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Bis(μ-diphenylamido-κ²*N*:*N*)bis[tris(tetrahydrofuran-κ*O*)potassium(I)]

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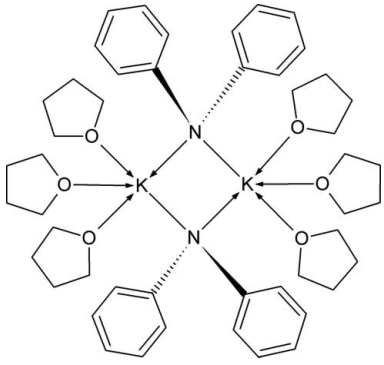
Key indicators: single-crystal X-ray study; T = 183 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.060; wR factor = 0.169; data-to-parameter ratio = 20.8.

The title compound, $[K_2(C_4H_8O)_6(C_{12}H_{10}N)_2]$, contains two K atoms which are coordinated by two bridging diphenylamide ligands to yield a centrosymmetric four-membered KNKN ring. Each K atom is additionally coordinated by three molecules of tetrahydrofuran, yielding a square pyramid in which one tetrahydrofuran molecule occupies the apical position. One CH_2 group and four H atoms are disordered equally over two positions.

Related literature

Synthesis of various transition metal diphenylamides: Seidel & Reichardt (1974); Villanueva *et al.* (1994); Barnhart *et al.* (1995); Tayebani *et al.* (1998); Gamer *et al.* (2001); Hitchcock *et al.* (2002); Hevia *et al.* (2002). Potassium diphenylamide as a catalyst in polymerization reactions: Longi *et al.* (1965); Kucera & Jelinek (1959); Grogler & Windemuth (1966). Synthesis of the title compound: Barnhart *et al.* (1995); Bergstrom *et al.* (1942); Cheshko & Goncharenko (1971); Fröhlich (1975).



Experimental

Crystal data

$[K_2(C_4H_8O)_6(C_{12}H_{10}N)_2]$
M_r = 847.24
Monoclinic, $P2_1/c$
a = 9.6546 (3) Å
b = 13.9660 (8) Å
c = 17.7356 (9) Å
β = 92.639 (3)°
V = 2388.9 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 183 (2) K
0.05 × 0.05 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.977, T_{max} = 0.997
15517 measured reflections
5438 independent reflections
3612 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.169
S = 0.95
5438 reflections
261 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Siemens, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2447).

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supplementary materials

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Bis(μ-diphenylamido-κ^2*N*:*N*)bis[tris(tetrahydrofuran-κ*O*)potassium(I)]

M. Gärtner, H. Görls and M. Westerhausen

Comment

One the one hand, potassium diphenylamide has been used for the synthesis of various transition metal diphenylamides, for example those of Cr (Seidel & Reichardt, 1974), Pd (Villanueva *et al.*, 1994), Th (Barnhart *et al.*, 1995), Nb (Tayebani *et al.*, 1998), Y and Sm (Gamer *et al.*, 2001), Yb (Hitchcock *et al.*, 2002), and Re (Hevia *et al.*, 2002). On the other hand, lactones and α,β-unsaturated cycloketones (Longi *et al.*, 1965), octamethylcyclotetrasiloxane (Kucera & Jelinek, 1959), and isocyanates (Grogler & Windemuth, 1966) could be polymerized with potassium diphenylamide as a catalyst. Though this compound is often cited in literature (Barnhart *et al.*, 1995; Bergstrom *et al.*, 1942; Cheshko & Goncharenko, 1971; Fröhlich, 1975), neither an exact synthetic approach nor structural or spectroscopic data have been published. Here we present a straightforward synthesis of potassium diphenylamide and the first molecular structure of a simple potassium amide. $KNPh_2$ was made of potassium and diphenylamine in boiling THF. Cooling a solution to −90 °C led to the formation of single crystals of $(thf)_3K(\mu_2\text{-}NPh_2)_2K(thf)_3$. In the crystalline state, potassium diphenylamide forms a dimeric centrosymmetric molecule with a four-membered KNK^iN^i ring [symmetry code: (i) 2 − *x*, 1 − *y*, −*z*]. Additionally, each K atom is saturated with three THF molecules. The coordination sphere of K is square-pyramidal with O2 occupying the apical position and N1, N1^i, O1, and O3 lying on the basal positions.

