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# Potassium *cis*-[(*R*)-aspartato(2−)][(*S*)-aspartato(2−)]-cobaltate(III) 3.5-hydrate at 120 K

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**Key indicators**

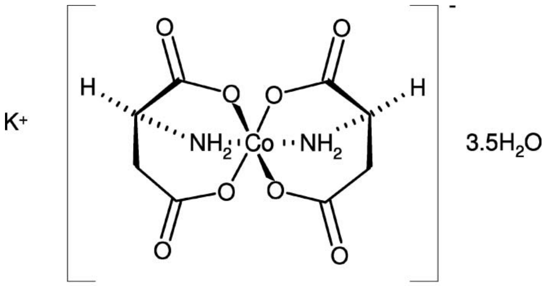
Single-crystal X-ray study
*T* = 120 K
Mean $\sigma$(C–C) = 0.008 Å
Some non-H atoms missing
*R* factor = 0.065
*wR* factor = 0.175
Data-to-parameter ratio = 16.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, K[Co($C_4H_4NO_4$)$_2$]3.5$H_2O$, is a by-product resulting from adventitious oxidation, in the presence of racemic aspartic acid, of cobalt(II) in a cobaltous starting material. The presence of both enantiomeric forms of the tridentate aspartate ligand in the cobaltate anion is significant in eliminating the possibility of the existence of isomeric forms of the *cis*(N) isomer.

## Comment

As part of our continuing study of transition metal complexes with amino acids (Felcman & de Miranda, 1997; de Miranda & Felcman, 2001; de Miranda *et al.*, 2002; Felcman *et al.*, 2003), we have isolated and characterized the title compound, (I), from an aqueous reaction mixture containing DL-aspartic acid (asp), guanidinoacetic acid (gaa) and Co$^{II}$ (1:1:1). Crystals of (I) were obtained after several months. No crystalline complex containing gaa, either alone or in a mixed complex with asp, appeared in a similar time.



(I)

The asymmetric unit in the structure of (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. In the anion, an enantiomeric pair of asp dianions, with identical numbering of the atoms and distinguished by the suffixes *A* and *B*, act as tridentate ligands, creating octahedral coordination of atom Co1. The enantiomeric relationship of the asp dianions in the complex anion is evident in the torsion angles given in Table 1 and significant in later discussion of the isomerism of such complexes.

The K$^+$ cation caps one face of the coordination octahedron of the anion to give a Co1···K1 distance of 3.6502 (14) Å. Its sevenfold coordination (Fig. 2) is completed by two non-coordinating O atoms associated with two further cobaltate anions and by two water molecules. A complex arrangement of K—O bonds connects the ions in layers parallel to (100), as shown schematically in Fig. 3. The connectivity creates, as the sub-unit, rings of four anions with four bridging K$^+$ ions, two of which are seen in the case of the eight octahedra nearest the

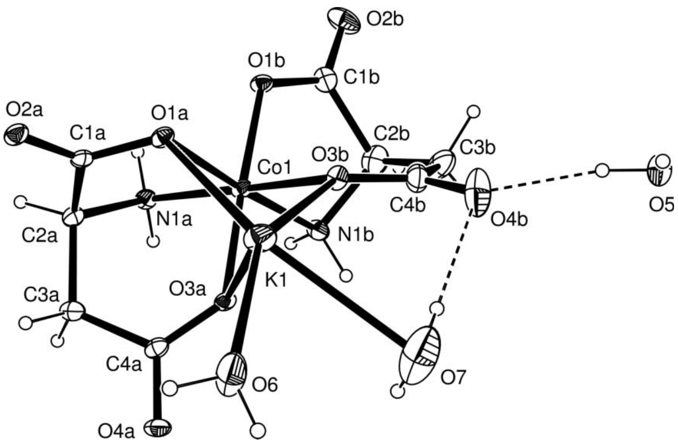


**Figure 1**
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 20% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

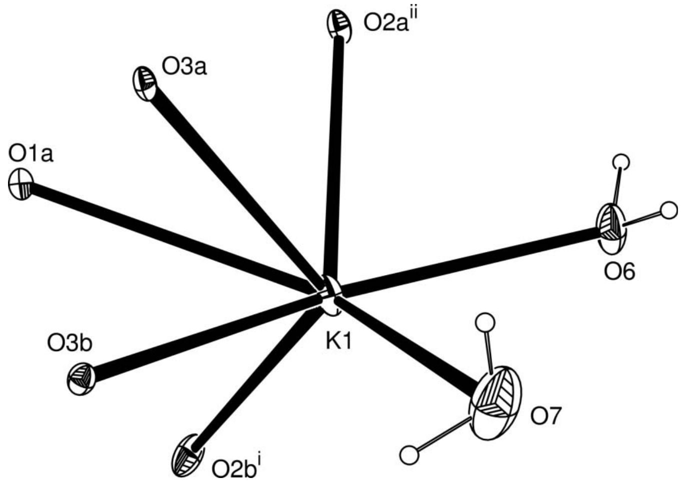


**Figure 2**
The coordination of the $K^+$ cation in (I). Displacement ellipsoids are drawn at the 10% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry codes: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $-x + 2, -y + 1, -z + 1$.]

horizontal mid-line of Fig. 3. When the layer is seen edge-on, as in Fig. 4, it is clear that the distribution of the anions creates grooves running in the direction of *c* in which the $K^+$ cations lie.

