Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Poly[[μ-dioxane-κ^2O:O'-dioxane-κO-μ-diphenylamido-κ^2N:N-potassium] dioxane solvate]

Martin Gärtner, Helmar Görls and Matthias Westerhausen*

Institute of Inorganic and Analytical Chemistry, Friedrich-Schiller-Universität Jena, August-Bebel-Strasse 2, D-07743 Jena, Germany
Correspondence e-mail: m.we@uni-jena.de

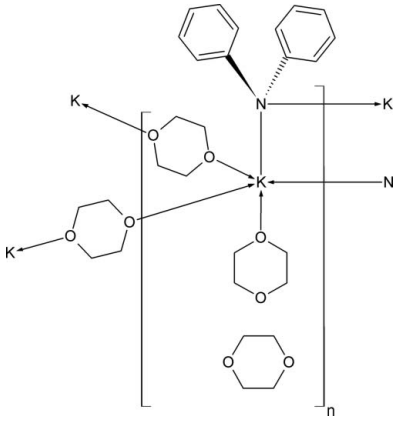
Received 19 July 2007; accepted 31 July 2007

Key indicators: single-crystal X-ray study; T = 183 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.107; data-to-parameter ratio = 18.9.

The title compound, $\{[K(C_{12}H_{10}N)(C_4H_8O_2)_2]\cdot C_4H_8O_2\}_n$, contains a K atom surrounded by two bridging diphenylamido ligands and three molecules of dioxane. Two of these dioxane ligands are located on a centre of inversion and form bridges to neighbouring K atoms, yielding two-dimensional layers. The K atom is in a distorted square-pyramidal environment in which one bridging dioxane molecule occupies the apical position.

Related literature

For the synthesis of transition metal diphenylamides, see: 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). For potassium diphenylamide as a catalyst in polymerization reactions, see: Longi *et al.* (1965); Kucera & Jelinek (1959); Grogler & Windemuth (1966). For the synthesis of the title compound, see: Barnhart *et al.* (1995); Bergstrom *et al.* (1942); Cheshko & Goncharenko (1971); Fröhlich (1975). For the synthesis and crystal structure of bis[tris(tetrahydrofuran-O)(μ_2-diphenylamido)potassium], see: Gärtner *et al.* (2007).



Experimental

Crystal data

$[K(C_{12}H_{10}N)(C_4H_8O_2)_2]\cdot C_4H_8O_2$
M_r = 471.62
Triclinic, $P\overline{1}$
a = 10.5417 (5) Å
b = 10.6758 (6) Å
c = 12.7711 (5) Å
α = 70.419 (3)°
β = 86.123 (3)°
γ = 66.278 (2)°
V = 1235.97 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 183 (2) K
0.06 × 0.06 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.967, T_{max} = 0.987
8425 measured reflections
5472 independent reflections
3974 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.107
S = 1.01
5472 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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: *SHELXTL/PC* (Siemens, 1990); software used to prepare material for publication: *SHELXL97*.

We thank the Deutsche Forschungsgemeinschaft (DFG, Bonn–Band Godesberg, Germany) for generous financial support. We also acknowledge funding from the Fonds der Chemischen Industrie (Frankfurt/Main, Germany). In addition, MG is very grateful to the Verband der Chemischen Industrie (VCI/FCI) for a scholarship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2446).

References

Barnhart, D. M., Clark, D. L., Grumbine, S. K. & Watkin, J. G. (1995). *Inorg. Chem.* **34**, 1695–1699.
Bergstrom, F. W., Granara, I. M. & Erickson, V. (1942). *J. Org. Chem.* **7**, 98–102.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Cheshko, F. F. & Goncharenko, I. A. (1971). US Patent 320 499.
Fröhlich, H. O. (1975). *Z. Chem.* **15**, 316–317.
Gamer, M. T., Dehnen, S. & Roesky, P. W. (2001). *Organometallics*, **20**, 4230–4236.
Gärtner, M., Görls, H. & Westerhausen, M. (2007). *Acta Cryst.* E**63**, m2289.
Grogler, G. & Windemuth, E. (1966). Br. Patent 1 034 152.
Hevia, E., Perez, J. R. V. & Miguel, D. (2002). *Organometallics*, **21**, 1966–1974.
Hitchcock, P. B., Khvostov, A. V., Lappert, M. F. & Protchenko, A. V. (2002). *J. Organomet. Chem.* **647**, 198–204.
Kucera, M. & Jelinek, M. (1959). Czech. Patent 89809.
Longi, P., Greco, F. & Mapelli, F. (1965). *Chim. Ind. (Milan)*, **47**, 951–954.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Seidel, W. & Reichardt, W. (1974). *Z. Anorg. Allg. Chem.* **404**, 225–229.

