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Bis(1,10-phenanthroline)(2,2,6,6-tetramethylheptane-3,5-dionato)potassium(I) benzene sesquisolvate

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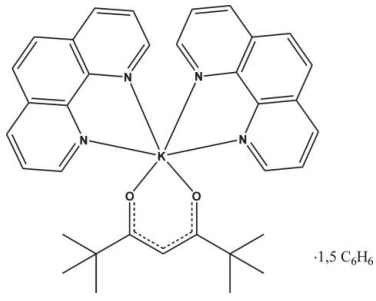
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.066; wR factor = 0.126; data-to-parameter ratio = 21.2.

The title compound, $[K(C_{11}H_{19}O_2)(C_{12}H_8N_2)_2]\cdot1.5C_6H_6$, is a potassium heteroligand β-diketonate complex with a mononuclear molecular structure in which a K^+ cation is coordinated by a dipivaloylmethanate anion (2,2,6,6-tetramethylheptane-3,5-dionate, dpm) and two 1,10-phenanthroline (phen) molecules as bidentate ligands. The coordination number (CN) of K in the $K(dpm)(phen)_2$ molecule is 6 and the coordinating atoms form a distorted trigonal prism. Face-to-face stacking interactions between phen ligands of neighbouring molecules [with perpendicular separations of 3.48 (5) Å] cause them to associate into chains along the [001] direction. The benzene solvent molecules in the structural cavities are edge-to-face stacked with the phen ligands.

Related literature

For background information, see: Romanov *et al.* (2004); Murzina *et al.* (2006). For related crystal structures, see: Bombieri *et al.*, (1984); Soboleva *et al.* (1995); Minacheva *et al.* (2003); Rogachev *et al.* (2005). For related literature, see: Dance (2003).



Experimental

Crystal data

$[K(C_{11}H_{19}O_2)(C_{12}H_8N_2)_2]\cdot1.5C_6H_6$
M_r = 699.93
Monoclinic, $P2_1/c$
a = 10.110 (2) Å
b = 22.419 (5) Å
c = 17.099 (3) Å
β = 98.55 (3)°
V = 3832.5 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 100 (2) K
0.50 × 0.30 × 0.20 mm

Data collection

Stoe IPDS diffractometer
Absorption correction: none
26291 measured reflections
9857 independent reflections
6012 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.126
S = 1.00
9857 reflections
466 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

K—O2	2.6051 (16)	K—N1	2.866 (2)
K—O1	2.6946 (15)	K—N4	2.8749 (19)
K—N2	2.852 (2)	K—N3	2.890 (2)
O2—K—O1	66.14 (5)	N4—K—N3	56.63 (6)
N2—K—N1	57.30 (5)		

Data collection: *IPDS* (Stoe & Cie, 1996); cell refinement: *IPDS*; data reduction: *IPDS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff *et al.*, 1996); software used to prepare material for publication: *publCIF* (Version 1.0c; Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2459).

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supplementary materials

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Bis(1,10-phenanthroline)(2,2,6,6-tetramethylheptane-3,5-dionato)potassium(I) benzene sesquisolvate

D. M. Tsymbarenko, I. E. Korsakov, A. R. Kaul, E. Kemnitz and S. I. Troyanov

Comment

Metal β-diketonates (especially acetylacetonates and dipivaloylmethanates) are widely used as volatile precursors for MOCVD deposition of thin films. In order to obtain a volatile precursor one should synthesize a substance with molecular crystal structure and low inter-molecular interactions. This task has not been solved yet for potassium, a large single charged cation that forms usually ionic crystals. Unfortunately, the coordination sphere of potassium (also Alkali Earth and Rare Earth elements) is not saturated by only the β-diketonate anion. This leads to the polymerization due to bridging function of ligands or solvent molecules and therefore to the reduction of volatility. A potassium precursor is essential for MOCVD of ferroelectric $KNbO_3$ thin films (Romanov *et al.*, 2004, Murzina *et al.*, 2006). We report here the first potassium heteroligand β-diketonate complex with a mononuclear structure $K(dpm)(phen)_2 \cdot 1.5C_6H_6$.

The crystal structure is built by the packing of voluminous $K(dpm)(phen)_2$ molecules and solvate benzene molecules lying in the lattice cavities. In the $K(dpm)(phen)_2$ molecule, the potassium cation has a distorted trigonal-prismatical coordination (CN=6) formed by four nitrogen atoms from two chelating phenanthroline ligands and by two oxygen atoms from chelating dipivaloylmethanate-anion (Fig. 1). The ligands do not exhibit a bridging function, therefore the molecules are monomeric and the compound has a molecular structure. The K···O1 and K···O2 distances are similar because of electron density delocalization in the chelating part of the dpm^--anion. The K^+ ion is displaced from the planes of the phenanthroline ligangs by 0.80 (1) Å. The K···N distances are comparable with those found in $[K_2(phen)_6]^{2+}[BPh_4]^-_2$ (Bombieri *et al.*, 1984).