Also shown in Fig. 4 are the water molecules which, for the choice of origin used in the refinement of the structure, occur in layers centred on $x = \frac{1}{2}$ and alternate with layers of anions centred on $x = 0$, the whole arrangement being replicated by cell translation in the direction of *a*. As shown in Table 2, a large number of N—H···O and O—H···O hydrogen bonds are present in the structure of (I). Only those hydrogen bonds involving the O5 water molecule, which is the only water molecule not contributing to the immediate coordination of the $K^+$ ion, provide connectivity between adjacent layers of ions. The O5—H5*A*···O4*B* hydrogen bond is directed to one of the neighbouring layers and the other three, of the form N1*B*—H11*B*···O5$^{iv}$, O6—H6*A*···O5$^{v}$ and O5—H5*B*···O2*B*$^{v}$ [symmetry codes: (iv) $-x + 1, -y + 1, -z$; (v) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$], to the other. Also given in Table 2 are details of two weak C—H···O hydrogen bonds.

The asp ligands can be considered to have three distinct atom types available for bonding to the central metal atom because, on the basis of the labelling scheme used in this report, atom O1 is part of the carboxylate group directly

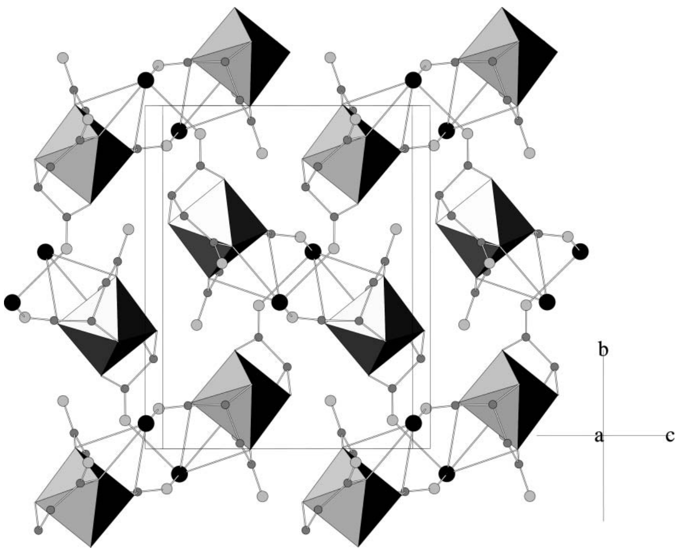


**Figure 3**
A schematic view of a layer of ions in (I). The cobaltate anions are represented by coordination octahedra. Circles of arbitrary radii represent other atoms, large and black for K and lighter and decreasing in size for O and C in that order. H atoms and water molecules have been omitted for clarity.

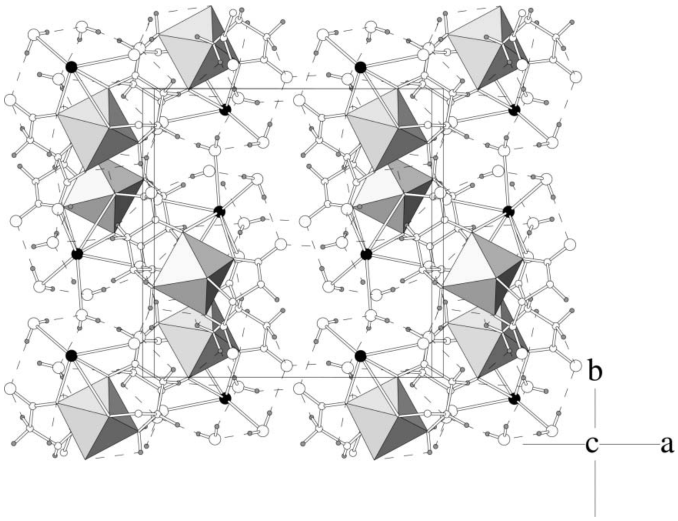


**Figure 4**
A schematic representation of the unit cell contents of (I), viewed along [001]. The cobaltate anions are represented by coordination octahedra. Circles of arbitrary radii represent other atoms, large and black for K and lighter and decreasing in size for O, C and H in that order. Dashed lines represent hydrogen bonds.