 doi:10.1107/S1600536807037427

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1990). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tayebani, M., Gambarotta, S. & Yap, G. (1998). *Organometallics*, **17**, 3639–3641.
Villanueva, L. A., Abboud, K. A. & Boncella, J. M. (1994). *Organometallics*, **13**, 3921–3931.

supplementary materials

Acta Cryst. (2007). E**63**, m2287-m2288 [**doi:10.1107/S1600536807037427**]

Poly[[μ-dioxane-κ^2O:O'-dioxane-κO-μ-diphenylamido-κ^2N:N-potassium] dioxane solvate]

M. Gärtner, H. Görls and M. Westerhausen

Comment

In the past, potassium diphenylamide has been used for the synthesis of a lot of 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). Potassium diphenylamide also served as a catalyst in the polymerization of lactones and α,β-unsaturated cycloketones (Longi *et al.*, 1965), octamethylcyclotetrasiloxane (Kucera & Jelinek, 1959), and isocyanates (Grogler & Windemuth, 1966). This compound is often mentioned in literature (Barnhart *et al.*, 1995; Bergstrom *et al.*, 1942; Cheshko & Goncharenko, 1971; Fröhlich, 1975), but neither a complete synthetic method nor structural or spectroscopic data have been published. The reaction of potassium with diphenylamine in boiling THF gives $(thf)_3K(\mu_2\text{-}NPh_2)_2K(thf)_3$. Recrystallization from hot dioxane yields single crystals of $[(\mu\text{-}O,O'\text{-dx})(\text{dx-}O)K(\mu\text{-}NPh_2)]_\infty$ ((I)) at ambient temperature. In the molecular structure of (I), potassium diphenylamide forms a dimeric molecule with a centosymmetric four-membered KNK^iN^i ring [symmetry code: (i) $2-x$, $1-y$, $-z$]. Additionally, each K atom is saturated with three molecules of dioxane, two of them are attached to neighbouring K atoms with the other oxygen atom. This leads to the formation of parallel layers parallel to (001). The coordination sphere of K is distorted square-pyramidal with O4 occupying the apical position and N1, $N1^i$, O1, and O3 lying on the basal positions. The compound cocrystallizes with a molecule of dioxane in the asymmetric unit.

Experimental

All manipulations were carried out in an atmosphere of argon using standard Schlenk techniques. THF, diethyl ether and dioxane were 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.

Bis[tris(tetrahydrofuran-*O*)(μ_2-diphenylamido)potassium] was prepared according to literature procedure (Gärtner *et al.*, 2007) and recrystallized from hot 1,4-dioxane. Storage of this solution at ambient temperature led to the formation of single crystals within 12 h.

Physical data:

Mp: 90°C (decomposition).

^{1}H NMR (400 MHz) δ 3.56 (dx), 6.14–6.18 (m, 2H), 6.85–6.90 (m, 8H).

^{13}C NMR (100 MHz) δ 67.7 (dx), 112.3 (2 C, *p*–C), 118.0 (4 C, *o*–C), 129.7 (4 C, *m*–C), 158.3 (2 C, *i*–C).

MS (DEI, *m/z* [%]): 208 (M^+, [1]).

IR (cm^{-1}): 1592, 1570, 1551, 1313, 1255, 1212, 1165, 1118, 1080, 1047, 985, 888, 871, 798, 753, 710, 700, 613, 523, 503.

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.

Figures

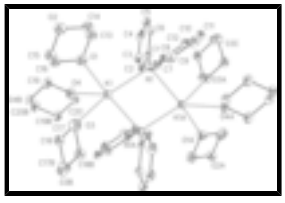


Fig. 1. The molecular structure of (I), showing 40% prabability displacement ellipsoides and the atom-numbering scheme. H atoms have been omitted for clarity.

