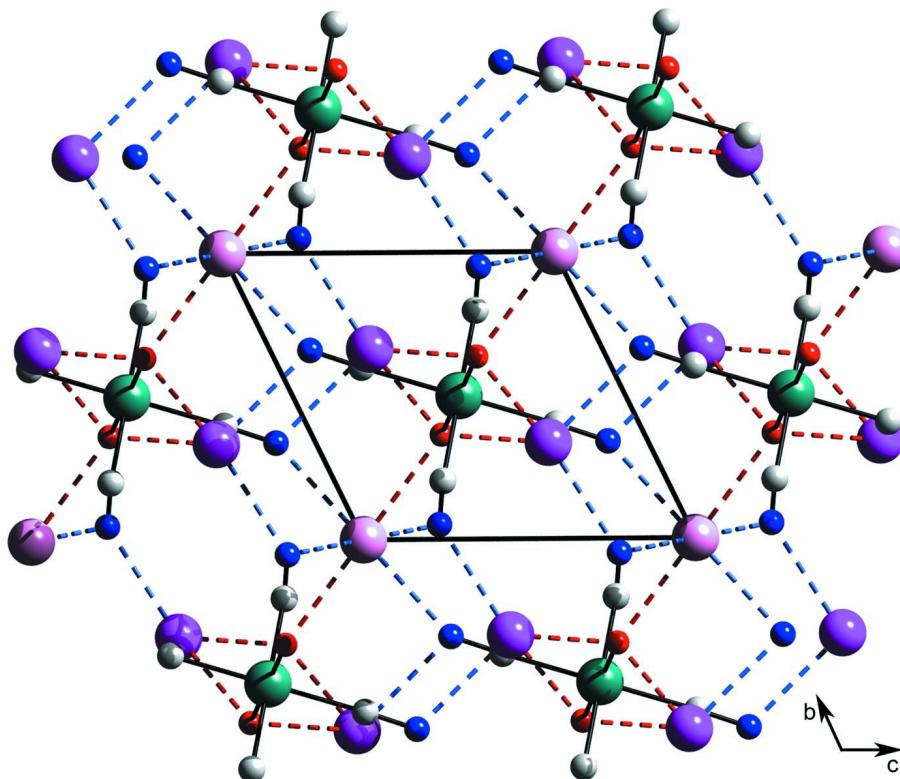


Figure 2

The extended packing motif of (I) in the bc plane showing the octahedral and tetrahedral environments about the K^+ ions. Dashed lines indicate the $K \cdots O$ (red) and $K \cdots N$ (blue) interionic interactions [K1 in light pink, K2 in dark pink].

tripotassium *trans*-tetracyanidodioxidotechnetate(V)

Crystal data

$K_3[TcO_2(CN)_4]$
 $M_r = 351.38$
 Triclinic, $P\bar{1}$
 Hall symbol: $-P\ 1$
 $a = 6.2539$ (6) Å
 $b = 6.9389$ (6) Å
 $c = 7.4347$ (7) Å
 $\alpha = 108.305$ (1)°
 $\beta = 109.816$ (2)°
 $\gamma = 104.143$ (1)°
 $V = 265.01$ (4) Å³

$Z = 1$
 $F(000) = 168$
 $D_x = 2.202$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 3550 reflections
 $\theta = 3.2$ – 30.0 °
 $\mu = 2.51$ mm⁻¹
 $T = 90$ K
 Fragment, yellow
 $0.32 \times 0.20 \times 0.14$ mm

Data collection

Bruker SMART APEX
 diffractometer
 Radiation source: normal-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 2007)
 $T_{\min} = 0.490$, $T_{\max} = 0.705$

3949 measured reflections
 1097 independent reflections
 1064 reflections with $> \sigma(I)$
 $R_{\text{int}} = 0.072$
 $\theta_{\max} = 26.5^\circ$, $\theta_{\min} = 3.2^\circ$
 $h = -7 \rightarrow 7$
 $k = -8 \rightarrow 8$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0114P)^2 + 0.0622P]$
$R[F^2 > 2\sigma(F^2)] = 0.020$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.049$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.09$	$\Delta\rho_{\max} = 0.77 \text{ e } \text{\AA}^{-3}$
1097 reflections	$\Delta\rho_{\min} = -0.90 \text{ e } \text{\AA}^{-3}$
68 parameters	Extinction correction: <i>SHELXTL</i> (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.024 (3)
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. A suitable crystal was mounted on a glass fiber and immediately transferred to the goniostat bathed in a cold stream.