attached to the asymmetric centre (C2) of the ligand and is distinguishable, therefore, from atom O3, which is part of a carboxylate group which is $\beta$ to the asymmetric centre. When the octahedral complex is formed with one asp in each of its two enantiomeric forms, as is the case in (I), only one *cis*(N) isomer is possible in which *cis*(O1) and *cis*(O3) also occur. If, however, both asp ligands in the complex have the same enantiomeric form, say L, as in the cobaltate(III) compounds described by Oonish *et al.* (1973, 1975), the *cis*(N) arrangement is found in two isomeric forms, one with *trans*(O1) and the other with *trans*(O3), as is clearly demonstrated by Oonish *et al.* (1973).

The presence of $Co^{III}$ in (I), determined by the application of charge-balance considerations to the structural model, is at variance with the nature of the $Co^{II}$ salt starting material. However, the bond lengths and angles within the cobaltate anion in (I) are in good agreement with those found in other structures containing this type of anion such as, for example, those described by Oonish *et al.* (1973, 1975) and several other related structures. In contrast, recourse to the Cambridge Structural Database (Version 5.26; Allen, 2002) by means of the Chemical Database Service of the EPSRC (Fletcher *et al.*, 1996) has revealed only one example of a cobaltous aspartate species, namely cobaltous aspartate trihydrate, (II) (Doyne *et al.*, 1957), which is polymeric, has a Co:asp ratio of 1:1 [as distinct from 1:2 in (I)] and displays different (slightly longer) Co—N and C—O bond distances from those observed in the cobaltate anion in (I). It seems reasonable to suggest that, had not oxidation of the $Co^{II}$ of the starting material to $Co^{III}$ taken place resulting in the formation of (I), then (II) might well have been the product of the reaction.

## Experimental

To a hot solution (333 K) of guanidinoacetic acid (0.3513 g, 3 mmol) and DL-aspartic acid (0.3993 g, 3 mmol) in deionized water (100 ml) was slowly added a solution of cobalt(II) nitrate hexahydrate (0.8732 g, 3 mmol) in deionized water (5 ml). The reaction mixture was stirred at 333 K for 8 h, slowly cooled to 277 K, and the pH adjusted to 6.0 with KOH (3 *M*). The initial white precipitate which formed was filtered off and the filtrate was stored in a covered, but not sealed, vessel. Dark-blue crystals began to form after the fifth month and were collected after six months, washed with absolute ethanol and dried at 323 K. Although electron paramagnetic resonance spectroscopy indicated the presence of at least some $Co^{II}$ in the bulk product, it is clear that the sample crystal, containing $Co^{III}$, is a by-product of this reaction, arising from $Co^{III}$ either present as an impurity or created by oxidation of the initial $Co^{II}$ by oxygen in the air.

### *Crystal data*

$K[Co(C_4H_5NO_4)_2]\cdot 3.5H_2O$
$M_r = 423.27$
Monoclinic, $P2_1/c$
$a = 12.4853$ (13) Å
$b = 12.4689$ (13) Å
$c = 9.6914$ (8) Å
$\beta = 92.952$ (7)°
$V = 1506.7$ (3) Å$^3$
$Z = 4$
$D_x = 1.866$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 3129 reflections
$\theta = 2.9$–27.5°
$\mu = 1.48$ mm$^{-1}$
$T = 120$ (2) K
Block, dark blue
$0.40 \times 0.30 \times 0.08$ mm

### *Data collection*

Nonius KappaCCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.517$, $T_{max} = 0.891$
16788 measured reflections
3330 independent reflections
2568 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 27.5°$
$h = -16 \rightarrow 15$
$k = -16 \rightarrow 16$
$l = -12 \rightarrow 12$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.175$
$S = 1.09$
3330 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0839P)^2 + 4.5102P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.24$ e Å$^{-3}$
$\Delta\rho_{min} = -0.64$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Co1—O1*B* | 1.886 (3) | K1—O6 | 2.766 (4) |
| Co1—O3*A* | 1.899 (3) | K1—O7 | 2.799 (6) |
| Co1—N1*A* | 1.902 (4) | K1—O2*A*$^{i}$ | 2.821 (4) |
| Co1—N1*B* | 1.909 (4) | K1—O2*B*$^{ii}$ | 2.862 (4) |
| Co1—O3*B* | 1.917 (3) | K1—O3*A* | 3.078 (4) |
| Co1—O1*A* | 1.921 (3) | K1—O1*A* | 3.092 (3) |
| K1—O3*B* | 2.759 (3) | | |
| O1*B*—Co1—O3*A* | 177.34 (14) | N1*A*—Co1—O3*B* | 174.29 (15) |
| O1*B*—Co1—N1*A* | 89.62 (16) | N1*B*—Co1—O3*B* | 88.27 (16) |
| O3*A*—Co1—N1*A* | 91.48 (16) | O1*B*—Co1—O1*A* | 90.54 (13) |
| O1*B*—Co1—N1*B* | 86.38 (15) | O3*A*—Co1—O1*A* | 91.97 (13) |
| O3*A*—Co1—N1*B* | 91.08 (15) | N1*A*—Co1—O1*A* | 84.57 (15) |
| N1*A*—Co1—N1*B* | 97.02 (18) | N1*B*—Co1—O1*A* | 176.52 (15) |
| O1*B*—Co1—O3*B* | 92.85 (15) | O3*B*—Co1—O1*A* | 90.26 (14) |
| O3*A*—Co1—O3*B* | 86.27 (14) | | |
| N1*A*—C2*A*—C3*A*—C4*A* | 50.6 (6) | N1*B*—C2*B*—C3*B*—C4*B* | −45.0 (7) |
| C1*A*—C2*A*—C3*A*—C4*A* | −67.7 (5) | C1*B*—C2*B*—C3*B*—C4*B* | 72.8 (7) |