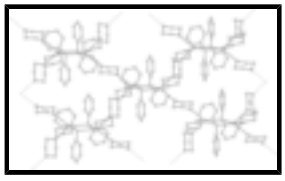


Fig. 2. The crystal structure of one layer. H atoms have been omitted for clarity.

Poly[[μ-dioxane-κ^2*O*:*O*'-dioxane-κ*O*-μ-diphenylamido-κ^2*N*:*N*-\ potassium] dioxane solvate]

Crystal data

$[K(C_{12}H_{10}N)(C_4H_8O_2)_2]\cdot C_4H_8O_2$	$Z = 2$
$M_r = 471.62$	$F_{000} = 504$
Triclinic, $P\overline{1}$	$D_x = 1.267$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.5417$ (5) Å	Cell parameters from 8425 reflections
$b = 10.6758$ (6) Å	$\theta = 2.1$–27.4°
$c = 12.7711$ (5) Å	$\mu = 0.25$ mm^{-1}
$\alpha = 70.419$ (3)°	$T = 183$ (2) K
$\beta = 86.123$ (3)°	Prism, colourless
$\gamma = 66.278$ (2)°	0.06 × 0.06 × 0.05 mm
$V = 1235.97$ (10) Å^3	

Data collection

Nonius KappaCCD diffractometer	5472 independent reflections
Radiation source: fine-focus sealed tube	3974 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.029$
$T = 183$(2) K	$\theta_{max} = 27.4^\circ$
φ and ω scans	$\theta_{min} = 2.1^\circ$

Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.967, T_{max} = 0.987
8425 measured reflections

h = −13→12
k = −13→11
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.107
S = 1.01
5472 reflections
289 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
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.0755P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
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}
K1	0.80940 (3)	0.58975 (4)	0.06727 (3)	0.02744 (12)
O1	0.73827 (13)	0.51425 (15)	0.28382 (9)	0.0402 (3)
O2	0.68752 (13)	0.35147 (15)	0.49469 (10)	0.0424 (3)
O3	0.58105 (12)	0.54644 (14)	0.04898 (10)	0.0352 (3)
O4	0.61613 (12)	0.86555 (13)	0.04205 (10)	0.0368 (3)
O5	0.88112 (13)	0.75650 (14)	0.39904 (10)	0.0433 (3)
O6	0.71297 (15)	1.04991 (15)	0.27924 (11)	0.0492 (4)
N1	1.08797 (13)	0.42696 (15)	0.14643 (10)	0.0259 (3)
C1	1.12229 (16)	0.53691 (18)	0.15025 (13)	0.0245 (4)
C2	1.07544 (16)	0.66780 (18)	0.05752 (13)	0.0281 (4)
H2A	1.0300	0.6720	−0.0061	0.034*
C3	1.09369 (18)	0.7895 (2)	0.05649 (15)	0.0347 (4)
H3A	1.0599	0.8752	−0.0071	0.042*
C4	1.16059 (19)	0.7883 (2)	0.14690 (16)	0.0388 (4)