The phenanthroline molecules lie in nearly orthogonal planes and participate in intermolecular stacking interaction of the face-to-face type with the neighboring $K(dpm)(phen)_2$ molecules (Fig. 2). No intramolecular stacking interaction similar to that found in $[K_2(phen)_6]^{2+}[BPh_4]^-_2$ (Bombieri *et al.*, 1984) occurs in the title crystal structure. The distance between parallel planes of phen-ligands (3.48 (5) Å) is typical for stacking distances in related compounds like $Ba(dpm)_2(phen)_2$ (Soboleva *et al.*, 1995) or $La(dpm)_3(phen)$ (Minacheva *et al.*, 2003) or $La(hfa)_3(phen)_2$ (Rogachev *et al.*, 2005). The stacking interaction between phen-ligands of neighboring molecules causes their association with the formation of chains along [001] direction. The solvate benzene molecules are edge-to-face stacked with phenanthroline ligands, while the molecular centroid separations are 4.9 – 5.2 Å, being in a good agreement with the values observed for the stacking interaction in a benzene pair $(C_6H_6)_2$ (Dance, 2003).

Experimental

The potassium *tert*-butyloxide (0.192 g, 1.72 mmol) and 1,10-phenanthroline (0.619 g, 3.44 mmol) were dissolved in dried benzene (15 ml) and stirred at room temperature. Then the solution of dipivaloylmethane (2,2,6,6-tetramethylheptane-3,5-dione, 0.332 g, 1.80 mmol) in benzene (5 ml) was added slowly under continuous stirring of mixture. All operations were

performed in a glove box. X-ray quality single-crystals were obtained by slow evaporation of benzene solution in evacuated sealed ampoules during two months.

Refinement

H-atoms were placed in idealized positions and refined using a riding model with C—H = 0.95 Å (or 0.98 Å) and with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

Figures

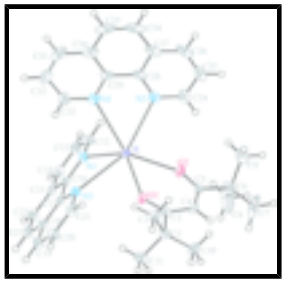

Fig. 1. The molecular structure of K(dpm)(phen)$_2$, with atom labels and 50% probability displacement ellipsoids for non-H atoms.

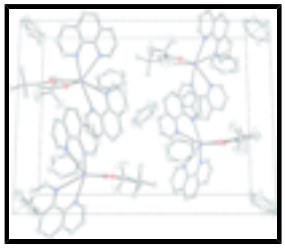

Fig. 2. The crystal unit cell of K(dpm)(phen)$_2$·1.5C_6H_6 viewed along the *a* axis, showing the stacking interaction between phen-ligands from the neighboring molecules of K(dpm)(phen)$_2$. H atoms were omitted for clarity.

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Crystal data

$[K(C_{11}H_{19}O_2)(C_{12}H_8N_2)_2]\cdot 1.5C_6H_6$	F_{000} = 1484
M_r = 699.93	D_x = 1.213 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 11312 reflections
a = 10.110 (2) Å	θ = 4–29º
b = 22.419 (5) Å	μ = 0.18 mm^{-1}
c = 17.099 (3) Å	*T* = 100 (2) K
β = 98.55 (3)º	Block, colourless
V = 3832.5 (13) Å^3	0.50 × 0.30 × 0.20 mm
Z = 4	

Data collection

Stoe IPDS diffractometer	6012 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.087

Monochromator: graphite	θ_{max} = 29.2º
T = 100(2) K	θ_{min} = 3.4º
φ scans	h = −13→10
Absorption correction: none	k = −30→30
26291 measured reflections	l = −20→22
9857 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.066	H-atom parameters constrained
$wR(F^2)$ = 0.126	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 +]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
9857 reflections	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
466 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
K	0.15866 (5)	0.739467 (18)	0.81820 (3)	0.01858 (11)
O1	0.07787 (15)	0.62510 (6)	0.82200 (9)	0.0186 (3)
O2	0.35426 (16)	0.66272 (6)	0.83782 (10)	0.0243 (4)
N1	0.1315 (2)	0.81523 (7)	0.68171 (12)	0.0213 (4)
N2	−0.06129 (19)	0.73100 (7)	0.69319 (12)	0.0207 (4)
N3	0.27796 (19)	0.78168 (7)	0.97236 (12)	0.0210 (4)
N4	0.0723 (2)	0.84560 (7)	0.89032 (12)	0.0235 (4)
C1	−0.0546 (2)	0.53894 (9)	0.90463 (15)	0.0231 (5)
H1A	−0.1196	0.5070	0.9083	0.035*
H1B	−0.1022	0.5762	0.8893	0.035*
H1C	0.0014	0.5442	0.9561	0.035*
C2	0.0337 (2)	0.52239 (8)	0.84243 (14)	0.0179 (4)