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

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1*A*—H11*A*···O2*A*$^{iii}$ | 0.92 | 2.29 | 2.930 (5) | 126 |
| N1*A*—H11*A*···O4*A*$^{iv}$ | 0.92 | 2.32 | 3.011 (5) | 131 |
| N1*A*—H12*A*···O4*A*$^{v}$ | 0.92 | 2.06 | 2.981 (5) | 177 |
| N1*B*—H11*B*···O5$^{vi}$ | 0.92 | 2.04 | 2.942 (6) | 168 |
| N1*B*—H12*B*···O1*B*$^{iii}$ | 0.92 | 2.34 | 2.925 (5) | 121 |
| N1*B*—H12*B*···O1*A*$^{iii}$ | 0.92 | 2.49 | 3.166 (5) | 130 |
| O5—H5*A*···O4*B* | 0.84 | 1.87 | 2.685 (6) | 163 |
| O5—H5*B*···O2*B*$^{vii}$ | 0.84 | 1.94 | 2.777 (6) | 169 |
| O6—H6*A*···O5$^{vii}$ | 0.84 | 2.07 | 2.826 (6) | 149 |
| O6—H6*B*···O4*A*$^{viii}$ | 0.84 | 2.08 | 2.914 (6) | 174 |
| O7—H7*A*···O6$^{ix}$ | 0.84 | 2.23 | 3.074 (11) | 17 |
| O7—H7*B*···O4*B* | 0.84 | 2.20 | 3.038 (10) | 179 |
| C2*B*—H21*B*···O3*B*$^{iii}$ | 1.00 | 2.48 | 3.419 (7) | 156 |
| C3*A*—H32*A*···O5$^{x}$ | 0.99 | 2.59 | 3.328 (7) | 132 |

Symmetry codes: (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $-x+2, -y+1, -z$; (vi) $-x+1, -y+1, -z$; (vii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (viii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ix) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (x) $x+1, y, z$.

As indicated by *PLATON* (Spek, 2003), the structural model used here sustains two symmetry-related [at $(\frac{1}{2}, 0, 0)$ and $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$] solvent-accessible regions, each of volume 19 Å$^3$, per unit cell. Excluded from each of these regions of the structural model were two low electron density (approximately 2 e Å$^{-3}$) features. This was accompanied by the supression, by means of the SQUEEZE option of *PLATON*, of their contribution to the intensity data. These features, less than 3 Å from the $K^+$ ion, less than 1 Å apart and with site occupancy factors estimated to be of the order of 0.25, are perceived as representing additional highly disordered water molecules solvating the $K^+$ ion and present in total to the extent of $0.5H_2O$ per formula unit. The additional half-molecule of water has been included in the molecular formula but is, of course, absent from the structural model. In the final stages of refinement, H atoms attached to C and N atoms were placed in calculated positions, with C—H distances for tertiary and secondary C atoms of 1.00 and 0.99 Å, respectively, and for N, treated as secondary C, with N—H distance 0.92 Å. These H atoms were then refined with a riding model, with $U_{iso}(H) = 1.2U_{eq}(C,N)$. Approximate positions for the H atoms of the water molecules were obtained from difference maps, the geometry of the water molecules idealized to give O—H distances of 0.84 Å and H—O—H angles in the range 101–105°, and the H atoms then refined with a riding model, with $U_{iso}(H) = 1.5U_{eq}(O)$. In the final difference map, the largest peak is 1.01 Å from atom C2*B*.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *ATOMS for Windows* (Dowty, 1998); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

We acknowledge the use of the Chemical Database Service at Daresbury and the X-ray Crystallographic Service in Southampton, England, both services being provided by the EPSRC.