Experimental

All manipulations were carried out in an atmosphere of argon using standard Schlenk techniques. THF was dried (Na/benzophenone) and distilled prior to use. K and diphenylamine were purchased form Aldrich. ^{1}H NMR and ^{13}C NMR spectra were recorded at [D_8]THF solution at ambient temperature on a Bruker AC 400 MHz s pectrometer and were referenced to deuterated THF as an internal standard. A suspension of potassium (4.4 g, 112.5 mmol) and diphenylamine (15.2 g, 90.0 mmol, 0.8 eq.) in THF (100 ml) was heated under reflux for 2 h. Thereafter the suspension was cooled to ambient temperature and the excess of K was removed by filtation. The yield of 92% was determined by acidic titration of an hydrolysed aliquot. Cooling of this solution to −90 °C yields single crystals within 12 h, which were suitable for X-ray diffraction studies. Physical data: Mp: 100 °C (decomposition). ^{1}H NMR (400 MHz) δ 1.77 (thf), 3.60 (thf), 6.12–6.18 (m, 4H), 6.85–6.87 (m, 16H). ^{13}C NMR (100 MHz) δ 25.2 (thf), 67.3 (thf), 112.1 (4 C, *p*–C), 118.0 (8 C, *o*–C), 129.7 (8 C, *m*–C), 158.5 (4 C, *i*–C).

Refinement

All hydrogen atoms were calculated at idealized positions with $C_{aromatic}$—H = 0.95Å or $C_{methylene}$-H = 0.99Å and were refined with 1.5 times the isotropic displacement parameter of the corresponding carbon atoms. The tetrahydrofuran carbon atom C15 has a 50:50 occupancy disorder over two positions. Although the other tetrahedrofuran carbon atoms show high values for U(eq) no disorder could be resolved.

Figures

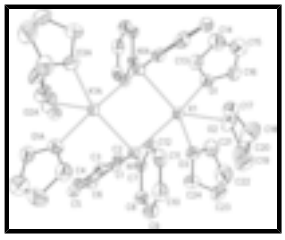


Fig. 1. The molecular structure of the title compound, showing 40% probability displacement ellipsoides and the atom-numbering scheme. H atoms have been omitted for clarity.

Bis(μ-diphenylamido-κ^2*N*:*N*)bis[tris(tetrahydrofuran-κ*O*)potassium(I)]

Crystal data

$[K_2(C_4H_8O)_6(C_{12}H_{10}N)_2]$	F_{000} = 912
M_r = 847.24	D_x = 1.178 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 15517 reflections
a = 9.6546 (3) Å	θ = 2.8–27.5º
b = 13.9660 (8) Å	μ = 0.25 mm^{-1}
c = 17.7356 (9) Å	T = 183 (2) K
β = 92.639 (3)º	Prism, colourless
V = 2388.9 (2) Å^3	0.05 × 0.05 × 0.04 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	5438 independent reflections
Radiation source: fine-focus sealed tube	3612 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.050
T = 183(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (Blessing, 1995)	h = −10→12
T_{min} = 0.977, T_{max} = 0.997	k = −18→15
15517 measured reflections	l = −23→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.060	H-atom parameters constrained
$wR(F^2)$ = 0.169	$w = 1/[\sigma^2(F_o^2) + (0.0746P)^2 + 1.881P]$ where $P = (F_o^2 + 2F_c^2)/3$

