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Poly[potassium- μ -2-[2-(carboxymethyl)-phenyl]acetato]Reyes García-Zarracino,^a Marcela Rangel-Marrón,^a Hugo Tlahuext^{b*} and Herbert Höpfl^b

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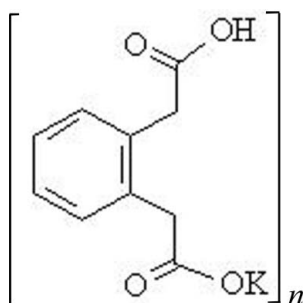
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.041; wR factor = 0.101; data-to-parameter ratio = 12.4.

In the title salt, $[\text{K}(\text{C}_{10}\text{H}_9\text{O}_4)]_n$, the K^+ ions are coordinated by six O atoms from three different anions, and there is a cation- π interaction at *ca* 3.14 Å. The 2-[2-(carboxymethyl)phenyl]acetate anions are stabilized by intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, and the K^+ cations are linked into one-dimensional coordination polymers running along the b axis; these are further interconnected by weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For general background, see: Atwood & Steed (2004); Ma & Dougherty (1997); Kumpf & Dougherty (1993); Heginbotham *et al.* (1994). For coordination polymers, see: Chae *et al.* (2004); García-Zarracino *et al.* (2003); García-Zarracino & Höpfl (2004). For analysis of hydrogen-bonding patterns, see: Bernstein *et al.* (1995); Desiraju (2002).



Experimental

Crystal data

$[\text{K}(\text{C}_{10}\text{H}_9\text{O}_4)]$
 $M_r = 232.27$

Monoclinic, $P2_1/c$
 $a = 8.3365$ (14) Å

$b = 6.7886$ (11) Å
 $c = 17.651$ (3) Å
 $\beta = 92.543$ (3)°
 $V = 997.9$ (3) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.52$ mm⁻¹
 $T = 293$ (2) K
 $0.45 \times 0.30 \times 0.28$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.713$, $T_{\max} = 0.864$

8711 measured reflections
1727 independent reflections
1560 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.101$
 $S = 1.08$
1727 reflections
139 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{O4}-\text{H4}^i\cdots\text{O1}^i$ | 1.01 (3) | 2.57 (3) | 3.248 (2) | 125 (2) |
| $\text{O4}-\text{H4}^i\cdots\text{O2}^i$ | 1.01 (3) | 1.47 (3) | 2.471 (2) | 176 (3) |
| $\text{C2}-\text{H2A}^i\cdots\text{O2}^{ii}$ | 0.97 | 2.53 | 3.480 (3) | 167 |

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT-Plus-NT (Bruker, 2001); data reduction: SAINT-Plus-NT; program(s) used to solve structure: SHELXTL-NT (Sheldrick, 2008); program(s) used to refine structure: SHELXTL-NT; molecular graphics: SHELXTL-NT; software used to prepare material for publication: PLATON (Spek, 2003) and publCIF (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2283).

References

- Atwood, J. L. & Steed, J. W. (2004). Editors. *Encyclopedia of Supramolecular Chemistry*. Boca Raton: CRC Press, Taylor & Francis Group.
- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2000). SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2001). SAINT-Plus-NT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chae, H. K., Siberio-Peréz, D. Y., Kim, J., Go, Y., Eddaoudi, M., Matzger, J., O'Keefe, M. & Yaghi, O. M. (2004). *Nature (London)*, **427**, 523–527.
- Desiraju, G. R. (2002). *Acc. Chem. Res.* **35**, 565–573.
- García-Zarracino, R. & Höpfl, H. (2004). *Angew. Chem. Int. Ed.* **43**, 1507–1511.
- García-Zarracino, R., Ramos-Quiñones, J. & Höpfl, H. (2003). *Inorg. Chem.* **42**, 3835–3845.
- Heginbotham, L., Lu, Z., Abramson, T. & MacKinnon, R. (1994). *Biophys. J.* **66**, 1061–1067.
- Kumpf, R. A. & Dougherty, D. A. (1993). *Science*, **261**, 1708–1710.
- Ma, J. C. & Dougherty, D. A. (1997). *Chem. Rev.* **97**, 1303–1324.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Westrip, S. P. (2008). publCIF. In preparation.

supplementary materials

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Poly[potassium- μ -2-[2-(carboxymethyl)phenyl]acetato]

R. García-Zarracino, M. Rangel-Marrón, H. Tlahuext and H. Höpfl

Comment

Hydrogen bonding, cation- π , and π - π interactions are principal forces which determine the structure, self-assembly and recognition in many chemical and biological systems (Atwood & Steed, 2004). Cation- π interactions are now recognized as important non-covalent binding forces in biological systems (Ma & Dougherty, 1997). It has been postulated that the aromatic side chains of amino acids might determine K^+ transport selectivity in transmembrane protein channels (Kumpf & Dougherty, 1993; Heginbotham *et al.*, 1994).