C3	0.1318 (2)	0.57547 (8)	0.83721 (13)	0.0161 (4)
C4	0.2711 (2)	0.56481 (8)	0.85082 (14)	0.0191 (5)
H4	0.2992	0.5248	0.8611	0.023*
C5	0.3723 (2)	0.60814 (8)	0.85059 (14)	0.0183 (4)
C6	0.5213 (2)	0.58812 (9)	0.86698 (16)	0.0240 (5)
C7	0.5438 (3)	0.52337 (13)	0.8882 (3)	0.0878 (17)
H7A	0.5089	0.4985	0.8426	0.132*
H7B	0.4972	0.5135	0.9329	0.132*
H7C	0.6398	0.5160	0.9029	0.132*
C8	0.5829 (4)	0.6024 (3)	0.7945 (3)	0.1001 (18)
H8A	0.6784	0.5925	0.8041	0.150*
H8B	0.5718	0.6450	0.7824	0.150*
H8C	0.5387	0.5789	0.7498	0.150*
C9	0.5923 (3)	0.62340 (17)	0.9365 (3)	0.0737 (13)
H9A	0.6868	0.6118	0.9466	0.111*
H9B	0.5506	0.6151	0.9836	0.111*
H9C	0.5855	0.6661	0.9244	0.111*
C10	0.1010 (2)	0.46250 (9)	0.86561 (17)	0.0282 (6)
H10A	0.0324	0.4322	0.8695	0.042*
H10B	0.1584	0.4665	0.9168	0.042*
H10C	0.1552	0.4505	0.8253	0.042*
C11	−0.0556 (3)	0.51592 (10)	0.76224 (16)	0.0317 (6)
H11A	−0.0012	0.5031	0.7224	0.048*
H11B	−0.0974	0.5544	0.7466	0.048*
H11C	−0.1251	0.4861	0.7664	0.048*
C12	0.2269 (3)	0.85464 (9)	0.67433 (16)	0.0268 (5)
H12	0.2831	0.8672	0.7209	0.032*
C13	0.2496 (3)	0.87876 (9)	0.60206 (16)	0.0283 (6)
H13	0.3200	0.9065	0.6002	0.034*
C14	0.1695 (2)	0.86196 (9)	0.53437 (15)	0.0238 (5)
H14	0.1840	0.8773	0.4846	0.029*
C15	0.0645 (2)	0.82131 (8)	0.53949 (14)	0.0193 (5)
C16	0.0504 (2)	0.79856 (8)	0.61459 (13)	0.0167 (4)
C17	−0.0537 (2)	0.75454 (8)	0.62091 (14)	0.0180 (4)
C18	−0.1392 (2)	0.73712 (9)	0.55162 (14)	0.0211 (5)
C19	−0.2382 (2)	0.69438 (10)	0.55995 (16)	0.0268 (5)
H19	−0.2994	0.6820	0.5153	0.032*
C20	−0.2456 (2)	0.67086 (10)	0.63288 (16)	0.0280 (5)
H20	−0.3117	0.6420	0.6395	0.034*
C21	−0.1542 (2)	0.68994 (9)	0.69768 (15)	0.0254 (5)
H21	−0.1591	0.6725	0.7478	0.031*
C22	−0.0260 (2)	0.80299 (9)	0.47072 (14)	0.0231 (5)
H22	−0.0173	0.8193	0.4205	0.028*
C23	−0.1230 (2)	0.76289 (10)	0.47713 (14)	0.0237 (5)
H23	−0.1820	0.7515	0.4311	0.028*
C24	0.3764 (2)	0.75095 (10)	1.01334 (16)	0.0286 (6)
H24	0.4222	0.7227	0.9858	0.034*
C25	0.4175 (3)	0.75747 (12)	1.09456 (17)	0.0373 (7)
H25	0.4899	0.7346	1.1207	0.045*