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# supporting information

*Acta Cryst.* (2006). E**62**, m52–m55    [doi:10.1107/S160053680503998X]

# Potassium *cis*-[(*R*)-aspartato(2–)][(*S*)-aspartato(2–)]cobaltate(III) 3.5-hydrate at 120 K

**Otavio Versiane, Judith Felcman, Jussara Lopes de Miranda, R. Alan Howie, Janet M. S. Skakle and James L. Wardell**

## S1. Comment

As part of our continuing study of transition metal complexes with amino acids (Felcman & de Miranda, 1997; de Miranda & Felcman, 2001; de Miranda *et al.*, 2002; Felcman *et al.*, 2003), we have isolated and characterized the title compound, (I), from an aqueous reaction mixture containing *DL*-aspartic acid (asp), guanidineacetic acid (gaa) and Co$^{II}$ (1:1:1). Crystals of (I) were obtained after several months. No crystalline complex containing gaa, either alone or in a mixed complex with asp, appeared in a similar time.

The asymmetric unit in the structure of (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. In the anion, an enantiomeric pair of asp dianions, with identical labelling of the atoms and distinguished by the suffixes *A* and *B*, act as tridentate ligands, creating octahedral coordination of atom Co1. The enantiomeric relationship of the asp dianions in the complex anion is evident in the torsion angles given in Table 1 and significant in later discussion of the isomerism of such complexes.

The K atom caps one face of the coordination octahedron of the anion to give a Co1···K1 distance of 3.6502 (14) Å. Its sevenfold coordination (Fig. 2) is completed by two non-coordinating O atoms associated with two further cobaltate anions and by two water molecules. A complex arrangement of K—O bonds connects the ions in layers parallel to (100), as shown schematically in Fig. 3. The connectivity creates, as the sub-unit, rings of four anions with four bridging K atoms, two of which are seen in the case of the eight octahedra nearest the horizontal mid-line of Fig. 3. When the layer is seen edge-on, as in Fig. 4, it is clear that the distribution of the anions creates grooves running in the direction of c in which the K cations lie.

Also shown in Fig. 4 are the water molecules which, for the choice of origin used in the refinement of the structure, occur in layers centred on $x = 1/2$ and alternate with layers of anions centred on $x = 0$, the whole arrangement being replicated by cell translation in the direction of a. As shown in Table 2, a large number of N—H···O and O—H···O hydrogen bonds are present in the structure of (I). Only those hydrogen bonds involving the O5 water molecule, which is the only water molecule not contributing to the immediate coordination of the K atom, provide connectivity between adjacent layers of ions. The O5—H5*A*···O4*B* hydrogen bond is directed to one of the neighbouring layers and the other three, of the form N1*B*—H11*B*···O5$^{iv}$, O6—H6*A*···O5$^{v}$ and O5—H5*B*···O2*B*$^{v}$ [symmetry codes: (iv) $-x + 1, -y + 1, -z$; (v) $-x + 1, y + 1/2, -z + 1/2$], to the other. Also given in Table 2 are details of two weak C—H···O hydrogen bonds.

The asp ligands can be considered to have three distinct atom types available for bonding to the central metal atom because, on the basis of the labelling scheme used in this report, atom O1 is part of the carboxyl group directly attached to the asymmetric centre (C2) of the ligand and is distinguishable, therefore, from atom O3, which is part of a carboxyl group which is *β* to the asymmetric centre. When the octahedral complex is formed with one asp in each of its two enantiomeric forms, as is the case in (I), only one *cis*(N) isomer is possible in which *cis*(O1) and *cis*(O3) also occur. If, however, both asp ligands in the complex have the same enantiomeric form, say *L*, as in the cobaltate(III) compounds described by Oonish *et al.* (1973, 1975), the *cis*(N) arrangement is found in two isomeric forms, one with *trans*(O1) and the other with *trans*(O3), as is clearly demonstrated by Oonish *et al.* (1973).

The presence of $Co^{III}$ in (I), determined by the application of charge-balance considerations to the structural model, is at variance with the nature of the $Co^{II}$ salt starting material. However, the bond lengths and angles within the cobaltate anion in (I) are in good agreement with those found in other structures containing this type of anion such as, for example, those described by Oonish *et al.* (1973, 1975) and several other related structures. In contrast, recourse to the Cambridge Structural Database (**Version?**; Allen, 2002) by means the Chemical Database Service of the EPSRC (Fletcher *et al.*, 1996) has revealed only one example of a cobaltous aspartate species, namely cobaltous aspartate trihydrate, (II) (Doyne *et al.*, 1957), which is polymeric, has a Co:asp ratio of 1:1 [as distinct from 1:2 in (I)] and displays different (slightly longer) Co—N and C—O bond distances from those observed in the cobaltate anion in (I). It seems reasonable to suggest that, had not oxidation of the $Co^{II}$ of the starting material to $Co^{III}$ taken place resulting in the formation of (I), then (II) might well have been the product of the reaction.