H4A	1.1736	0.8717	0.1460	0.047*
C5	1.20816 (18)	0.6613 (2)	0.23914 (16)	0.0366 (4)
H5A	1.2539	0.6586	0.3020	0.044*
C6	1.19035 (17)	0.5389 (2)	0.24136 (14)	0.0308 (4)
H6A	1.2246	0.4540	0.3056	0.037*
C7	1.16056 (16)	0.28448 (18)	0.21367 (12)	0.0249 (4)
C8	1.09112 (18)	0.18982 (19)	0.24413 (13)	0.0296 (4)
H8A	0.9948	0.2278	0.2227	0.036*
C9	1.1595 (2)	0.0436 (2)	0.30425 (14)	0.0357 (4)
H9A	1.1093	−0.0165	0.3235	0.043*
C10	1.2995 (2)	−0.0164 (2)	0.33671 (14)	0.0377 (5)
H10A	1.3461	−0.1170	0.3778	0.045*
C11	1.37052 (18)	0.0734 (2)	0.30805 (14)	0.0349 (4)
H11A	1.4669	0.0337	0.3301	0.042*
C12	1.30370 (17)	0.21958 (19)	0.24804 (13)	0.0295 (4)
H12A	1.3555	0.2781	0.2294	0.035*
C13	0.8367 (2)	0.4525 (2)	0.37896 (15)	0.0424 (5)
H13A	0.8192	0.5251	0.4161	0.051*
H13B	0.9321	0.4269	0.3541	0.051*
C14	0.82513 (19)	0.3192 (2)	0.46008 (16)	0.0420 (5)
H14A	0.8500	0.2437	0.4248	0.050*
H14B	0.8913	0.2804	0.5260	0.050*
C15	0.5912 (2)	0.4105 (2)	0.40013 (15)	0.0388 (5)
H15A	0.4957	0.4343	0.4245	0.047*
H15B	0.6111	0.3375	0.3629	0.047*
C16	0.60096 (19)	0.5448 (2)	0.31960 (14)	0.0365 (4)
H16A	0.5341	0.5838	0.2541	0.044*
H16B	0.5760	0.6196	0.3556	0.044*
C17	0.5678 (2)	0.4124 (2)	0.10436 (16)	0.0408 (5)
H17A	0.4899	0.4284	0.1531	0.061*
H17B	0.6540	0.3416	0.1519	0.061*
C18	0.45827 (19)	0.6481 (2)	−0.02155 (16)	0.0374 (4)
H18A	0.4689	0.7400	−0.0615	0.056*
H18B	0.3779	0.6697	0.0243	0.056*
C19	0.51208 (19)	0.9214 (2)	0.11231 (14)	0.0392 (5)
H19A	0.5408	0.9780	0.1460	0.059*
H19B	0.5035	0.8395	0.1735	0.059*
C20	0.62656 (18)	0.98288 (19)	−0.04698 (14)	0.0322 (4)
H20A	0.6962	0.9445	−0.0968	0.048*
H20B	0.6581	1.0407	−0.0169	0.048*
C21	0.7750 (2)	0.8442 (2)	0.44988 (15)	0.0436 (5)
H21A	0.8030	0.8128	0.5301	0.052*
H21B	0.6884	0.8311	0.4435	0.052*
C22	0.7485 (2)	1.0009 (2)	0.39619 (16)	0.0507 (6)
H22A	0.6717	1.0596	0.4313	0.061*
H22B	0.8326	1.0153	0.4086	0.061*
C23	0.8202 (2)	0.9608 (2)	0.22953 (16)	0.0429 (5)
H23A	0.9073	0.9722	0.2377	0.051*
H23B	0.7941	0.9924	0.1489	0.051*