In complex **I** each K^+ ion is coordinated by six oxygen atoms from three different ligand molecules and there is a cation- π interaction (Fig.1) forming a distorted square-face monocapped prism (Fig. 2). The centroid of the aryl ring (C_g , C3—C8) is situated 3.138 Å from the K^+ ion [sum of ionic and van der Waals radii $K^+\cdots C(Ar)$ 3.37 Å]. Since the K — C_{arene} contacts [C3—K = 3.344 (2), C4—K = 3.260 (2) and C5—K = 3.334 (2) Å] and [C6—K = 3.508 (2), C7—K = 3.596, C8—K = 3.511 (2)] the K -arene interaction can be regarded as η^3 -coordination. The oxygen atoms of the carboxyl and carboxylate groups are forming bridging units between two K^+ cations, thus generating a one-dimensional coordination polymer, running along the b axis (Chae *et al.*, 2004; García-Zarracino *et al.*, 2003; García-Zarracino & Höpfl, 2004) (Fig. 2). The coordination polymer is stabilized by intramolecular O4—H4' \cdots O2 hydrogen bonds. The crystal structure is stabilized by weak C—H \cdots O hydrogen bonds forming $R_2^2(8)$ motifs, (Bernstein *et al.*, 1995; Desiraju, 2002) between adjacent coordination polymers (Fig. 3, Table 1).

Experimental

Single Crystals of (I) were obtained by slow evaporation of a solution containing 1,2-phenylenediacetic acid (1.0 g, 5.15 mmol), potassium hydroxide (0.289 g, 5.15 mmol) in MeOH/H₂O (5:1).

Refinement

Aromatic and methylene H atoms were positioned geometrically and constrained using the riding-model approximation [C -H_{aryl} = 0.93 Å, $U_{iso}(H_{aryl}) = 1.2 U_{eq}(C_{aryl})$; C -H_{methylene} = 0.97 Å, $U_{iso}(H_{methylene}) = 1.5 U_{eq}(C_{methylene})$]. Atom H4', which is involved in a hydrogen-bonding interaction, was located by difference Fourier map, constrained using the riding-model approximation [$U_{iso}(O4-H') = 1.5 U_{eq}(O4)$] and the coordinates were refined freely.

Figures

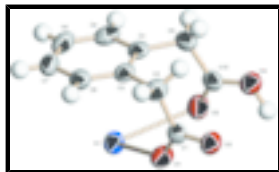


Fig. 1. Asymmetric unit of **I** showing 50% probability displacement ellipsoids and the atomic numbering. H atoms are shown as small spheres of arbitrary radius.

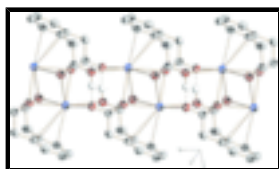


Fig. 2. View of the one-dimensional coordination polymer chain running along to the *b* axis. Dashed and dotted lines indicate C_{aryl}...K⁺ and O—H...O contacts, respectively. H atoms not involved in hydrogen-bonding have been omitted for clarity.

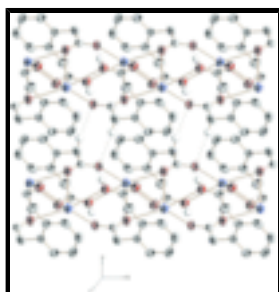


Fig. 3. View of the C—H...O hydrogen bonds forming $R_2^2(8)$ motifs between adjacent coordination polymers. The hydrogen bonds are represented by dotted lines and H atoms not involved in hydrogen-bonding have been omitted for clarity.

Poly[potassium- μ -2-[2-(carboxymethyl)phenyl]acetato]

Crystal data

[K(C₁₀H₉O₄)]

$M_r = 232.27$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.3365$ (14) Å

$b = 6.7886$ (11) Å

$c = 17.651$ (3) Å

$\beta = 92.543$ (3)°

$V = 997.9$ (3) Å³

$Z = 4$

$F_{000} = 480$

$D_x = 1.546$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4833 reflections

$\theta = 2.3$ – 28.2 °

$\mu = 0.52$ mm⁻¹

$T = 293$ (2) K

Rectangular, colourless

$0.45 \times 0.30 \times 0.28$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

1727 independent reflections

1560 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.3$ °

$h = -9 \rightarrow 9$

$T_{\min} = 0.713$, $T_{\max} = 0.864$
8711 measured reflections

$k = -8 \rightarrow 8$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.041$

H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.101$

$$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.3852P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$S = 1.08$

$$(\Delta/\sigma)_{\max} = 0.001$$

1727 reflections

$$\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$$

139 parameters

$$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|--------------|----------------------------------|
| K1 | 0.38073 (6) | 0.33470 (7) | 0.44502 (3) | 0.0455 (2) |
| O1 | 0.2998 (2) | 0.6914 (2) | 0.51210 (9) | 0.0487 (4) |
| O2 | 0.26242 (19) | 1.0124 (2) | 0.52043 (9) | 0.0449 (4) |
| O3 | 0.5412 (2) | 0.6608 (2) | 0.38998 (11) | 0.0569 (5) |
| O4 | 0.5243 (2) | 0.9834 (2) | 0.37776 (9) | 0.0471 (4) |
| H4' | 0.612 (4) | 0.991 (4) | 0.4186 (16) | 0.071* |
| C1 | 0.2360 (3) | 0.8431 (3) | 0.49014 (12) | 0.0350 (5) |
| C2 | 0.1154 (3) | 0.8450 (3) | 0.42353 (13) | 0.0449 (6) |
| H2A | 0.0091 | 0.8637 | 0.4426 | 0.054* |
| H2B | 0.1379 | 0.9572 | 0.3917 | 0.054* |
| C3 | 0.1135 (3) | 0.6626 (3) | 0.37550 (12) | 0.0362 (5) |
| C4 | 0.2223 (3) | 0.6353 (3) | 0.31858 (12) | 0.0378 (5) |
| C5 | 0.2138 (3) | 0.4620 (4) | 0.27695 (12) | 0.0458 (6) |
| H5 | 0.2859 | 0.4419 | 0.2389 | 0.055* |
| C6 | 0.1017 (3) | 0.3192 (4) | 0.29041 (14) | 0.0495 (6) |