CAUTION! All syntheses and characterizations were performed with ^{99}Tc , which is a β -emitting isotope with a half-life of 2.13×10^5 years. The handling of small quantities (generally <100 milligrams) of this material does not pose a serious health hazard since common laboratory materials provide adequate shielding. However, radiation safety procedures must be used to prevent contamination.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
K1	0.5000	0.0000	1.0000	0.01358 (15)
K2	0.04367 (6)	0.33536 (7)	0.69923 (6)	0.01283 (13)
Tc1	0.5000	0.5000	0.5000	0.00885 (11)
O1	0.6628 (2)	0.3641 (2)	0.39016 (18)	0.0123 (3)
N1	0.6522 (3)	0.3389 (3)	0.8804 (2)	0.0159 (4)
N2	-0.0098 (3)	0.0509 (3)	0.2506 (2)	0.0161 (4)
C1	0.5999 (3)	0.3996 (3)	0.7507 (3)	0.0120 (4)
C2	0.1707 (3)	0.2034 (3)	0.3254 (3)	0.0123 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0127 (3)	0.0130 (3)	0.0118 (3)	0.0033 (2)	0.0024 (2)	0.0064 (2)
K2	0.0118 (2)	0.0155 (3)	0.0129 (2)	0.00591 (17)	0.00588 (15)	0.00746 (18)
Tc1	0.00763 (14)	0.01073 (17)	0.00889 (14)	0.00383 (10)	0.00354 (9)	0.00513 (10)
O1	0.0110 (6)	0.0132 (7)	0.0131 (6)	0.0052 (5)	0.0050 (5)	0.0063 (5)
N1	0.0157 (7)	0.0175 (10)	0.0145 (7)	0.0059 (7)	0.0061 (6)	0.0084 (7)
N2	0.0139 (7)	0.0172 (9)	0.0164 (7)	0.0056 (7)	0.0055 (6)	0.0085 (7)
C1	0.0100 (8)	0.0120 (10)	0.0121 (8)	0.0033 (7)	0.0055 (6)	0.0035 (7)
C2	0.0136 (8)	0.0157 (11)	0.0106 (8)	0.0084 (8)	0.0055 (6)	0.0071 (7)

Geometric parameters (Å, °)