C24	0.8439 (2)	0.8044 (2)	0.28301 (14)	0.0392 (5)
H24A	0.7582	0.7920	0.2717	0.047*
H24B	0.9190	0.7445	0.2475	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.02222 (18)	0.0292 (2)	0.0288 (2)	−0.00831 (15)	0.00330 (14)	−0.01012 (15)
O1	0.0457 (8)	0.0467 (8)	0.0278 (6)	−0.0231 (6)	0.0062 (6)	−0.0072 (6)
O2	0.0423 (7)	0.0463 (8)	0.0301 (7)	−0.0195 (6)	0.0013 (6)	−0.0002 (6)
O3	0.0338 (6)	0.0393 (8)	0.0407 (7)	−0.0212 (6)	0.0021 (5)	−0.0155 (6)
O4	0.0379 (7)	0.0247 (7)	0.0328 (6)	−0.0029 (5)	0.0073 (5)	−0.0045 (5)
O5	0.0380 (7)	0.0413 (8)	0.0410 (7)	−0.0046 (6)	−0.0057 (6)	−0.0148 (6)
O6	0.0516 (8)	0.0381 (8)	0.0445 (8)	−0.0112 (7)	0.0051 (7)	−0.0067 (6)
N1	0.0259 (7)	0.0243 (8)	0.0247 (7)	−0.0097 (6)	−0.0015 (6)	−0.0048 (6)
C1	0.0210 (8)	0.0273 (9)	0.0251 (8)	−0.0088 (7)	0.0051 (6)	−0.0105 (7)
C2	0.0257 (8)	0.0295 (10)	0.0275 (8)	−0.0103 (7)	0.0059 (7)	−0.0096 (7)
C3	0.0354 (10)	0.0285 (10)	0.0403 (10)	−0.0138 (8)	0.0148 (8)	−0.0131 (8)
C4	0.0376 (10)	0.0376 (11)	0.0567 (12)	−0.0226 (9)	0.0221 (9)	−0.0290 (10)
C5	0.0291 (9)	0.0478 (12)	0.0447 (11)	−0.0168 (9)	0.0091 (8)	−0.0295 (9)
C6	0.0271 (8)	0.0341 (10)	0.0303 (9)	−0.0093 (7)	0.0027 (7)	−0.0138 (8)
C7	0.0272 (8)	0.0276 (9)	0.0200 (8)	−0.0101 (7)	0.0044 (6)	−0.0100 (7)
C8	0.0309 (9)	0.0327 (10)	0.0272 (8)	−0.0145 (8)	0.0037 (7)	−0.0107 (7)
C9	0.0496 (11)	0.0315 (10)	0.0297 (9)	−0.0212 (9)	0.0084 (8)	−0.0097 (8)
C10	0.0498 (11)	0.0252 (10)	0.0290 (9)	−0.0072 (9)	0.0024 (8)	−0.0080 (7)
C11	0.0309 (9)	0.0336 (11)	0.0311 (9)	−0.0024 (8)	−0.0005 (7)	−0.0125 (8)
C12	0.0273 (8)	0.0328 (10)	0.0281 (8)	−0.0113 (7)	0.0051 (7)	−0.0117 (7)
C13	0.0433 (11)	0.0502 (13)	0.0381 (10)	−0.0249 (10)	0.0028 (9)	−0.0129 (9)
C14	0.0380 (10)	0.0400 (12)	0.0398 (10)	−0.0109 (9)	−0.0039 (8)	−0.0084 (9)
C15	0.0379 (10)	0.0422 (12)	0.0332 (9)	−0.0171 (9)	0.0020 (8)	−0.0077 (8)
C16	0.0403 (10)	0.0331 (11)	0.0294 (9)	−0.0109 (8)	0.0015 (8)	−0.0072 (8)
C17	0.0466 (11)	0.0386 (12)	0.0405 (10)	−0.0233 (9)	−0.0045 (9)	−0.0081 (9)
C18	0.0341 (9)	0.0324 (11)	0.0475 (11)	−0.0127 (8)	0.0035 (8)	−0.0165 (9)
C19	0.0388 (10)	0.0320 (11)	0.0302 (9)	−0.0044 (8)	0.0078 (8)	−0.0037 (8)
C20	0.0330 (9)	0.0313 (10)	0.0337 (9)	−0.0132 (8)	0.0081 (7)	−0.0134 (8)
C21	0.0491 (12)	0.0400 (12)	0.0324 (10)	−0.0091 (9)	0.0039 (9)	−0.0119 (9)
C22	0.0660 (14)	0.0420 (13)	0.0418 (11)	−0.0167 (11)	0.0051 (10)	−0.0177 (10)
C23	0.0393 (11)	0.0545 (13)	0.0372 (10)	−0.0230 (10)	0.0071 (8)	−0.0140 (9)
C24	0.0355 (10)	0.0505 (13)	0.0345 (10)	−0.0160 (9)	0.0029 (8)	−0.0196 (9)

Geometric parameters (Å, °)

K1—O3	2.6659 (12)	C8—C9	1.383 (2)
K1—O4	2.7478 (12)	C8—H8A	0.9500
K1—O1	2.7518 (12)	C9—C10	1.379 (3)
K1—N1	2.7899 (13)	C9—H9A	0.9500
K1—N1^{i}	2.9022 (14)	C10—C11	1.386 (3)
K1—C2	3.2154 (17)	C10—H10A	0.9500