supplementary materials

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|-----|-------------|------------|--------------|------------|
| H6 | 0.0988 | 0.2039 | 0.2619 | 0.059* |
| C7 | -0.0052 (3) | 0.3471 (3) | 0.34570 (14) | 0.0498 (6) |
| H7 | -0.0816 | 0.2512 | 0.3549 | 0.060* |
| C8 | 0.0006 (3) | 0.5179 (3) | 0.38770 (13) | 0.0434 (6) |
| H8 | -0.0730 | 0.5365 | 0.4251 | 0.052* |
| C9 | 0.3453 (3) | 0.7892 (4) | 0.30161 (13) | 0.0497 (6) |
| H9A | 0.2919 | 0.9158 | 0.2963 | 0.060* |
| H9B | 0.3909 | 0.7583 | 0.2534 | 0.060* |
| C10 | 0.4803 (3) | 0.8065 (3) | 0.36163 (12) | 0.0393 (5) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| K1 | 0.0465 (3) | 0.0342 (3) | 0.0548 (3) | 0.0001 (2) | -0.0082 (2) | 0.0024 (2) |
| O1 | 0.0540 (10) | 0.0367 (9) | 0.0542 (10) | 0.0060 (8) | -0.0139 (8) | -0.0016 (7) |
| O2 | 0.0498 (10) | 0.0346 (8) | 0.0497 (9) | 0.0011 (7) | -0.0047 (7) | -0.0062 (7) |
| O3 | 0.0503 (10) | 0.0450 (10) | 0.0735 (12) | -0.0046 (8) | -0.0172 (9) | 0.0114 (9) |
| O4 | 0.0466 (10) | 0.0432 (9) | 0.0508 (9) | -0.0003 (7) | -0.0053 (8) | 0.0004 (7) |
| C1 | 0.0315 (11) | 0.0362 (12) | 0.0379 (11) | -0.0003 (9) | 0.0064 (9) | 0.0003 (9) |
| C2 | 0.0455 (13) | 0.0393 (13) | 0.0492 (13) | 0.0099 (10) | -0.0077 (11) | -0.0032 (10) |
| C3 | 0.0352 (11) | 0.0354 (11) | 0.0372 (11) | 0.0058 (9) | -0.0090 (9) | -0.0001 (9) |
| C4 | 0.0335 (11) | 0.0442 (12) | 0.0350 (11) | 0.0027 (9) | -0.0077 (9) | 0.0037 (9) |
| C5 | 0.0416 (13) | 0.0596 (15) | 0.0357 (12) | 0.0124 (11) | -0.0051 (10) | -0.0076 (11) |
| C6 | 0.0541 (15) | 0.0428 (14) | 0.0498 (14) | 0.0022 (11) | -0.0170 (12) | -0.0106 (11) |
| C7 | 0.0502 (14) | 0.0435 (13) | 0.0542 (15) | -0.0105 (11) | -0.0139 (12) | 0.0053 (11) |
| C8 | 0.0387 (13) | 0.0508 (14) | 0.0405 (12) | -0.0034 (10) | -0.0013 (10) | 0.0031 (10) |
| C9 | 0.0455 (14) | 0.0583 (15) | 0.0446 (13) | -0.0076 (12) | -0.0052 (11) | 0.0133 (11) |
| C10 | 0.0339 (12) | 0.0448 (14) | 0.0395 (12) | -0.0037 (10) | 0.0056 (9) | 0.0053 (10) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------------------|-------------|--------|-----------|
| K1—O1 ⁱ | 2.7422 (17) | O4—H4' | 1.00 (3) |
| K1—O2 ⁱⁱ | 2.7654 (17) | C1—C2 | 1.513 (3) |
| K1—O3 | 2.7837 (18) | C2—C3 | 1.500 (3) |
| K1—O1 | 2.7912 (16) | C2—H2A | 0.9700 |
| K1—O4 ⁱⁱ | 2.9419 (17) | C2—H2B | 0.9700 |
| K1—O3 ⁱ | 2.956 (2) | C3—C8 | 1.384 (3) |
| K1—C4 | 3.260 (2) | C3—C4 | 1.396 (3) |
| K1—C5 | 3.334 (2) | C4—C5 | 1.387 (3) |
| K1—C3 | 3.344 (2) | C4—C9 | 1.503 (3) |
| K1—C6 | 3.508 (2) | C5—C6 | 1.374 (3) |
| K1—C8 | 3.511 (2) | C5—H5 | 0.9300 |
| K1—K1 ⁱ | 3.5235 (10) | C6—C7 | 1.364 (4) |
| O1—C1 | 1.215 (2) | C6—H6 | 0.9300 |
| O1—K1 ⁱ | 2.7422 (17) | C7—C8 | 1.376 (3) |
| O2—C1 | 1.282 (2) | C7—H7 | 0.9300 |
| O2—K1 ⁱⁱⁱ | 2.7654 (17) | C8—H8 | 0.9300 |