C26	0.3527 (3)	0.79685 (12)	1.13567 (17)	0.0378 (7)
H26	0.3787	0.8017	1.1910	0.045*
C27	0.2462 (3)	0.83057 (10)	1.09509 (15)	0.0299 (6)
C28	0.2124 (2)	0.82164 (9)	1.01298 (14)	0.0208 (5)
C29	0.1039 (2)	0.85520 (9)	0.96913 (15)	0.0218 (5)
C30	0.0349 (3)	0.89660 (10)	1.01031 (17)	0.0306 (6)
C31	−0.0709 (3)	0.92863 (10)	0.9654 (2)	0.0420 (8)
H31	−0.1204	0.9569	0.9906	0.050*
C32	−0.1018 (3)	0.91895 (11)	0.8863 (2)	0.0430 (8)
H32	−0.1726	0.9402	0.8556	0.052*
C33	−0.0270 (3)	0.87695 (10)	0.85130 (18)	0.0336 (6)
H33	−0.0488	0.8705	0.7960	0.040*
C34	0.1722 (3)	0.87284 (12)	1.13465 (17)	0.0399 (7)
H34	0.1947	0.8788	1.1900	0.048*
C35	0.0715 (3)	0.90404 (11)	1.09369 (19)	0.0417 (8)
H35	0.0234	0.9317	1.1208	0.050*
C36	0.4871 (3)	0.85637 (12)	0.85390 (18)	0.0366 (6)
H36	0.4323	0.8620	0.8938	0.044*
C37	0.5279 (3)	0.90462 (12)	0.8136 (2)	0.0456 (8)
H37	0.5013	0.9437	0.8258	0.055*
C38	0.6068 (3)	0.89627 (16)	0.7558 (2)	0.0589 (10)
H38	0.6328	0.9296	0.7274	0.071*
C39	0.6488 (3)	0.83982 (17)	0.7386 (2)	0.0526 (9)
H39	0.7054	0.8343	0.6995	0.063*
C40	0.6082 (3)	0.79190 (14)	0.77838 (19)	0.0448 (7)
H40	0.6362	0.7529	0.7665	0.054*
C41	0.5269 (3)	0.79958 (12)	0.83556 (17)	0.0364 (6)
H41	0.4983	0.7660	0.8624	0.044*
C42	0.4839 (3)	0.95220 (12)	1.0482 (2)	0.0540 (9)
H42	0.4730	0.9190	1.0812	0.065*
C43	0.3789 (3)	0.97211 (11)	0.9961 (2)	0.0485 (9)
H43	0.2944	0.9531	0.9934	0.058*
C44	0.3940 (3)	1.02007 (13)	0.9467 (3)	0.0586 (10)
H44	0.3206	1.0335	0.9097	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K	0.0224 (2)	0.01534 (17)	0.0169 (3)	0.00253 (18)	−0.00066 (17)	−0.00038 (18)
O1	0.0193 (8)	0.0167 (6)	0.0203 (9)	0.0024 (6)	0.0049 (6)	0.0020 (6)
O2	0.0182 (8)	0.0173 (7)	0.0370 (11)	0.0016 (6)	0.0024 (7)	0.0033 (6)
N1	0.0246 (11)	0.0176 (8)	0.0207 (12)	0.0001 (7)	−0.0005 (8)	−0.0001 (7)
N2	0.0203 (10)	0.0195 (8)	0.0220 (12)	0.0001 (7)	0.0027 (8)	0.0017 (7)
N3	0.0237 (10)	0.0223 (8)	0.0167 (12)	−0.0040 (7)	0.0026 (8)	0.0025 (7)
N4	0.0255 (11)	0.0196 (8)	0.0249 (13)	−0.0003 (7)	0.0021 (8)	0.0003 (7)
C1	0.0178 (11)	0.0241 (10)	0.0290 (15)	−0.0024 (9)	0.0092 (10)	0.0015 (9)
C2	0.0191 (11)	0.0162 (9)	0.0195 (13)	−0.0024 (8)	0.0061 (9)	0.0005 (8)
C3	0.0183 (11)	0.0182 (9)	0.0130 (12)	−0.0008 (8)	0.0063 (8)	−0.0002 (7)