K1—C1	3.3009 (16)	C11—C12	1.381 (2)
K1—C1^{i}	3.3953 (16)	C11—H11A	0.9500
K1—C2^{i}	3.4006 (18)	C12—H12A	0.9500
K1—C7^{i}	3.4257 (15)	C13—C14	1.499 (3)
K1—K1^{i}	4.2249 (7)	C13—H13A	0.9900
O1—C16	1.429 (2)	C13—H13B	0.9900
O1—C13	1.439 (2)	C14—H14A	0.9900
O2—C14	1.425 (2)	C14—H14B	0.9900
O2—C15	1.424 (2)	C15—C16	1.496 (3)
O3—C17	1.427 (2)	C15—H15A	0.9900
O3—C18	1.427 (2)	C15—H15B	0.9900
O4—C20	1.419 (2)	C16—H16A	0.9900
O4—C19	1.436 (2)	C16—H16B	0.9900
O5—C21	1.420 (2)	C17—C18^{ii}	1.501 (3)
O5—C24	1.422 (2)	C17—H17A	0.9900
O6—C23	1.424 (2)	C17—H17B	0.9900
O6—C22	1.428 (2)	C18—C17^{ii}	1.501 (3)
N1—C1	1.376 (2)	C18—H18A	0.9900
N1—C7	1.383 (2)	C18—H18B	0.9900
N1—K1^{i}	2.9022 (14)	C19—C20^{iii}	1.506 (2)
C1—C6	1.418 (2)	C19—H19A	0.9900
C1—C2	1.419 (2)	C19—H19B	0.9900
C1—K1^{i}	3.3953 (16)	C20—C19^{iii}	1.506 (2)
C2—C3	1.384 (3)	C20—H20A	0.9900
C2—K1^{i}	3.4005 (18)	C20—H20B	0.9900
C2—H2A	0.9500	C21—C22	1.493 (3)
C3—C4	1.387 (3)	C21—H21A	0.9900
C3—H3A	0.9500	C21—H21B	0.9900
C4—C5	1.393 (3)	C22—H22A	0.9900
C4—H4A	0.9500	C22—H22B	0.9900
C5—C6	1.384 (3)	C23—C24	1.497 (3)
C5—H5A	0.9500	C23—H23A	0.9900
C6—H6A	0.9500	C23—H23B	0.9900
C7—C12	1.412 (2)	C24—H24A	0.9900
C7—C8	1.415 (2)	C24—H24B	0.9900
C7—K1^{i}	3.4257 (15)		
O3—K1—O4	81.76 (4)	C6—C5—C4	121.50 (17)
O3—K1—O1	77.02 (4)	C6—C5—H5A	119.3
O4—K1—O1	86.30 (4)	C4—C5—H5A	119.3
O3—K1—N1	138.80 (4)	C5—C6—C1	121.69 (16)
O4—K1—N1	136.28 (4)	C5—C6—H6A	119.2
O1—K1—N1	88.65 (4)	C1—C6—H6A	119.2
O3—K1—N1^{i}	97.79 (4)	N1—C7—C12	125.56 (15)
O4—K1—N1^{i}	110.13 (4)	N1—C7—C8	118.58 (14)
O1—K1—N1^{i}	162.11 (4)	C12—C7—C8	115.63 (15)
N1—K1—N1^{i}	84.17 (4)	N1—C7—K1^{i}	56.53 (8)