| | | | |
|---------------------------------------|-------------|---------------------------|-------------|
| O3—C10 | 1.210 (3) | C9—C10 | 1.515 (3) |
| O3—K1 ⁱ | 2.956 (2) | C9—H9A | 0.9700 |
| O4—C10 | 1.284 (3) | C9—H9B | 0.9700 |
| O4—K1 ⁱⁱⁱ | 2.9420 (17) | | |
| O1 ⁱ —K1—O2 ⁱⁱ | 100.31 (5) | C8—K1—K1 ⁱ | 114.41 (4) |
| O1 ⁱ —K1—O3 | 70.84 (5) | C1—O1—K1 ⁱ | 123.28 (14) |
| O2 ⁱⁱ —K1—O3 | 169.83 (5) | C1—O1—K1 | 135.31 (14) |
| O1 ⁱ —K1—O1 | 100.90 (5) | K1 ⁱ —O1—K1 | 79.10 (5) |
| O2 ⁱⁱ —K1—O1 | 112.58 (5) | C1—O2—K1 ⁱⁱⁱ | 124.52 (13) |
| O3—K1—O1 | 65.62 (6) | C10—O3—K1 | 126.51 (15) |
| O1 ⁱ —K1—O4 ⁱⁱ | 69.61 (5) | C10—O3—K1 ⁱ | 117.98 (15) |
| O2 ⁱⁱ —K1—O4 ⁱⁱ | 73.33 (5) | K1—O3—K1 ⁱ | 75.67 (5) |
| O3—K1—O4 ⁱⁱ | 107.07 (6) | C10—O4—K1 ⁱⁱⁱ | 136.90 (14) |
| O1—K1—O4 ⁱⁱ | 169.97 (5) | C10—O4—H4' | 113.6 (16) |
| O1 ⁱ —K1—O3 ⁱ | 63.90 (5) | K1 ⁱⁱⁱ —O4—H4' | 87.9 (16) |
| O2 ⁱⁱ —K1—O3 ⁱ | 66.52 (5) | O1—C1—O2 | 124.2 (2) |
| O3—K1—O3 ⁱ | 104.32 (5) | O1—C1—C2 | 121.41 (19) |
| O1—K1—O3 ⁱ | 67.66 (5) | O2—C1—C2 | 114.38 (18) |
| O4 ⁱⁱ —K1—O3 ⁱ | 109.28 (5) | C3—C2—C1 | 114.97 (18) |
| O1 ⁱ —K1—C4 | 126.04 (6) | C3—C2—H2A | 108.5 |
| O2 ⁱⁱ —K1—C4 | 133.04 (5) | C1—C2—H2A | 108.5 |
| O3—K1—C4 | 56.60 (5) | C3—C2—H2B | 108.5 |
| O1—K1—C4 | 69.46 (5) | C1—C2—H2B | 108.5 |
| O4 ⁱⁱ —K1—C4 | 113.01 (5) | H2A—C2—H2B | 107.5 |
| O3 ⁱ —K1—C4 | 137.12 (5) | C8—C3—C4 | 119.1 (2) |
| O1 ⁱ —K1—C5 | 128.62 (6) | C8—C3—C2 | 119.2 (2) |
| O2 ⁱⁱ —K1—C5 | 119.32 (6) | C4—C3—C2 | 121.7 (2) |
| O3—K1—C5 | 70.82 (6) | C8—C3—K1 | 85.25 (13) |
| O1—K1—C5 | 93.06 (5) | C4—C3—K1 | 74.46 (11) |
| O4 ⁱⁱ —K1—C5 | 90.70 (5) | C2—C3—K1 | 110.62 (12) |
| O3 ⁱ —K1—C5 | 159.86 (6) | C5—C4—C3 | 118.3 (2) |
| C4—K1—C5 | 24.26 (6) | C5—C4—C9 | 120.2 (2) |
| O1 ⁱ —K1—C3 | 140.12 (5) | C3—C4—C9 | 121.5 (2) |
| O2 ⁱⁱ —K1—C3 | 117.21 (5) | C5—C4—K1 | 80.87 (12) |
| O3—K1—C3 | 70.43 (5) | C3—C4—K1 | 81.18 (12) |
| O1—K1—C3 | 53.74 (5) | C9—C4—K1 | 108.22 (13) |
| O4 ⁱⁱ —K1—C3 | 131.90 (5) | C6—C5—C4 | 121.7 (2) |
| O3 ⁱ —K1—C3 | 117.95 (5) | C6—C5—K1 | 85.62 (14) |
| C4—K1—C3 | 24.36 (5) | C4—C5—K1 | 74.87 (12) |
| C5—K1—C3 | 41.93 (5) | C6—C5—H5 | 119.1 |
| O1 ⁱ —K1—C6 | 144.60 (6) | C4—C5—H5 | 119.1 |
| O2 ⁱⁱ —K1—C6 | 96.45 (6) | K1—C5—H5 | 110.3 |
| O3—K1—C6 | 93.71 (6) | C7—C6—C5 | 119.8 (2) |