C4	0.0210 (11)	0.0127 (8)	0.0245 (14)	0.0036 (8)	0.0065 (9)	0.0011 (8)
C5	0.0187 (11)	0.0196 (9)	0.0175 (13)	0.0029 (8)	0.0051 (9)	0.0005 (8)
C6	0.0141 (11)	0.0207 (10)	0.0375 (16)	0.0022 (8)	0.0049 (10)	0.0026 (9)
C7	0.0196 (16)	0.0328 (15)	0.205 (5)	0.0059 (12)	−0.004 (2)	0.023 (2)
C8	0.038 (2)	0.198 (5)	0.073 (3)	0.058 (3)	0.037 (2)	0.062 (3)
C9	0.0305 (18)	0.081 (2)	0.100 (3)	0.0238 (17)	−0.0241 (19)	−0.042 (2)
C10	0.0239 (13)	0.0173 (9)	0.0453 (18)	0.0005 (9)	0.0116 (11)	0.0050 (9)
C11	0.0427 (16)	0.0267 (11)	0.0250 (16)	−0.0116 (11)	0.0027 (12)	−0.0003 (9)
C12	0.0276 (13)	0.0194 (10)	0.0309 (16)	−0.0043 (9)	−0.0038 (11)	−0.0031 (9)
C13	0.0256 (13)	0.0190 (10)	0.0403 (17)	−0.0012 (9)	0.0046 (11)	0.0047 (9)
C14	0.0247 (12)	0.0204 (10)	0.0282 (15)	0.0072 (9)	0.0099 (10)	0.0074 (9)
C15	0.0207 (12)	0.0166 (9)	0.0210 (14)	0.0056 (8)	0.0049 (9)	0.0011 (8)
C16	0.0152 (11)	0.0156 (8)	0.0194 (13)	0.0043 (7)	0.0027 (9)	0.0003 (8)
C17	0.0176 (11)	0.0172 (9)	0.0194 (13)	0.0061 (8)	0.0036 (8)	0.0001 (8)
C18	0.0203 (11)	0.0217 (9)	0.0208 (13)	0.0055 (9)	0.0009 (9)	−0.0031 (9)
C19	0.0186 (12)	0.0290 (11)	0.0314 (16)	−0.0012 (9)	−0.0011 (10)	−0.0074 (10)
C20	0.0173 (12)	0.0293 (11)	0.0375 (17)	−0.0056 (9)	0.0047 (10)	0.0003 (10)
C21	0.0210 (12)	0.0266 (10)	0.0296 (16)	0.0004 (9)	0.0065 (10)	0.0039 (9)
C22	0.0279 (13)	0.0266 (10)	0.0156 (14)	0.0085 (9)	0.0058 (10)	0.0043 (8)
C23	0.0243 (12)	0.0271 (10)	0.0181 (13)	0.0070 (9)	−0.0019 (9)	−0.0033 (9)
C24	0.0242 (13)	0.0300 (12)	0.0310 (16)	−0.0031 (9)	0.0022 (10)	0.0086 (9)
C25	0.0310 (14)	0.0456 (14)	0.0316 (17)	−0.0126 (12)	−0.0077 (11)	0.0166 (12)
C26	0.0439 (17)	0.0471 (15)	0.0200 (16)	−0.0288 (13)	−0.0028 (12)	0.0033 (11)
C27	0.0368 (15)	0.0334 (12)	0.0204 (15)	−0.0224 (11)	0.0076 (11)	−0.0022 (10)
C28	0.0243 (12)	0.0198 (9)	0.0192 (14)	−0.0112 (8)	0.0060 (9)	−0.0022 (8)
C29	0.0238 (12)	0.0181 (9)	0.0251 (15)	−0.0084 (8)	0.0087 (10)	−0.0041 (8)
C30	0.0299 (14)	0.0248 (11)	0.0419 (18)	−0.0103 (10)	0.0210 (12)	−0.0102 (10)
C31	0.0318 (15)	0.0245 (12)	0.076 (3)	−0.0001 (11)	0.0271 (15)	−0.0089 (13)
C32	0.0308 (16)	0.0278 (12)	0.070 (3)	0.0064 (11)	0.0058 (15)	0.0025 (13)
C33	0.0321 (15)	0.0274 (11)	0.0398 (18)	0.0021 (10)	0.0003 (12)	0.0025 (10)
C34	0.060 (2)	0.0430 (14)	0.0216 (17)	−0.0269 (14)	0.0208 (14)	−0.0147 (12)
C35	0.056 (2)	0.0329 (13)	0.045 (2)	−0.0174 (13)	0.0366 (16)	−0.0201 (12)
C36	0.0315 (15)	0.0456 (14)	0.0315 (18)	−0.0009 (12)	0.0013 (12)	0.0012 (11)
C37	0.0307 (16)	0.0387 (14)	0.064 (2)	−0.0054 (12)	−0.0029 (15)	0.0056 (14)
C38	0.041 (2)	0.072 (2)	0.063 (3)	−0.0188 (16)	0.0051 (17)	0.0289 (18)
C39	0.0329 (18)	0.093 (3)	0.033 (2)	−0.0104 (17)	0.0060 (14)	−0.0017 (17)
C40	0.0405 (18)	0.0554 (17)	0.036 (2)	0.0009 (14)	−0.0015 (14)	−0.0138 (14)
C41	0.0383 (16)	0.0397 (13)	0.0287 (18)	−0.0027 (12)	−0.0033 (12)	0.0060 (11)
C42	0.0445 (19)	0.0283 (13)	0.093 (3)	−0.0069 (12)	0.0244 (19)	0.0141 (15)
C43	0.0297 (16)	0.0271 (12)	0.094 (3)	−0.0072 (11)	0.0253 (16)	−0.0026 (14)
C44	0.0370 (18)	0.0374 (15)	0.101 (3)	0.0001 (13)	0.0097 (19)	0.0148 (16)