O3—K1—C2	172.89 (4)	C12—C7—K1^{i}	97.44 (10)
O4—K1—C2	95.44 (4)	C8—C7—K1^{i}	113.50 (10)
O1—K1—C2	109.43 (4)	C9—C8—C7	121.84 (16)
N1—K1—C2	46.22 (4)	C9—C8—H8A	119.1
N1^{i}—K1—C2	76.96 (4)	C7—C8—H8A	119.1
O3—K1—C1	161.37 (4)	C10—C9—C8	121.11 (18)
O4—K1—C1	112.45 (4)	C10—C9—H9A	119.4
O1—K1—C1	91.47 (4)	C8—C9—H9A	119.4
N1—K1—C1	24.31 (4)	C9—C10—C11	118.45 (17)
N1^{i}—K1—C1	88.71 (4)	C9—C10—H10A	120.8
C2—K1—C1	25.11 (4)	C11—C10—H10A	120.8
O3—K1—C1^{i}	79.22 (4)	C12—C11—C10	121.14 (17)
O4—K1—C1^{i}	121.11 (4)	C12—C11—H11A	119.4
O1—K1—C1^{i}	140.27 (4)	C10—C11—H11A	119.4
N1—K1—C1^{i}	88.71 (4)	C11—C12—C7	121.84 (17)
N1^{i}—K1—C1^{i}	23.62 (4)	C11—C12—H12A	119.1
C2—K1—C1^{i}	96.83 (4)	C7—C12—H12A	119.1
C1—K1—C1^{i}	101.77 (4)	O1—C13—C14	110.72 (16)
O3—K1—C2^{i}	78.16 (4)	O1—C13—H13A	109.5
O4—K1—C2^{i}	142.84 (4)	C14—C13—H13A	109.5
O1—K1—C2^{i}	118.58 (4)	O1—C13—H13B	109.5
N1—K1—C2^{i}	75.36 (4)	C14—C13—H13B	109.5
N1^{i}—K1—C2^{i}	43.66 (4)	H13A—C13—H13B	108.1
C2—K1—C2^{i}	100.68 (4)	O2—C14—C13	110.90 (15)
C1—K1—C2^{i}	95.12 (4)	O2—C14—H14A	109.5
C1^{i}—K1—C2^{i}	24.10 (4)	C13—C14—H14A	109.5
O3—K1—C7^{i}	95.50 (4)	O2—C14—H14B	109.5
O4—K1—C7^{i}	86.71 (4)	C13—C14—H14B	109.5
O1—K1—C7^{i}	170.42 (4)	H14A—C14—H14B	108.0
N1—K1—C7^{i}	100.93 (4)	O2—C15—C16	110.51 (16)
N1^{i}—K1—C7^{i}	23.43 (4)	O2—C15—H15A	109.5
C2—K1—C7^{i}	77.77 (4)	C16—C15—H15A	109.5
C1—K1—C7^{i}	97.25 (4)	O2—C15—H15B	109.5
C1^{i}—K1—C7^{i}	41.30 (4)	C16—C15—H15B	109.5
C2^{i}—K1—C7^{i}	64.71 (4)	H15A—C15—H15B	108.1
O3—K1—K1^{i}	126.16 (3)	O1—C16—C15	110.88 (15)
O4—K1—K1^{i}	135.53 (3)	O1—C16—H16A	109.5
O1—K1—K1^{i}	129.65 (3)	C15—C16—H16A	109.5
N1—K1—K1^{i}	43.11 (3)	O1—C16—H16B	109.5
N1^{i}—K1—K1^{i}	41.07 (3)	C15—C16—H16B	109.5
C2—K1—K1^{i}	52.27 (3)	H16A—C16—H16B	108.1
C1—K1—K1^{i}	51.88 (3)	O3—C17—C18^{ii}	110.80 (15)

C1^{i}—K1—K1^{i}	49.89 (3)	O3—C17—H17A	109.5
C2^{i}—K1—K1^{i}	48.41 (3)	C18^{ii}—C17—H17A	109.5
C7^{i}—K1—K1^{i}	59.66 (3)	O3—C17—H17B	109.5
C16—O1—C13	109.81 (13)	C18^{ii}—C17—H17B	109.5
C16—O1—K1	126.57 (9)	H17A—C17—H17B	108.1
C13—O1—K1	123.22 (10)	O3—C18—C17^{ii}	110.30 (15)
C14—O2—C15	109.87 (13)	O3—C18—H18A	109.6
C17—O3—C18	109.81 (13)	C17^{ii}—C18—H18A	109.6
C17—O3—K1	124.11 (10)	O3—C18—H18B	109.6
C18—O3—K1	125.98 (10)	C17^{ii}—C18—H18B	109.6
C20—O4—C19	109.49 (13)	H18A—C18—H18B	108.1
C20—O4—K1	118.01 (10)	O4—C19—C20^{iii}	111.02 (14)
C19—O4—K1	131.87 (10)	O4—C19—H19A	109.4
C21—O5—C24	109.73 (13)	C20^{iii}—C19—H19A	109.4
C23—O6—C22	109.40 (15)	O4—C19—H19B	109.4
C1—N1—C7	121.35 (13)	C20^{iii}—C19—H19B	109.4
C1—N1—K1	99.13 (9)	H19A—C19—H19B	108.0
C7—N1—K1	133.05 (10)	O4—C20—C19^{iii}	110.36 (15)
C1—N1—K1^{i}	98.73 (9)	O4—C20—H20A	109.6
C7—N1—K1^{i}	100.04 (9)	C19^{iii}—C20—H20A	109.6
K1—N1—K1^{i}	95.83 (4)	O4—C20—H20B	109.6
N1—C1—C6	126.92 (15)	C19^{iii}—C20—H20B	109.6
N1—C1—C2	117.45 (14)	H20A—C20—H20B	108.1
C6—C1—C2	115.41 (16)	O5—C21—C22	111.12 (16)
N1—C1—K1	56.56 (7)	O5—C21—H21A	109.4
C6—C1—K1	139.36 (11)	C22—C21—H21A	109.4
C2—C1—K1	74.07 (9)	O5—C21—H21B	109.4
N1—C1—K1^{i}	57.66 (8)	C22—C21—H21B	109.4
C6—C1—K1^{i}	141.23 (11)	H21A—C21—H21B	108.0
C2—C1—K1^{i}	78.15 (9)	O6—C22—C21	111.51 (17)
K1—C1—K1^{i}	78.23 (4)	O6—C22—H22A	109.3
C3—C2—C1	122.31 (16)	C21—C22—H22A	109.3
C3—C2—K1	134.02 (11)	O6—C22—H22B	109.3
C1—C2—K1	80.82 (10)	C21—C22—H22B	109.3
C3—C2—K1^{i}	139.91 (11)	H22A—C22—H22B	108.0
C1—C2—K1^{i}	77.74 (10)	O6—C23—C24	110.94 (15)
K1—C2—K1^{i}	79.32 (4)	O6—C23—H23A	109.5
C3—C2—H2A	118.8	C24—C23—H23A	109.5
C1—C2—H2A	118.8	O6—C23—H23B	109.5
K1—C2—H2A	56.3	C24—C23—H23B	109.5
K1^{i}—C2—H2A	55.1	H23A—C23—H23B	108.0
C2—C3—C4	120.98 (17)	O5—C24—C23	110.51 (16)
C2—C3—H3A	119.5	O5—C24—H24A	109.5
C4—C3—H3A	119.5	C23—C24—H24A	109.5