supplementary materials

| | | | |
|---|-------------|----------------------------|--------------|
| O1—K1—C6 | 101.04 (5) | C7—C6—K1 | 82.60 (14) |
| O4 ⁱⁱ —K1—C6 | 85.95 (5) | C5—C6—K1 | 71.39 (13) |
| O3 ⁱ —K1—C6 | 151.19 (6) | C7—C6—H6 | 120.1 |
| C4—K1—C6 | 41.56 (6) | C5—C6—H6 | 120.1 |
| C5—K1—C6 | 23.00 (6) | K1—C6—H6 | 116.5 |
| C3—K1—C6 | 47.66 (5) | C6—C7—C8 | 119.6 (2) |
| O1 ⁱ —K1—C8 | 162.86 (5) | C6—C7—K1 | 75.30 (14) |
| O2 ⁱⁱ —K1—C8 | 94.72 (5) | C8—C7—K1 | 75.38 (13) |
| O3—K1—C8 | 93.40 (5) | C6—C7—H7 | 120.2 |
| O1—K1—C8 | 65.34 (5) | C8—C7—H7 | 120.2 |
| O4 ⁱⁱ —K1—C8 | 123.29 (5) | K1—C7—H7 | 120.2 |
| O3 ⁱ —K1—C8 | 115.89 (5) | C7—C8—C3 | 121.5 (2) |
| C4—K1—C8 | 41.24 (5) | C7—C8—K1 | 82.34 (14) |
| C5—K1—C8 | 46.86 (6) | C3—C8—K1 | 71.62 (12) |
| C3—K1—C8 | 23.13 (5) | C7—C8—H8 | 119.3 |
| C6—K1—C8 | 39.43 (6) | C3—C8—H8 | 119.3 |
| O1 ⁱ —K1—K1 ⁱ | 51.07 (3) | K1—C8—H8 | 117.3 |
| O2 ⁱⁱ —K1—K1 ⁱ | 116.32 (4) | C4—C9—C10 | 113.96 (18) |
| O3—K1—K1 ⁱ | 54.37 (4) | C4—C9—H9A | 108.8 |
| O1—K1—K1 ⁱ | 49.84 (4) | C10—C9—H9A | 108.8 |
| O4 ⁱⁱ —K1—K1 ⁱ | 120.59 (4) | C4—C9—H9B | 108.8 |
| O3 ⁱ —K1—K1 ⁱ | 49.95 (3) | C10—C9—H9B | 108.8 |
| C4—K1—K1 ⁱ | 100.37 (4) | H9A—C9—H9B | 107.7 |
| C5—K1—K1 ⁱ | 121.87 (5) | O3—C10—O4 | 124.2 (2) |
| C3—K1—K1 ⁱ | 97.39 (4) | O3—C10—C9 | 120.7 (2) |
| C6—K1—K1 ⁱ | 141.83 (5) | O4—C10—C9 | 115.0 (2) |
| O1 ⁱ —K1—O1—C1 | 126.9 (2) | C3—K1—C4—C9 | 120.3 (2) |
| O2 ⁱⁱ —K1—O1—C1 | -127.0 (2) | C6—K1—C4—C9 | -147.96 (19) |
| O3—K1—O1—C1 | 63.9 (2) | C8—K1—C4—C9 | 150.69 (19) |
| O4 ⁱⁱ —K1—O1—C1 | 108.4 (3) | K1 ⁱ —K1—C4—C9 | 35.31 (16) |
| O3 ⁱ —K1—O1—C1 | -177.4 (2) | C3—C4—C5—C6 | 0.2 (3) |
| C4—K1—O1—C1 | 2.3 (2) | C9—C4—C5—C6 | -179.2 (2) |
| C5—K1—O1—C1 | -3.4 (2) | K1—C4—C5—C6 | 74.9 (2) |
| C3—K1—O1—C1 | -19.0 (2) | C3—C4—C5—K1 | -74.75 (17) |
| C6—K1—O1—C1 | -25.2 (2) | C9—C4—C5—K1 | 105.83 (18) |
| C8—K1—O1—C1 | -42.4 (2) | O1 ⁱ —K1—C5—C6 | 142.02 (14) |
| K1 ⁱ —K1—O1—C1 | 126.9 (2) | O2 ⁱⁱ —K1—C5—C6 | 6.69 (16) |
| O1 ⁱ —K1—O1—K1 ⁱ | 0.0 | O3—K1—C5—C6 | -174.26 (16) |
| O2 ⁱⁱ —K1—O1—K1 ⁱ | 106.11 (5) | O1—K1—C5—C6 | -111.41 (15) |
| O3—K1—O1—K1 ⁱ | -62.98 (5) | O4 ⁱⁱ —K1—C5—C6 | 77.89 (14) |
| O4 ⁱⁱ —K1—O1—K1 ⁱ | -18.4 (3) | O3 ⁱ —K1—C5—C6 | -95.1 (2) |
| O3 ⁱ —K1—O1—K1 ⁱ | 55.70 (5) | C4—K1—C5—C6 | -124.5 (2) |
| C4—K1—O1—K1 ⁱ | -124.58 (6) | C3—K1—C5—C6 | -92.50 (16) |