$S = 0.95$	$(\Delta/\sigma)_{max} < 0.001$
5438 reflections	$\Delta\rho_{max} = 0.39$ e Å^{-3}
261 parameters	$\Delta\rho_{min} = -0.29$ 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 $F^2 > \sigma(F^2)$ 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}	Occ. (<1)
K1	0.93906 (6)	0.41278 (4)	0.08773 (3)	0.04375 (18)	
O1	0.7561 (2)	0.26550 (15)	0.09945 (12)	0.0612 (6)	
O2	1.1232 (2)	0.3131 (2)	0.17851 (13)	0.0791 (7)	
O3	0.8091 (2)	0.48090 (16)	0.20882 (12)	0.0649 (6)	
N1	1.0160 (2)	0.60408 (14)	0.06975 (11)	0.0385 (5)	
C1	0.9378 (2)	0.68619 (17)	0.06408 (12)	0.0353 (5)	
C2	0.7919 (3)	0.67893 (18)	0.07046 (14)	0.0401 (6)	
H2A	0.7530	0.6191	0.0836	0.048*	
C3	0.7053 (3)	0.7565 (2)	0.05804 (14)	0.0456 (6)	
H3A	0.6082	0.7487	0.0624	0.055*	
C4	0.7564 (3)	0.8449 (2)	0.03953 (15)	0.0482 (6)	
H4A	0.6960	0.8979	0.0313	0.058*	
C5	0.8984 (3)	0.8547 (2)	0.03313 (15)	0.0473 (6)	
H5A	0.9355	0.9152	0.0201	0.057*	
C6	0.9866 (3)	0.77787 (19)	0.04540 (14)	0.0414 (6)	
H6A	1.0833	0.7871	0.0411	0.050*	
C7	1.1538 (2)	0.60788 (17)	0.09161 (13)	0.0376 (5)	
C8	1.2149 (3)	0.6754 (2)	0.14269 (14)	0.0451 (6)	
H8A	1.1591	0.7248	0.1622	0.054*	
C9	1.3538 (3)	0.6710 (2)	0.16476 (17)	0.0548 (7)	
H9A	1.3912	0.7175	0.1990	0.066*	
C10	1.4393 (3)	0.6009 (2)	0.13837 (18)	0.0574 (8)	
H10A	1.5349	0.5992	0.1536	0.069*	
C11	1.3826 (3)	0.5329 (2)	0.08893 (17)	0.0518 (7)	
H11A	1.4398	0.4838	0.0702	0.062*	
C12	1.2436 (3)	0.53584 (19)	0.06666 (14)	0.0424 (6)	
H12A	1.2072	0.4878	0.0334	0.051*	

C13	0.6390 (3)	0.2422 (2)	0.05079 (19)	0.0605 (8)	
H13A	0.6549	0.2623	−0.0016	0.073*	
H13B	0.5548	0.2748	0.0677	0.073*	
C14	0.6220 (5)	0.1361 (3)	0.0549 (3)	0.1017 (15)	
H14A	0.5362	0.1191	0.0802	0.122*	0.50
H14B	0.6185	0.1075	0.0037	0.122*	0.50
H14C	0.5226	0.1186	0.0532	0.122*	0.50
H14D	0.6665	0.1046	0.0122	0.122*	0.50
C15	0.7479 (10)	0.1027 (7)	0.1000 (6)	0.091 (3)*	0.50
H15A	0.7210	0.0557	0.1385	0.109*	0.50
H15B	0.8136	0.0714	0.0666	0.109*	0.50
C15A	0.6902 (7)	0.1071 (5)	0.1273 (4)	0.0568 (15)*	0.50
H15C	0.7262	0.0408	0.1252	0.068*	0.50
H15D	0.6267	0.1126	0.1694	0.068*	0.50
C16	0.8095 (4)	0.1814 (2)	0.13461 (19)	0.0685 (9)	
H16A	0.9112	0.1785	0.1301	0.082*	0.50
H16B	0.7899	0.1819	0.1889	0.082*	0.50
H16C	0.8359	0.1936	0.1883	0.082*	0.50
H16D	0.8920	0.1582	0.1089	0.082*	0.50
C17	1.2484 (4)	0.2853 (2)	0.14584 (18)	0.0630 (8)	
H17A	1.2664	0.3262	0.1018	0.076*	
H17B	1.2427	0.2178	0.1289	0.076*	
C18	1.3586 (4)	0.2967 (4)	0.2044 (2)	0.0874 (12)	
H18A	1.3732	0.2365	0.2331	0.105*	
H18B	1.4468	0.3152	0.1820	0.105*	
C19	1.3105 (5)	0.3717 (3)	0.2526 (2)	0.0923 (14)	
H19A	1.3312	0.4358	0.2320	0.111*	
H19B	1.3528	0.3663	0.3043	0.111*	
C20	1.1599 (5)	0.3541 (4)	0.2516 (2)	0.1056 (17)	
H20A	1.1371	0.3091	0.2924	0.127*	
H20B	1.1091	0.4147	0.2587	0.127*	
C21	0.7134 (4)	0.4283 (3)	0.2490 (2)	0.0849 (11)	
H21A	0.7362	0.3592	0.2479	0.102*	
H21B	0.6185	0.4373	0.2266	0.102*	
C22	0.7228 (6)	0.4653 (4)	0.3292 (3)	0.1159 (18)	
H22A	0.7229	0.4119	0.3659	0.139*	
H22B	0.6447	0.5089	0.3388	0.139*	
C23	0.8591 (6)	0.5181 (3)	0.3334 (2)	0.0988 (15)	
H23A	0.8574	0.5718	0.3698	0.119*	
H23B	0.9367	0.4746	0.3481	0.119*	
C24	0.8709 (4)	0.5535 (3)	0.2550 (2)	0.0758 (10)	
H24A	0.8211	0.6150	0.2476	0.091*	
H24B	0.9693	0.5628	0.2433	0.091*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0544 (4)	0.0404 (3)	0.0360 (3)	−0.0079 (3)	−0.0027 (2)	0.0040 (2)