C3—C4—C5	118.11 (18)	O5—C24—H24B	109.5
C3—C4—H4A	120.9	C23—C24—H24B	109.5
C5—C4—H4A	120.9	H24A—C24—H24B	108.1

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

Fig. 1

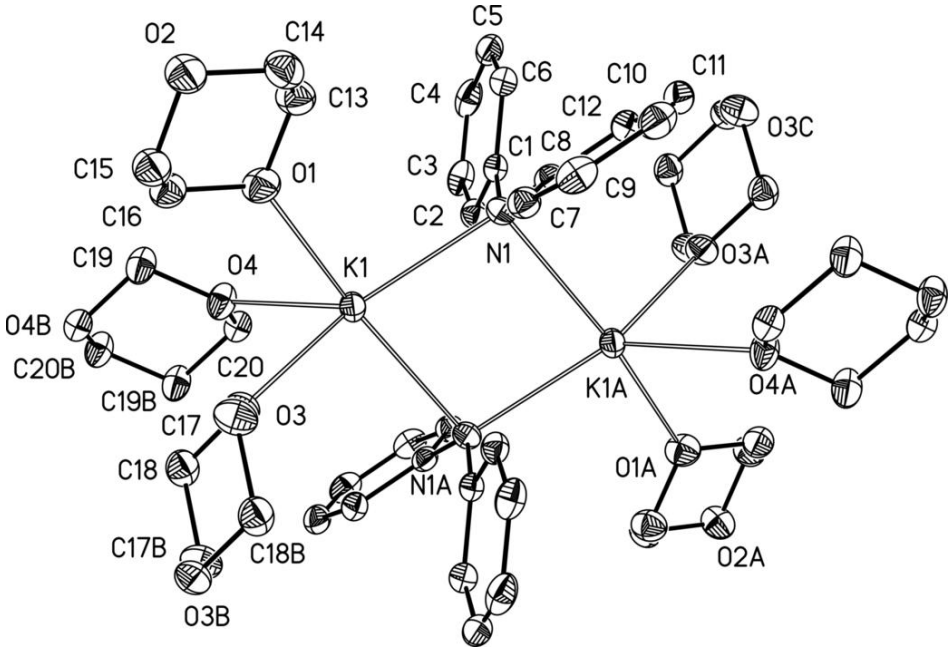

O2
C14
C5
C6
C10
C11
C13
C4
C12
O3C
C1
C8
C15
O1
C3
C9
C16
C2
C7
N1
C19
O4
K1
O3A
O4B
C20B
C20
O4A
C19B
K1A
C17
O3
C18
N1A
O1A
C17B
C18B
O2A
O3B

Fig. 2