Tc1—O1 ⁱ	1.7721 (12)	N2—K2 ^v	2.7876 (19)
Tc1—O1	1.7721 (12)	N2—K1 ^{vi}	2.8598 (15)
Tc1—C1 ⁱ	2.1423 (19)	K1—O1 ^{vii}	2.8235 (13)
Tc1—C1	2.1423 (19)	K1—O1 ^{viii}	2.8235 (13)
Tc1—C2	2.145 (2)	K1—N1	2.8315 (18)
Tc1—C2 ⁱ	2.145 (2)	K1—N1 ^{ix}	2.8315 (18)
O1—K2 ⁱⁱ	2.7937 (12)	K1—N2 ^x	2.8598 (15)
O1—K1 ⁱⁱⁱ	2.8235 (13)	K1—N2 ^v	2.8598 (15)
O1—K2 ⁱ	2.8262 (14)	K2—N2 ^v	2.7877 (19)
N1—C1	1.150 (3)	K2—O1 ^{xi}	2.7936 (12)
N1—K2 ^{iv}	2.8152 (16)	K2—N1 ^{iv}	2.8152 (16)
N1—K2 ⁱⁱ	3.1710 (16)	K2—O1 ⁱ	2.8262 (14)
N2—C2	1.151 (3)	K2—N1 ^{xi}	3.1710 (16)
O1 ⁱ —Tc1—O1	180.0	C2—N2—K2 ^v	124.85 (13)
O1 ⁱ —Tc1—C1 ⁱ	90.13 (6)	C2—N2—K1 ^{vi}	127.59 (15)
O1—Tc1—C1 ⁱ	89.87 (6)	K2 ^v —N2—K1 ^{vi}	107.51 (6)
O1 ⁱ —Tc1—C1	89.87 (6)	O1 ^{vii} —K1—O1 ^{viii}	180.00 (5)
O1—Tc1—C1	90.13 (6)	O1 ^{vii} —K1—N1	97.86 (4)
C1 ⁱ —Tc1—C1	179.999 (1)	O1 ^{viii} —K1—N1	82.14 (4)
O1 ⁱ —Tc1—C2	88.60 (6)	O1 ^{vii} —K1—N1 ^{ix}	82.14 (4)
O1—Tc1—C2	91.40 (6)	O1 ^{viii} —K1—N1 ^{ix}	97.86 (4)
C1 ⁱ —Tc1—C2	92.34 (7)	N1—K1—N1 ^{ix}	180.00 (7)
C1—Tc1—C2	87.66 (7)	O1 ^{vii} —K1—N2 ^x	104.18 (4)
O1 ⁱ —Tc1—C2 ⁱ	91.40 (6)	O1 ^{viii} —K1—N2 ^x	75.82 (4)
O1—Tc1—C2 ⁱ	88.60 (6)	N1—K1—N2 ^x	95.00 (5)
C1 ⁱ —Tc1—C2 ⁱ	87.66 (7)	N1 ^{ix} —K1—N2 ^x	85.00 (5)
C1—Tc1—C2 ⁱ	92.34 (7)	O1 ^{vii} —K1—N2 ^v	75.82 (4)
C2—Tc1—C2 ⁱ	180.0	O1 ^{viii} —K1—N2 ^v	104.18 (4)
Tc1—O1—K2 ⁱⁱ	111.70 (5)	N1—K1—N2 ^v	85.00 (5)
Tc1—O1—K1 ⁱⁱⁱ	131.58 (5)	N1 ^{ix} —K1—N2 ^v	95.00 (5)
K2 ⁱⁱ —O1—K1 ⁱⁱⁱ	107.62 (4)	N2 ^x —K1—N2 ^v	180.0
Tc1—O1—K2 ⁱ	106.54 (6)	N2 ^v —K2—O1 ^{xi}	123.16 (4)
K2 ⁱⁱ —O1—K2 ⁱ	98.26 (4)	N2 ^v —K2—N1 ^{iv}	101.64 (5)
K1 ⁱⁱⁱ —O1—K2 ⁱ	94.38 (4)	O1 ^{xi} —K2—N1 ^{iv}	125.77 (5)
N1—C1—Tc1	177.71 (16)	N2 ^v —K2—O1 ⁱ	139.59 (4)
N2—C2—Tc1	172.74 (15)	O1 ^{xi} —K2—O1 ⁱ	81.74 (4)
C1—N1—K2 ^{iv}	116.78 (14)	N1 ^{iv} —K2—O1 ⁱ	82.38 (5)
C1—N1—K1	145.03 (14)	N2 ^v —K2—N1 ^{xi}	84.21 (5)
K2 ^{iv} —N1—K1	94.44 (5)	O1 ^{xi} —K2—N1 ^{xi}	77.02 (4)
C1—N1—K2 ⁱⁱ	73.15 (11)	N1 ^{iv} —K2—N1 ^{xi}	79.06 (5)
K2 ^{iv} —N1—K2 ⁱⁱ	100.94 (5)	O1 ⁱ —K2—N1 ^{xi}	135.31 (4)
K1—N1—K2 ⁱⁱ	118.11 (6)		

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+1, y, z$; (iii) $x, y, z-1$; (iv) $-x+1, -y+1, -z+2$; (v) $-x, -y, -z+1$; (vi) $x-1, y, z-1$; (vii) $-x+1, -y, -z+1$; (viii) $x, y, z+1$; (ix) $-x+1, -y, -z+2$; (x) $x+1, y, z+1$; (xi) $x-1, y, z$.