O1	0.0723 (14)	0.0476 (12)	0.0624 (13)	−0.0130 (10)	−0.0101 (10)	0.0041 (10)
O2	0.0575 (13)	0.122 (2)	0.0572 (14)	0.0033 (13)	−0.0079 (10)	0.0204 (14)
O3	0.0830 (15)	0.0614 (13)	0.0510 (12)	−0.0142 (11)	0.0104 (10)	−0.0085 (10)
N1	0.0393 (11)	0.0356 (11)	0.0404 (11)	−0.0056 (8)	−0.0010 (8)	−0.0034 (9)
C1	0.0412 (13)	0.0376 (13)	0.0271 (11)	−0.0048 (10)	0.0001 (9)	−0.0035 (9)
C2	0.0425 (13)	0.0396 (14)	0.0382 (13)	−0.0068 (11)	0.0003 (10)	−0.0015 (10)
C3	0.0423 (14)	0.0528 (16)	0.0415 (14)	0.0012 (12)	−0.0016 (10)	−0.0049 (12)
C4	0.0566 (17)	0.0420 (15)	0.0451 (15)	0.0080 (12)	−0.0068 (12)	−0.0009 (12)
C5	0.0627 (17)	0.0390 (15)	0.0399 (14)	−0.0062 (12)	−0.0028 (12)	0.0009 (11)
C6	0.0464 (14)	0.0422 (14)	0.0358 (13)	−0.0076 (11)	0.0024 (10)	−0.0009 (11)
C7	0.0410 (13)	0.0397 (14)	0.0322 (12)	−0.0056 (10)	0.0029 (9)	0.0035 (10)
C8	0.0460 (15)	0.0482 (15)	0.0407 (14)	−0.0059 (11)	−0.0008 (11)	−0.0037 (11)
C9	0.0495 (16)	0.0614 (19)	0.0526 (17)	−0.0123 (14)	−0.0078 (12)	0.0009 (14)
C10	0.0407 (15)	0.066 (2)	0.0649 (19)	−0.0081 (14)	−0.0023 (13)	0.0171 (16)
C11	0.0483 (16)	0.0523 (17)	0.0556 (17)	0.0054 (13)	0.0112 (12)	0.0158 (13)
C12	0.0494 (15)	0.0396 (14)	0.0384 (13)	0.0001 (11)	0.0039 (10)	0.0050 (11)
C13	0.0472 (16)	0.070 (2)	0.0640 (19)	−0.0076 (14)	0.0014 (13)	0.0084 (16)
C14	0.115 (3)	0.075 (3)	0.112 (3)	−0.039 (2)	−0.035 (3)	0.002 (2)
C16	0.084 (2)	0.065 (2)	0.0558 (19)	−0.0097 (17)	−0.0108 (16)	0.0130 (16)
C17	0.082 (2)	0.0509 (18)	0.0555 (18)	0.0011 (16)	0.0028 (16)	−0.0037 (14)
C18	0.067 (2)	0.126 (4)	0.069 (2)	−0.008 (2)	−0.0059 (18)	0.003 (2)
C19	0.136 (4)	0.094 (3)	0.046 (2)	−0.045 (3)	0.000 (2)	−0.0012 (19)
C20	0.127 (4)	0.144 (4)	0.047 (2)	0.074 (3)	0.014 (2)	0.005 (2)
C21	0.090 (3)	0.088 (3)	0.079 (3)	−0.014 (2)	0.019 (2)	0.010 (2)
C22	0.155 (5)	0.114 (4)	0.084 (3)	0.012 (4)	0.062 (3)	0.011 (3)
C23	0.153 (4)	0.089 (3)	0.054 (2)	0.034 (3)	−0.007 (2)	−0.011 (2)
C24	0.101 (3)	0.061 (2)	0.065 (2)	−0.0071 (19)	−0.0024 (19)	−0.0148 (17)