| | | | |
|---|--------------|----------------------------|--------------|
| C5—K1—O1—K1 ⁱ | -130.30 (6) | C8—K1—C5—C6 | -59.92 (14) |
| C3—K1—O1—K1 ⁱ | -145.89 (8) | K1 ⁱ —K1—C5—C6 | -154.75 (13) |
| C6—K1—O1—K1 ⁱ | -152.05 (6) | O1 ⁱ —K1—C5—C4 | -93.44 (14) |
| C8—K1—O1—K1 ⁱ | -169.22 (6) | O2 ⁱⁱ —K1—C5—C4 | 131.23 (13) |
| O1 ⁱ —K1—O3—C10 | -169.7 (2) | O3—K1—C5—C4 | -49.72 (14) |
| O2 ⁱⁱ —K1—O3—C10 | -139.5 (3) | O1—K1—C5—C4 | 13.13 (14) |
| O1—K1—O3—C10 | -57.54 (19) | O4 ⁱⁱ —K1—C5—C4 | -157.57 (14) |
| O4 ⁱⁱ —K1—O3—C10 | 129.80 (19) | O3 ⁱ —K1—C5—C4 | 29.4 (3) |
| O3 ⁱ —K1—O3—C10 | -114.4 (2) | C3—K1—C5—C4 | 32.04 (12) |
| C4—K1—O3—C10 | 23.11 (18) | C6—K1—C5—C4 | 124.5 (2) |
| C5—K1—O3—C10 | 45.16 (19) | C8—K1—C5—C4 | 64.62 (13) |
| C3—K1—O3—C10 | 0.58 (19) | K1 ⁱ —K1—C5—C4 | -30.21 (15) |
| C6—K1—O3—C10 | 42.9 (2) | C4—C5—C6—C7 | 0.3 (3) |
| C8—K1—O3—C10 | 3.4 (2) | K1—C5—C6—C7 | 69.5 (2) |
| K1 ⁱ —K1—O3—C10 | -114.4 (2) | C4—C5—C6—K1 | -69.20 (19) |
| O1 ⁱ —K1—O3—K1 ⁱ | -55.29 (4) | O1 ⁱ —K1—C6—C7 | 178.97 (13) |
| O2 ⁱⁱ —K1—O3—K1 ⁱ | -25.1 (3) | O2 ⁱⁱ —K1—C6—C7 | 60.94 (15) |
| O1—K1—O3—K1 ⁱ | 56.88 (4) | O3—K1—C6—C7 | -119.50 (15) |
| O4 ⁱⁱ —K1—O3—K1 ⁱ | -115.78 (5) | O1—K1—C6—C7 | -53.63 (15) |
| O3 ⁱ —K1—O3—K1 ⁱ | 0.0 | O4 ⁱⁱ —K1—C6—C7 | 133.63 (15) |
| C4—K1—O3—K1 ⁱ | 137.53 (7) | O3 ⁱ —K1—C6—C7 | 9.7 (2) |
| C5—K1—O3—K1 ⁱ | 159.58 (6) | C4—K1—C6—C7 | -94.26 (16) |
| C3—K1—O3—K1 ⁱ | 115.00 (6) | C5—K1—C6—C7 | -124.9 (2) |
| C6—K1—O3—K1 ⁱ | 157.33 (5) | C3—K1—C6—C7 | -60.36 (14) |
| C8—K1—O3—K1 ⁱ | 117.82 (5) | C8—K1—C6—C7 | -28.65 (13) |
| K1 ⁱ —O1—C1—O2 | -42.1 (3) | K1 ⁱ —K1—C6—C7 | -89.04 (16) |
| K1—O1—C1—O2 | -152.13 (16) | O1 ⁱ —K1—C6—C5 | -56.10 (18) |
| K1 ⁱ —O1—C1—C2 | 138.33 (17) | O2 ⁱⁱ —K1—C6—C5 | -174.13 (14) |
| K1—O1—C1—C2 | 28.3 (3) | O3—K1—C6—C5 | 5.43 (15) |
| K1 ⁱⁱⁱ —O2—C1—O1 | 120.5 (2) | O1—K1—C6—C5 | 71.30 (15) |
| K1 ⁱⁱⁱ —O2—C1—C2 | -60.0 (2) | O4 ⁱⁱ —K1—C6—C5 | -101.44 (14) |
| O1—C1—C2—C3 | -15.3 (3) | O3 ⁱ —K1—C6—C5 | 134.65 (15) |
| O2—C1—C2—C3 | 165.1 (2) | C4—K1—C6—C5 | 30.67 (13) |
| C1—C2—C3—C8 | 97.3 (2) | C3—K1—C6—C5 | 64.57 (14) |
| C1—C2—C3—C4 | -83.0 (3) | C8—K1—C6—C5 | 96.28 (16) |
| C1—C2—C3—K1 | 1.1 (2) | K1 ⁱ —K1—C6—C5 | 35.89 (18) |
| O1 ⁱ —K1—C3—C8 | -172.79 (12) | C5—C6—C7—C8 | -0.2 (3) |
| O2 ⁱⁱ —K1—C3—C8 | -14.49 (15) | K1—C6—C7—C8 | 63.3 (2) |
| O3—K1—C3—C8 | 172.82 (15) | C5—C6—C7—K1 | -63.6 (2) |
| O1—K1—C3—C8 | -113.60 (15) | O1 ⁱ —K1—C7—C6 | -2.6 (3) |
| O4 ⁱⁱ —K1—C3—C8 | 77.11 (15) | O2 ⁱⁱ —K1—C7—C6 | -119.40 (15) |
| O3 ⁱ —K1—C3—C8 | -90.94 (13) | O3—K1—C7—C6 | 63.61 (15) |