Geometric parameters (Å, °)

K—O2	2.6051 (16)	C32—C33	1.397 (4)
K—O1	2.6946 (15)	C34—C35	1.344 (5)
K—N2	2.852 (2)	C36—C37	1.378 (4)
K—N1	2.866 (2)	C36—C41	1.385 (4)
K—N4	2.8749 (19)	C37—C38	1.372 (5)

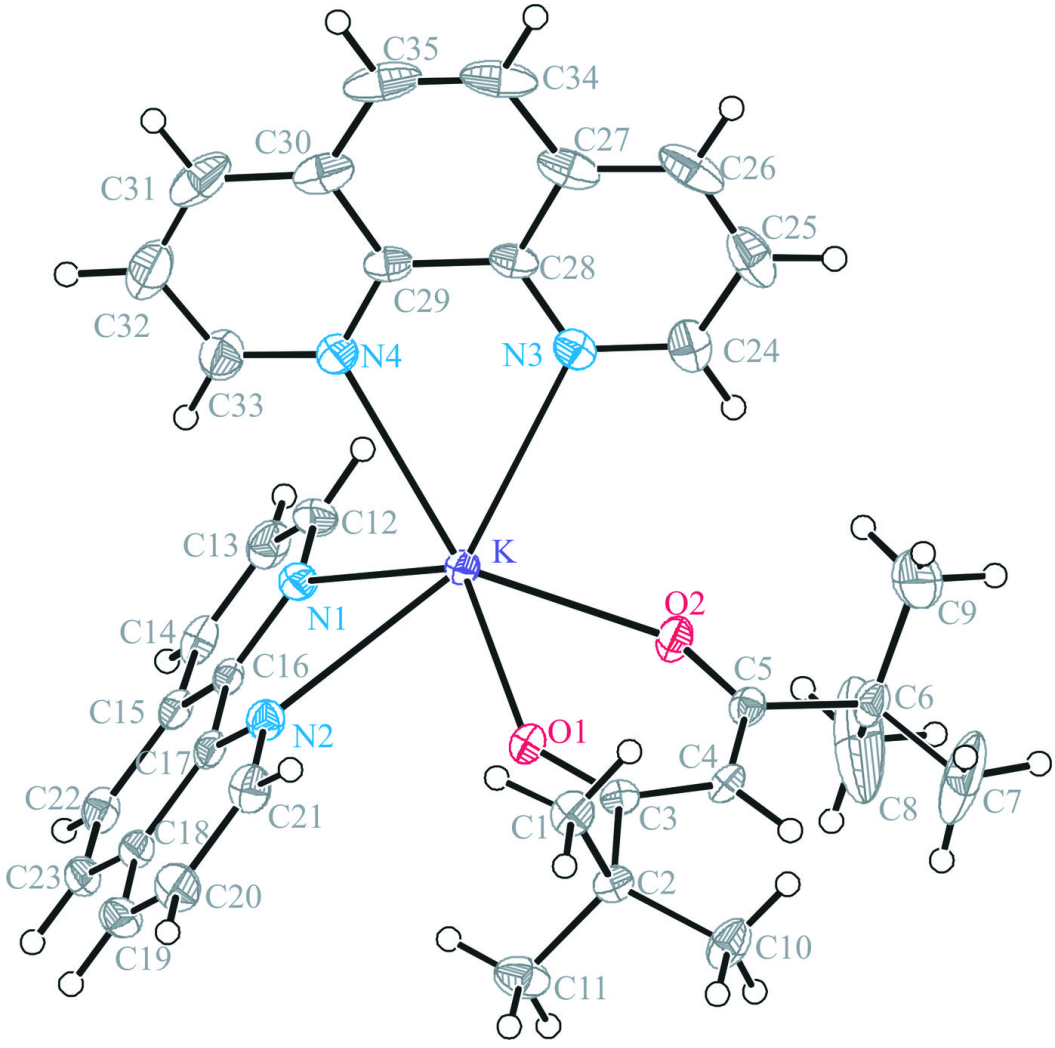
K—N3	2.890 (2)	C38—C39	1.380 (5)
O1—C3	1.249 (2)	C39—C40	1.367 (5)
O2—C5	1.252 (2)	C40—C41	1.378 (4)
N1—C12	1.328 (3)	C42—C43	1.355 (5)
N1—C16	1.360 (3)	C42—C44^{i}	1.373 (4)
N2—C21	1.326 (3)	C43—C44	1.390 (4)
N2—C17	1.357 (3)	C44—C42^{i}	1.373 (4)
N3—C24	1.323 (3)	C1—H1A	0.98
N3—C28	1.364 (3)	C1—H1B	0.98
N4—C33	1.322 (3)	C1—H1C	0.98
N4—C29	1.355 (3)	C4—H4	0.95
C1—C2	1.532 (3)	C7—H7A	0.98
C2—C10	1.531 (3)	C7—H7B	0.98
C2—C11	1.532 (3)	C7—H7C	0.98
C2—C3	1.560 (3)	C8—H8A	0.98
C3—C4	1.413 (3)	C8—H8B	0.98
C4—C5	1.412 (3)	C8—H8C	0.98
C5—C6	1.557 (3)	C9—H9A	0.98
C6—C8	1.501 (4)	C9—H9B	0.98
C6—C7	1.505 (3)	C9—H9C	0.98
C6—C9	1.517 (4)	C10—H10A	0.98
C12—C13	1.399 (4)	C10—H10B	0.98
C13—C14	1.363 (4)	C10—H10C	0.98
C14—C15	1.411 (3)	C11—H11A	0.98
C15—C16	1.408 (3)	C11—H11B	0.98
C15—C22	1.438 (3)	C11—H11C	0.98
C16—C17	1.458 (3)	C12—H12	0.95
C17—C18	1.414 (3)	C13—H13	0.95
C18—C19	1.408 (3)	C14—H14	0.95
C18—C23	1.430 (3)	C19—H19	0.95
C19—C20	1.366 (4)	C20—H20	0.95
C20—C21	1.400 (3)	C21—H21	0.95
C22—C23	1.346 (3)	C22—H22	0.95
C24—C25	1.397 (4)	C23—H23	0.95
C25—C26	1.356 (4)	C24—H24	0.95
C26—C27	1.411 (4)	C25—H25	0.95
C27—C28	1.409 (3)	C26—H26	0.95
C27—C34	1.437 (4)	C31—H31	0.95
C28—C29	1.444 (3)	C32—H32	0.95
C29—C30	1.410 (3)	C33—H33	0.95
C30—C31	1.416 (4)	C34—H34	0.95
C30—C35	1.428 (4)	C35—H35	0.95
C31—C32	1.360 (5)		
O2—K—O1	66.14 (5)	C31—C32—C33	118.3 (3)
O2—K—N2	123.01 (5)	N4—C33—C32	124.1 (3)
O1—K—N2	75.49 (5)	C35—C34—C27	120.3 (3)
O2—K—N1	118.37 (6)	C34—C35—C30	121.5 (3)
O1—K—N1	126.21 (5)	C37—C36—C41	119.3 (3)