Geometric parameters (Å, °)

K1—O3	2.709 (2)	C12—K1^{i}	3.268 (3)
K1—O2	2.725 (2)	C12—H12A	0.9500
K1—O1	2.726 (2)	C13—C14	1.492 (5)
K1—N1	2.795 (2)	C13—H13A	0.9900
K1—N1^{i}	2.856 (2)	C13—H13B	0.9900
K1—C12^{i}	3.268 (3)	C14—C15A	1.474 (8)
K1—C7^{i}	3.277 (2)	C14—C15	1.499 (10)
K1—C1^{i}	3.297 (2)	C14—H14A	0.9900
K1—C7	3.423 (2)	C14—H14B	0.9900
K1—C12	3.441 (3)	C14—H14C	0.9900
K1—K1^{i}	4.1643 (11)	C14—H14D	0.9900
O1—C16	1.416 (4)	C15—C16	1.380 (10)
O1—C13	1.428 (4)	C15—H15A	0.9900
O2—C17	1.418 (4)	C15—H15B	0.9900
O2—C20	1.446 (5)	C15A—C16	1.551 (7)
O3—C21	1.401 (4)	C15A—H15C	0.9900
O3—C24	1.418 (4)	C15A—H15D	0.9900
N1—C7	1.370 (3)	C16—H16A	0.9900