supplementary materials

| | | | |
|----------------------------|--------------|----------------------------|--------------|
| C4—K1—C3—C8 | 121.97 (19) | O1—K1—C7—C6 | 127.72 (15) |
| C5—K1—C3—C8 | 90.07 (15) | O4 ⁱⁱ —K1—C7—C6 | -47.38 (15) |
| C6—K1—C3—C8 | 58.20 (14) | O3 ⁱ —K1—C7—C6 | -173.97 (13) |
| K1 ⁱ —K1—C3—C8 | -139.20 (13) | C4—K1—C7—C6 | 63.69 (15) |
| O1 ⁱ —K1—C3—C4 | 65.24 (15) | C5—K1—C7—C6 | 30.06 (14) |
| O2 ⁱⁱ —K1—C3—C4 | -136.46 (12) | C3—K1—C7—C6 | 97.57 (17) |
| O3—K1—C3—C4 | 50.85 (12) | C8—K1—C7—C6 | 126.6 (2) |
| O1—K1—C3—C4 | 124.43 (14) | K1 ⁱ —K1—C7—C6 | 116.29 (14) |
| O4 ⁱⁱ —K1—C3—C4 | -44.86 (15) | O1 ⁱ —K1—C7—C8 | -129.2 (2) |
| O3 ⁱ —K1—C3—C4 | 147.09 (12) | O2 ⁱⁱ —K1—C7—C8 | 114.02 (14) |
| C5—K1—C3—C4 | -31.90 (12) | O3—K1—C7—C8 | -62.96 (15) |
| C6—K1—C3—C4 | -63.77 (13) | O1—K1—C7—C8 | 1.15 (14) |
| C8—K1—C3—C4 | -121.97 (19) | O4 ⁱⁱ —K1—C7—C8 | -173.96 (14) |
| K1 ⁱ —K1—C3—C4 | 98.82 (12) | O3 ⁱ —K1—C7—C8 | 59.46 (16) |
| O1 ⁱ —K1—C3—C2 | -53.34 (19) | C4—K1—C7—C8 | -62.89 (14) |
| O2 ⁱⁱ —K1—C3—C2 | 104.95 (15) | C5—K1—C7—C8 | -96.52 (16) |
| O3—K1—C3—C2 | -67.73 (15) | C3—K1—C7—C8 | -29.00 (13) |
| O1—K1—C3—C2 | 5.85 (14) | C6—K1—C7—C8 | -126.6 (2) |
| O4 ⁱⁱ —K1—C3—C2 | -163.45 (14) | K1 ⁱ —K1—C7—C8 | -10.29 (18) |
| O3 ⁱ —K1—C3—C2 | 28.50 (17) | C6—C7—C8—C3 | -0.4 (3) |
| C4—K1—C3—C2 | -118.6 (2) | K1—C7—C8—C3 | 62.90 (19) |
| C5—K1—C3—C2 | -150.48 (19) | C6—C7—C8—K1 | -63.3 (2) |
| C6—K1—C3—C2 | 177.65 (19) | C4—C3—C8—C7 | 0.9 (3) |
| C8—K1—C3—C2 | 119.4 (2) | C2—C3—C8—C7 | -179.4 (2) |
| K1 ⁱ —K1—C3—C2 | -19.76 (16) | K1—C3—C8—C7 | -68.4 (2) |
| C8—C3—C4—C5 | -0.8 (3) | C4—C3—C8—K1 | 69.28 (17) |
| C2—C3—C4—C5 | 179.52 (19) | C2—C3—C8—K1 | -111.00 (18) |
| K1—C3—C4—C5 | 74.57 (17) | O1 ⁱ —K1—C8—C7 | 142.7 (2) |
| C8—C3—C4—C9 | 178.6 (2) | O2 ⁱⁱ —K1—C8—C7 | -66.03 (14) |
| C2—C3—C4—C9 | -1.1 (3) | O3—K1—C8—C7 | 120.10 (14) |
| K1—C3—C4—C9 | -106.02 (19) | O1—K1—C8—C7 | -178.74 (15) |
| C8—C3—C4—K1 | -75.34 (18) | O4 ⁱⁱ —K1—C8—C7 | 7.10 (16) |
| C2—C3—C4—K1 | 104.94 (18) | O3 ⁱ —K1—C8—C7 | -132.18 (14) |
| O1 ⁱ —K1—C4—C5 | 105.32 (14) | C4—K1—C8—C7 | 94.82 (16) |
| O2 ⁱⁱ —K1—C4—C5 | -63.79 (16) | C5—K1—C8—C7 | 60.55 (15) |
| O3—K1—C4—C5 | 120.34 (15) | C3—K1—C8—C7 | 126.9 (2) |
| O1—K1—C4—C5 | -165.98 (15) | C6—K1—C8—C7 | 28.40 (14) |
| O4 ⁱⁱ —K1—C4—C5 | 24.49 (15) | K1 ⁱ —K1—C8—C7 | 172.23 (13) |
| O3 ⁱ —K1—C4—C5 | -165.60 (13) | O1 ⁱ —K1—C8—C3 | 15.9 (3) |
| C3—K1—C4—C5 | -120.7 (2) | O2 ⁱⁱ —K1—C8—C3 | 167.10 (13) |
| C6—K1—C4—C5 | -29.02 (13) | O3—K1—C8—C3 | -6.77 (14) |
| C8—K1—C4—C5 | -90.37 (15) | O1—K1—C8—C3 | 54.39 (13) |
| K1 ⁱ —K1—C4—C5 | 154.26 (13) | O4 ⁱⁱ —K1—C8—C3 | -119.77 (13) |
| O1 ⁱ —K1—C4—C3 | -133.94 (12) | O3 ⁱ —K1—C8—C3 | 100.94 (13) |