N2—K—N1	57.30 (5)	C38—C37—C36	120.1 (3)
O2—K—N4	139.87 (6)	C37—C38—C39	120.6 (3)
O1—K—N4	131.66 (6)	C40—C39—C38	119.3 (3)
N2—K—N4	97.12 (6)	C39—C40—C41	120.7 (3)
N1—K—N4	81.98 (6)	C40—C41—C36	120.0 (3)
O2—K—N3	83.86 (6)	C43—C42—C44^{i}	120.1 (3)
O1—K—N3	112.11 (5)	C42—C43—C44	120.5 (3)
N2—K—N3	151.44 (6)	C42^{i}—C44—C43	119.3 (3)
N1—K—N3	121.67 (5)	C2—C1—H1A	109
N4—K—N3	56.63 (6)	C2—C1—H1B	110
C3—O1—K	136.73 (14)	C2—C1—H1C	110
C5—O2—K	139.17 (14)	H1A—C1—H1B	109
C12—N1—C16	117.3 (2)	H1A—C1—H1C	109
C12—N1—K	119.26 (16)	H1B—C1—H1C	109
C16—N1—K	119.80 (13)	C3—C4—H4	117
C21—N2—C17	117.4 (2)	C5—C4—H4	117
C21—N2—K	118.88 (15)	C6—C7—H7A	109
C17—N2—K	120.89 (14)	C6—C7—H7B	109
C24—N3—C28	117.2 (2)	C6—C7—H7C	109
C24—N3—K	119.45 (16)	H7A—C7—H7B	109
C28—N3—K	120.72 (15)	H7A—C7—H7C	109
C33—N4—C29	117.8 (2)	H7B—C7—H7C	110
C33—N4—K	118.67 (17)	C6—C8—H8A	109
C29—N4—K	121.08 (14)	C6—C8—H8B	109
C10—C2—C11	109.19 (19)	C6—C8—H8C	109
C10—C2—C1	108.36 (18)	H8A—C8—H8B	110
C11—C2—C1	108.5 (2)	H8A—C8—H8C	109
C10—C2—C3	114.97 (18)	H8B—C8—H8C	110
C11—C2—C3	108.41 (18)	C6—C9—H9A	110
C1—C2—C3	107.21 (16)	C6—C9—H9B	109
O1—C3—C4	125.32 (18)	C6—C9—H9C	110
O1—C3—C2	115.46 (18)	H9A—C9—H9B	109
C4—C3—C2	119.21 (17)	H9A—C9—H9C	109
C5—C4—C3	126.05 (18)	H9B—C9—H9C	109
O2—C5—C4	125.9 (2)	C2—C10—H10A	109
O2—C5—C6	115.19 (18)	C2—C10—H10B	109
C4—C5—C6	118.94 (17)	C2—C10—H10C	109
C8—C6—C7	109.8 (3)	H10A—C10—H10B	110
C8—C6—C9	109.3 (3)	H10A—C10—H10C	109
C7—C6—C9	106.1 (3)	H10B—C10—H10C	110
C8—C6—C5	107.4 (2)	C2—C11—H11A	109
C7—C6—C5	115.4 (2)	C2—C11—H11B	109
C9—C6—C5	108.8 (2)	C2—C11—H11C	109
N1—C12—C13	123.9 (2)	H11A—C11—H11B	109
C14—C13—C12	119.2 (2)	H11A—C11—H11C	109
C13—C14—C15	118.9 (2)	H11B—C11—H11C	110
C16—C15—C14	118.0 (2)	N1—C12—H12	118
C16—C15—C22	120.3 (2)	C13—C12—H12	118