## S2. Experimental

To a hot solution (333 K) of guanidinoacetic acid (0.3513 g, 3 mmol) and *DL*-aspartic acid (0.3993 g, 3 mmol) in deionized water (**Volume?**) was slowly added a solution of cobalt(II) nitrate hexahydrate (0.8732 g, 3 mmol) in deionized water (5 ml). The reaction mixture was stirred at 333 K for 8 h, slowly cooled to 277 K, and the pH adjusted to 6.0 with KOH (3 *M*). The initial white precipitate which formed was filtered off and the filtrate was stored in a covered, but not sealed, vessel. Dark-blue crystals began to form after the fifth month and were collected after six months, washed with absolute alcohol [**ethanol?**] and dried at 323 K. Although electron paramagnetic resonance spectroscopy indicated the presence of at least some $Co^{II}$ in the bulk product, it is clear that the sample crystal, containing $Co^{III}$, is a by-product of this reaction, arising from $Co^{III}$ either present as an impurity or created by oxidation of the initial $Co^{II}$ by oxygen of the air.

## S3. Refinement

As indicated by *PLATON* (Spek, 2003), the structural model used here sustains two symmetry-related [at (1/2,0,0) and (1/2,1/2,1/2)] solvent-accessible regions, each of volume 19 Å$^3$, per unit cell. Excluded from each of these regions of the structural model were two low electron density (approximately 2 e Å$^{-3}$) features. This was accompanied by the supression, by means of the SQUEEZE option of *PLATON*, of their contribution to the intensity data. These features, less than 3 Å from the K atom, less than 1 Å apart and with site occupancy factors estimated to be of the order of 1/4, are perceived as representing additional highly disordered water molecules solvating the K atom and present in total to the extent of 0.5 $H_2O$ per formula unit. The additional half-molecule of water has been included in the molecular formula but is, of course, absent from the structural model. In the final stages of refinement, H atoms attached to C and N atoms were placed in calculated positions, with C—H distances for tertiary and secondary C atoms of 1.00 and 0.99 Å, respectively, and for N, treated as secondary C, with N—H distance 0.92 Å. These H atoms were then refined with a riding model, with $U_{iso}(H) = 1.2U_{eq}(C,N)$. Approximate positions for the H atoms of the water molecules were obtained from difference maps, the geometry of the water molecules idealized to give O—H distances of 0.84 Å and H—O—H angles in the range 100.9–104.8°, and the H atoms then refined with a riding model, with $U_{iso}(H) = 1.5U_{eq}(O)$. In the final difference map, the largest peak is 1.01 Å from atom C2*B*.

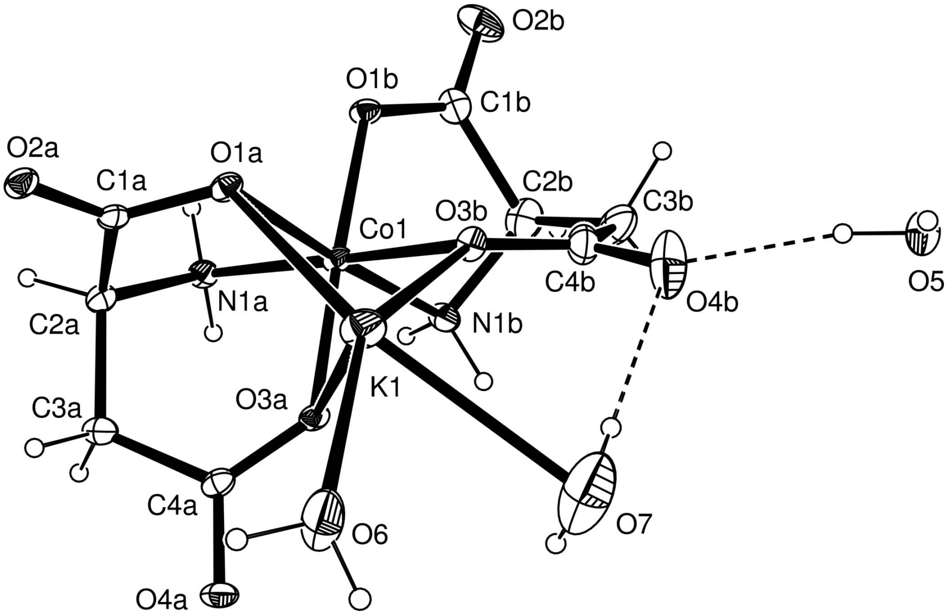

**Figure 1**

The asymmetric unit in (I). Displacement ellipsoids are drawn at the 20% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