N1—C1	1.374 (3)	C16—H16B	0.9900
N1—K1^{i}	2.856 (2)	C16—H16C	0.9900
C1—C6	1.409 (3)	C16—H16D	0.9900
C1—C2	1.422 (3)	C17—C18	1.461 (5)
C1—K1^{i}	3.297 (2)	C17—H17A	0.9900
C2—C3	1.380 (4)	C17—H17B	0.9900
C2—H2A	0.9500	C18—C19	1.442 (6)
C3—C4	1.375 (4)	C18—H18A	0.9900
C3—H3A	0.9500	C18—H18B	0.9900
C4—C5	1.387 (4)	C19—C20	1.473 (6)
C4—H4A	0.9500	C19—H19A	0.9900
C5—C6	1.381 (4)	C19—H19B	0.9900
C5—H5A	0.9500	C20—H20A	0.9900
C6—H6A	0.9500	C20—H20B	0.9900
C7—C8	1.418 (3)	C21—C22	1.511 (6)
C7—C12	1.412 (4)	C21—H21A	0.9900
C7—K1^{i}	3.277 (2)	C21—H21B	0.9900
C8—C9	1.380 (4)	C22—C23	1.508 (7)
C8—H8A	0.9500	C22—H22A	0.9900
C9—C10	1.377 (4)	C22—H22B	0.9900
C9—H9A	0.9500	C23—C24	1.486 (5)
C10—C11	1.389 (4)	C23—H23A	0.9900
C10—H10A	0.9500	C23—H23B	0.9900
C11—C12	1.381 (4)	C24—H24A	0.9900
C11—H11A	0.9500	C24—H24B	0.9900
O3—K1—O2	91.40 (8)	C11—C12—K1	141.01 (18)
O3—K1—O1	83.05 (7)	C7—C12—K1	77.40 (14)
O2—K1—O1	88.67 (7)	K1^{i}—C12—K1	76.69 (6)
O3—K1—N1	83.69 (6)	C11—C12—H12A	118.9
O2—K1—N1	112.72 (7)	C7—C12—H12A	118.9
O1—K1—N1	155.04 (7)	K1^{i}—C12—H12A	57.7
O3—K1—N1^{i}	154.28 (7)	K1—C12—H12A	54.6
O2—K1—N1^{i}	114.29 (7)	O1—C13—C14	106.4 (3)
O1—K1—N1^{i}	98.08 (6)	O1—C13—H13A	110.4
N1—K1—N1^{i}	85.07 (6)	C14—C13—H13A	110.4
O3—K1—C12^{i}	109.59 (7)	O1—C13—H13B	110.4
O2—K1—C12^{i}	156.84 (8)	C14—C13—H13B	110.4
O1—K1—C12^{i}	84.43 (6)	H13A—C13—H13B	108.6
N1—K1—C12^{i}	80.24 (6)	C15A—C14—C15	29.5 (4)
N1^{i}—K1—C12^{i}	45.49 (6)	C15A—C14—C13	105.6 (4)
O3—K1—C7^{i}	133.17 (7)	C15—C14—C13	104.3 (4)
O2—K1—C7^{i}	132.08 (8)	C15A—C14—H14A	83.5
O1—K1—C7^{i}	81.85 (6)	C15—C14—H14A	110.9
N1—K1—C7^{i}	92.05 (6)	C13—C14—H14A	110.9
N1^{i}—K1—C7^{i}	24.59 (6)	C15A—C14—H14B	133.0