C14—C15—C22	121.8 (2)	C12—C13—H13	120
N1—C16—C15	122.69 (19)	C14—C13—H13	120
N1—C16—C17	118.3 (2)	C13—C14—H14	121
C15—C16—C17	119.0 (2)	C15—C14—H14	120
N2—C17—C18	123.1 (2)	C18—C19—H19	120
N2—C17—C16	118.0 (2)	C20—C19—H19	120
C18—C17—C16	118.9 (2)	C19—C20—H20	120
C19—C18—C17	117.3 (2)	C21—C20—H20	121
C19—C18—C23	122.8 (2)	N2—C21—H21	118
C17—C18—C23	119.9 (2)	C20—C21—H21	118
C20—C19—C18	119.4 (2)	C15—C22—H22	120
C19—C20—C21	119.1 (2)	C23—C22—H22	120
N2—C21—C20	123.8 (2)	C18—C23—H23	119
C23—C22—C15	120.4 (2)	C22—C23—H23	119
C22—C23—C18	121.6 (2)	N3—C24—H24	118
N3—C24—C25	124.1 (3)	C25—C24—H24	118
C26—C25—C24	119.3 (3)	C24—C25—H25	120
C25—C26—C27	119.1 (3)	C26—C25—H25	120
C28—C27—C26	117.9 (3)	C25—C26—H26	120
C28—C27—C34	119.8 (3)	C27—C26—H26	120
C26—C27—C34	122.3 (3)	C30—C31—H31	120
N3—C28—C27	122.4 (2)	C32—C31—H31	120
N3—C28—C29	117.8 (2)	C31—C32—H32	121
C27—C28—C29	119.8 (2)	C33—C32—H32	121
N4—C29—C30	122.7 (2)	N4—C33—H33	118
N4—C29—C28	118.8 (2)	C32—C33—H33	118
C30—C29—C28	118.5 (2)	C27—C34—H34	120
C29—C30—C31	116.9 (3)	C35—C34—H34	120
C29—C30—C35	120.1 (3)	C30—C35—H35	119
C31—C30—C35	123.0 (3)	C34—C35—H35	119
C32—C31—C30	120.1 (2)		

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

Fig. 1


C35
C34
C30
C27
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C32
N4
N3
C24
C33
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C13
K
N1
O2
C9
C14
C16
C5
C6
C15
N2
O1
C4
C17
C22
C21
C3
C8
C7
C18
C1
C23
C20
C2
C19
C10
C11

Fig. 2