| | | | |
|----------------------------|--------------|------------------------------|--------------|
| O2 ⁱⁱ —K1—C4—C3 | 56.94 (14) | C4—K1—C8—C3 | -32.06 (12) |
| O3—K1—C4—C3 | -118.93 (14) | C5—K1—C8—C3 | -66.32 (14) |
| O1—K1—C4—C3 | -45.25 (12) | C6—K1—C8—C3 | -98.48 (16) |
| O4 ⁱⁱ —K1—C4—C3 | 145.22 (12) | K1 ⁱ —K1—C8—C3 | 45.36 (14) |
| O3 ⁱ —K1—C4—C3 | -44.87 (15) | C5—C4—C9—C10 | -108.5 (2) |
| C5—K1—C4—C3 | 120.7 (2) | C3—C4—C9—C10 | 72.1 (3) |
| C6—K1—C4—C3 | 91.71 (14) | K1—C4—C9—C10 | -18.6 (2) |
| C8—K1—C4—C3 | 30.37 (11) | K1—O3—C10—O4 | 134.88 (18) |
| K1 ⁱ —K1—C4—C3 | -85.01 (12) | K1 ⁱ —O3—C10—O4 | 42.3 (3) |
| O1 ⁱ —K1—C4—C9 | -13.62 (18) | K1—O3—C10—C9 | -46.6 (3) |
| O2 ⁱⁱ —K1—C4—C9 | 177.27 (14) | K1 ⁱ —O3—C10—C9 | -139.12 (17) |
| O3—K1—C4—C9 | 1.40 (14) | K1 ⁱⁱⁱ —O4—C10—O3 | -116.9 (2) |
| O1—K1—C4—C9 | 75.08 (15) | K1 ⁱⁱⁱ —O4—C10—C9 | 64.4 (3) |
| O4 ⁱⁱ —K1—C4—C9 | -94.45 (16) | C4—C9—C10—O3 | 42.3 (3) |
| O3 ⁱ —K1—C4—C9 | 75.46 (17) | C4—C9—C10—O4 | -139.0 (2) |
| C5—K1—C4—C9 | -118.9 (2) | | |

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------|----------|-------------|-------------|---------------|
| O4—H4' \cdots O1 ^{iv} | 1.01 (3) | 2.57 (3) | 3.248 (2) | 125 (2) |
| O4—H4' \cdots O2 ^{iv} | 1.01 (3) | 1.47 (3) | 2.471 (2) | 176 (3) |
| C2—H2A \cdots O2 ^v | 0.97 | 2.53 | 3.480 (3) | 167 |

Symmetry codes: (iv) $-x+1, -y+2, -z+1$; (v) $-x, -y+2, -z+1$.

Fig. 1

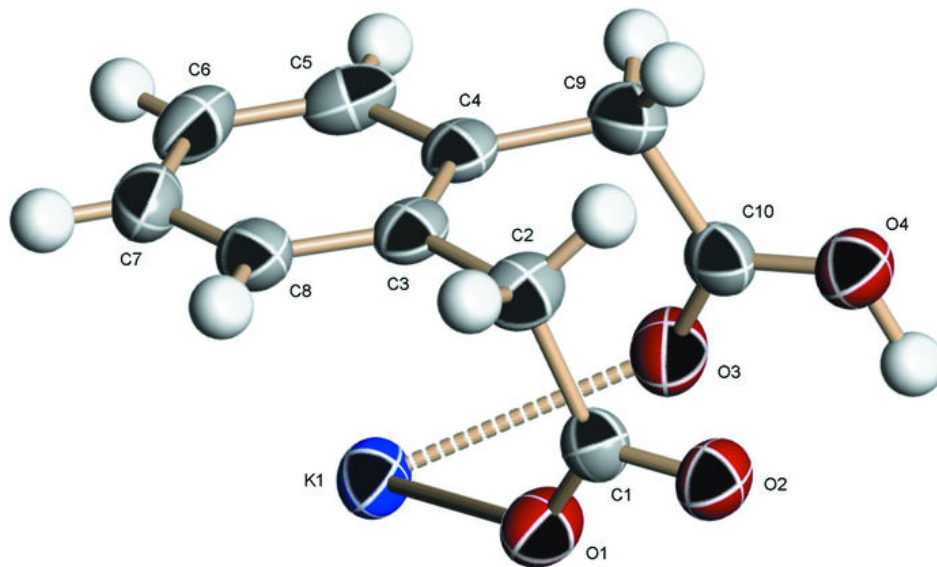


Fig. 2

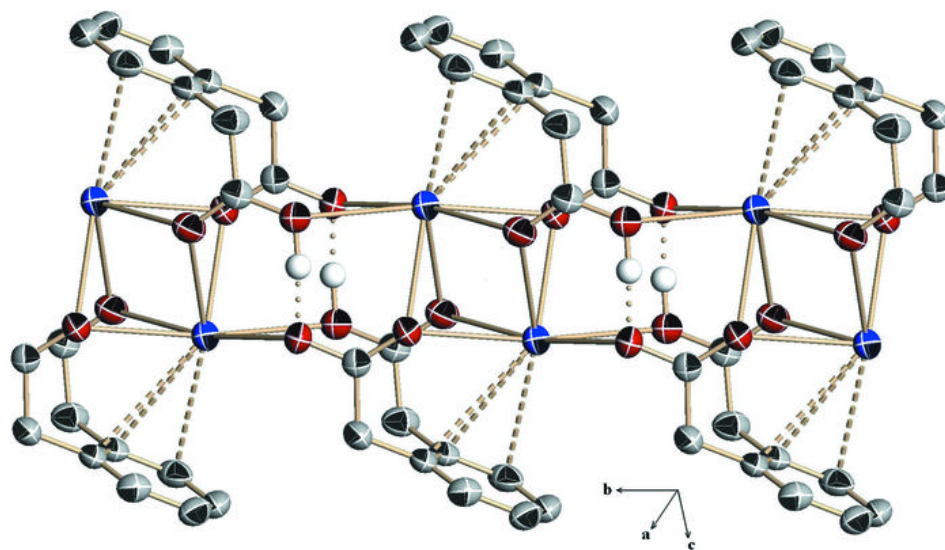


Fig. 3