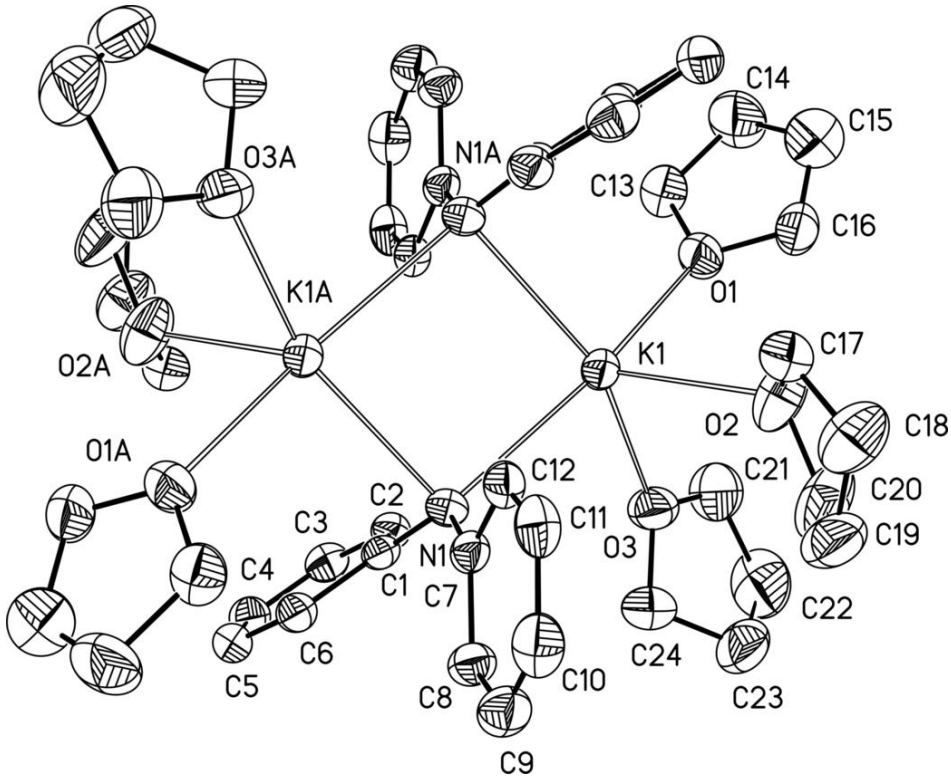
C12^{i}—K1—C7^{i}	24.92 (6)	C15—C14—H14B	110.9
O3—K1—C1^{i}	172.86 (6)	C13—C14—H14B	110.9
O2—K1—C1^{i}	91.05 (7)	H14A—C14—H14B	108.9
O1—K1—C1^{i}	90.30 (6)	C15A—C14—H14C	110.6
N1—K1—C1^{i}	101.53 (6)	C15—C14—H14C	134.4
N1^{i}—K1—C1^{i}	24.49 (6)	C13—C14—H14C	110.6
C12^{i}—K1—C1^{i}	66.97 (6)	H14A—C14—H14C	28.8
C7^{i}—K1—C1^{i}	42.55 (6)	H14B—C14—H14C	83.1
O3—K1—C7	90.39 (6)	C15A—C14—H14D	110.6
O2—K1—C7	90.97 (7)	C15—C14—H14D	84.3
O1—K1—C7	173.42 (7)	C13—C14—H14D	110.6
N1—K1—C7	22.70 (6)	H14A—C14—H14D	129.9
N1^{i}—K1—C7	88.06 (6)	H14B—C14—H14D	28.2
C12^{i}—K1—C7	98.34 (6)	H14C—C14—H14D	108.7
C7^{i}—K1—C7	103.18 (5)	C16—C15—C14	108.1 (6)
C1^{i}—K1—C7	96.27 (6)	C16—C15—H15A	110.1
O3—K1—C12	109.88 (7)	C14—C15—H15A	110.1
O2—K1—C12	77.41 (7)	C16—C15—H15B	110.1
O1—K1—C12	160.97 (7)	C14—C15—H15B	110.1
N1—K1—C12	43.72 (6)	H15A—C15—H15B	108.4
N1^{i}—K1—C12	76.47 (6)	C14—C15A—C16	100.9 (4)
C12^{i}—K1—C12	103.31 (6)	C14—C15A—H15C	111.6
C7^{i}—K1—C12	97.79 (6)	C16—C15A—H15C	111.6
C1^{i}—K1—C12	77.21 (6)	C14—C15A—H15D	111.6
C7—K1—C12	23.75 (6)	C16—C15A—H15D	111.6
O3—K1—K1^{i}	122.96 (5)	H15C—C15A—H15D	109.4
O2—K1—K1^{i}	122.77 (6)	C15—C16—O1	108.9 (4)
O1—K1—K1^{i}	134.82 (5)	C15—C16—C15A	29.2 (4)
N1—K1—K1^{i}	43.10 (4)	O1—C16—C15A	105.3 (3)
N1^{i}—K1—K1^{i}	41.97 (4)	C15—C16—H16A	109.9
C12^{i}—K1—K1^{i}	53.53 (5)	O1—C16—H16A	109.9
C7^{i}—K1—K1^{i}	53.16 (4)	C15A—C16—H16A	134.6
C1^{i}—K1—K1^{i}	60.66 (4)	C15—C16—H16B	109.9
C7—K1—K1^{i}	50.02 (4)	O1—C16—H16B	109.9
C12—K1—K1^{i}	49.79 (4)	C15A—C16—H16B	84.9
C16—O1—C13	109.8 (2)	H16A—C16—H16B	108.3
C16—O1—K1	115.80 (18)	C15—C16—H16C	130.7
C13—O1—K1	128.60 (18)	O1—C16—H16C	110.7
C17—O2—C20	107.2 (3)	C15A—C16—H16C	110.7
C17—O2—K1	116.32 (18)	H16A—C16—H16C	82.7
C20—O2—K1	117.0 (2)	H16B—C16—H16C	27.7
C21—O3—C24	110.6 (3)	C15—C16—H16D	82.8
C21—O3—K1	124.0 (2)	O1—C16—H16D	110.7
C24—O3—K1	120.7 (2)	C15A—C16—H16D	110.7