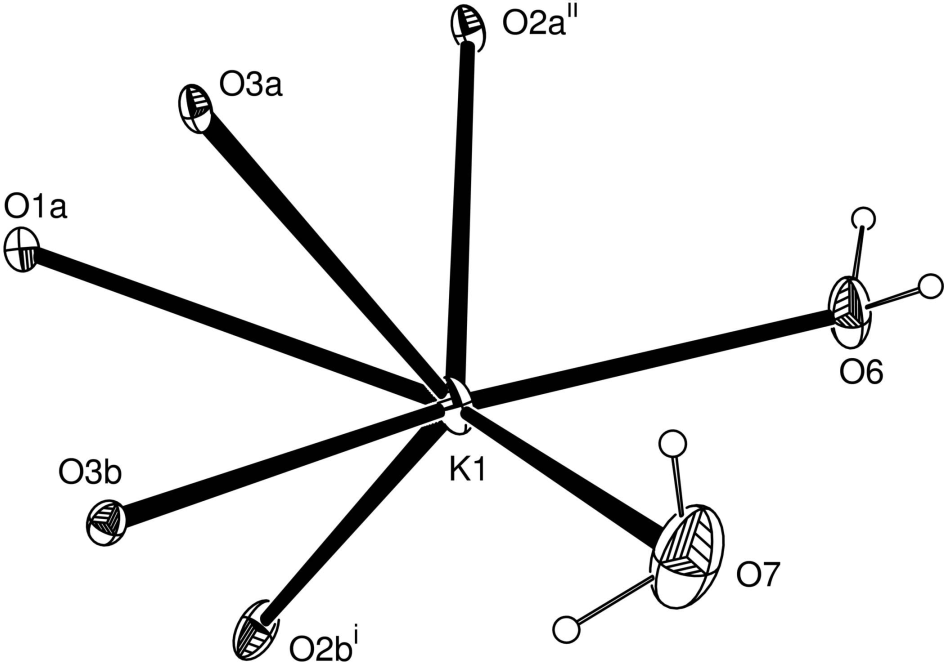

**Figure 2**

The coordination of the K atom in (I). Displacement ellipsoids are drawn at the 10% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry codes: (i) *x*, −*y* + 1/2, *z* + 1/2; (ii) −*x* + 2, −*y* + 1, −*z* + 1.]

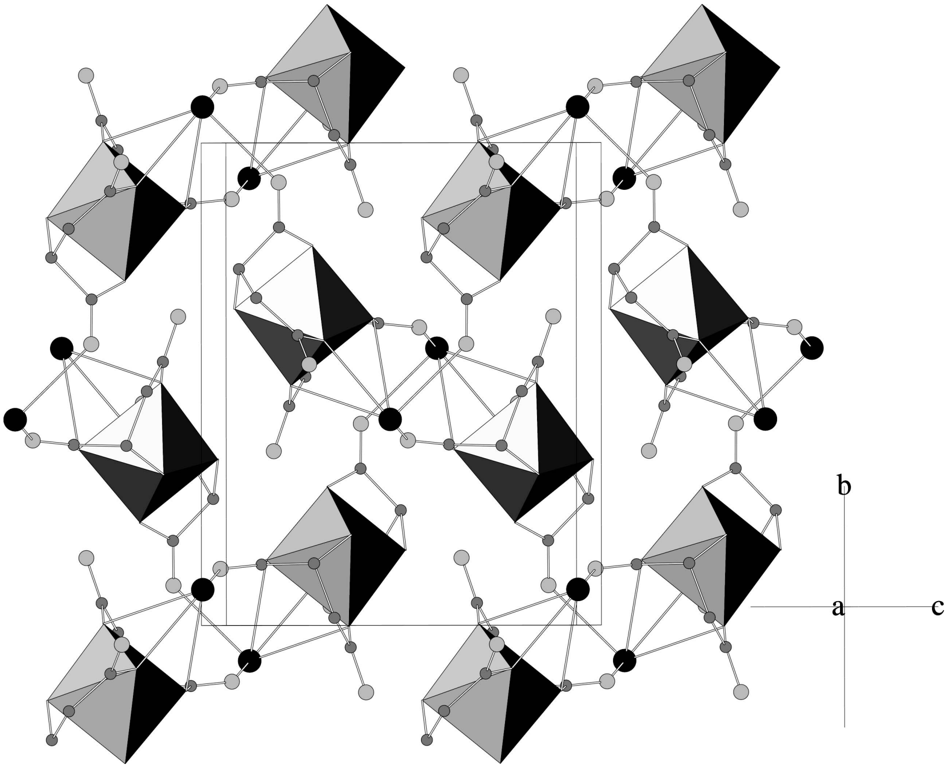

**Figure 3**

A schematic view of a layer of ions in (I). The cobaltate anions are represented by coordination octahedra. Circles of arbitrary radii represent other atoms, large and black for K and lighter and decreasing in size for O and C in that order. H atoms and water molecules have been omitted for clarity.