C7—N1—C1	120.8 (2)	H16A—C16—H16D	29.2
C7—N1—K1	105.35 (14)	H16B—C16—H16D	130.1
C1—N1—K1	131.14 (15)	H16C—C16—H16D	108.8
C7—N1—K1^{i}	95.21 (14)	O2—C17—C18	106.6 (3)
C1—N1—K1^{i}	96.02 (14)	O2—C17—H17A	110.4
K1—N1—K1^{i}	94.93 (6)	C18—C17—H17A	110.4
N1—C1—C6	126.0 (2)	O2—C17—H17B	110.4
N1—C1—C2	118.5 (2)	C18—C17—H17B	110.4
C6—C1—C2	115.2 (2)	H17A—C17—H17B	108.6
N1—C1—K1^{i}	59.49 (12)	C19—C18—C17	104.8 (4)
C6—C1—K1^{i}	92.98 (14)	C19—C18—H18A	110.8
C2—C1—K1^{i}	115.53 (15)	C17—C18—H18A	110.8
C3—C2—C1	121.8 (2)	C19—C18—H18B	110.8
C3—C2—H2A	119.1	C17—C18—H18B	110.8
C1—C2—H2A	119.1	H18A—C18—H18B	108.9
C4—C3—C2	121.4 (2)	C18—C19—C20	102.5 (3)
C4—C3—H3A	119.3	C18—C19—H19A	111.3
C2—C3—H3A	119.3	C20—C19—H19A	111.3
C3—C4—C5	118.4 (3)	C18—C19—H19B	111.3
C3—C4—H4A	120.8	C20—C19—H19B	111.3
C5—C4—H4A	120.8	H19A—C19—H19B	109.2
C6—C5—C4	121.0 (3)	O2—C20—C19	106.1 (3)
C6—C5—H5A	119.5	O2—C20—H20A	110.5
C4—C5—H5A	119.5	C19—C20—H20A	110.5
C5—C6—C1	122.2 (2)	O2—C20—H20B	110.5
C5—C6—H6A	118.9	C19—C20—H20B	110.5
C1—C6—H6A	118.9	H20A—C20—H20B	108.7
N1—C7—C8	125.2 (2)	O3—C21—C22	106.7 (4)
N1—C7—C12	119.1 (2)	O3—C21—H21A	110.4
C8—C7—C12	115.5 (2)	C22—C21—H21A	110.4
N1—C7—K1^{i}	60.20 (12)	O3—C21—H21B	110.4
C8—C7—K1^{i}	140.00 (17)	C22—C21—H21B	110.4
C12—C7—K1^{i}	77.16 (14)	H21A—C21—H21B	108.6
N1—C7—K1	51.96 (12)	C21—C22—C23	103.2 (3)
C8—C7—K1	140.94 (17)	C21—C22—H22A	111.1
C12—C7—K1	78.85 (14)	C23—C22—H22A	111.1
K1^{i}—C7—K1	76.82 (5)	C21—C22—H22B	111.1
C9—C8—C7	121.4 (3)	C23—C22—H22B	111.1
C9—C8—H8A	119.3	H22A—C22—H22B	109.1
C7—C8—H8A	119.3	C24—C23—C22	102.7 (4)
C10—C9—C8	121.7 (3)	C24—C23—H23A	111.2
C10—C9—H9A	119.1	C22—C23—H23A	111.2
C8—C9—H9A	119.1	C24—C23—H23B	111.2
C9—C10—C11	118.4 (3)	C22—C23—H23B	111.2
C9—C10—H10A	120.8	H23A—C23—H23B	109.1
C11—C10—H10A	120.8	O3—C24—C23	104.7 (3)
C12—C11—C10	120.6 (3)	O3—C24—H24A	110.8

C12—C11—H11A	119.7	C23—C24—H24A	110.8
C10—C11—H11A	119.7	O3—C24—H24B	110.8
C11—C12—C7	122.3 (3)	C23—C24—H24B	110.8
C11—C12—K1^{i}	136.53 (17)	H24A—C24—H24B	108.9
C7—C12—K1^{i}	77.91 (14)		

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

Fig. 1


O3A
N1A
C14
C15
C13
C16
O1
K1A
O2A
K1
C17
O2
C18
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C21
C20
C12
C2
C3
C11
O3
C19
N1
C4
C1
C7
C22
C6
C24
C5
C10
C8
C23
C9