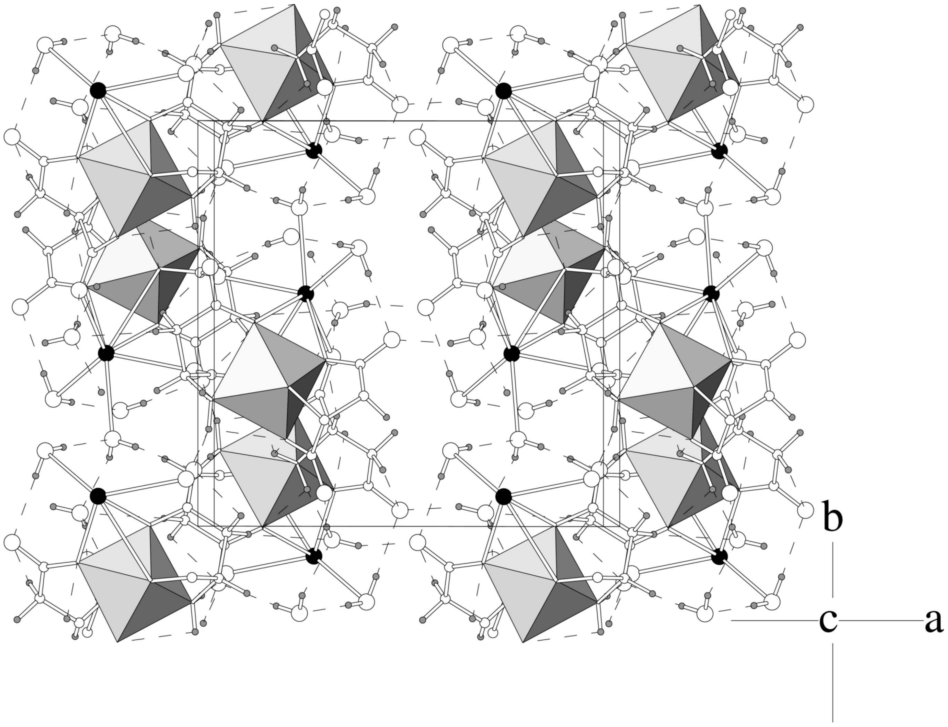

**Figure 4**

A schematic representation of the unit cell of (I), viewed along [001]. The cobaltate anions are represented by coordination octahedra. Circles of arbitrary radii represent other atoms, large and black for K and lighter and decreasing in size for O, C and H in that order. Dashed lines represent hydrogen bonds.

### Potassium *cis*-[(*R*)-aspartato(2-)][(S)-aspartato(2-)]cobaltate(III) 3.5-hydrate

*Crystal data*

$K[Co(C_4H_5NO_4)_2]\cdot 3.5H_2O$
$M_r = 423.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 12.4853$ (13) Å
$b = 12.4689$ (13) Å
$c = 9.6914$ (8) Å
$\beta = 92.952$ (7)°
$V = 1506.7$ (3) Å$^3$
$Z = 4$

$F(000) = 868$
$D_x = 1.866$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3129 reflections
$\theta = 2.9–27.5°$
$\mu = 1.48$ mm$^{-1}$
$T = 120$ K
Block, dark blue
0.40 × 0.30 × 0.08 mm

*Data collection*

Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)

$T_{\min} = 0.517$, $T_{\max} = 0.891$
16788 measured reflections
3330 independent reflections
2568 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.045$
$\theta_{\max} = 27.5°$, $\theta_{\min} = 3.2°$
$h = -16\rightarrow15$
$k = -16\rightarrow16$
$l = -12\rightarrow12$

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: geom and difmap |
| $R[F^2 > 2\sigma(F^2)] = 0.065$ | H-atom parameters constrained |
| $wR(F^2) = 0.175$ | $w = 1/[\sigma^2(F_o^2) + (0.0839P)^2 + 4.5102P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.09$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 3330 reflections | $\Delta\rho_{max} = 1.24$ e Å$^{-3}$ |
| 208 parameters | $\Delta\rho_{min} = -0.64$ e Å$^{-3}$ |
| 0 restraints | |
| Primary atom site location: structure-invariant direct methods | |

*Special details*

**Experimental**. Unit cell determined with *DIRAX* (Duisenberg, 1992; Duisenberg *et al.* 2000) but refined with the *DENZO*/*COLLECT HKL* package.
Refs as: Duisenberg, A. J. *M.* (1992). J. Appl. Cryst. 25, 92–96. Duisenberg, A. J. *M.*, Hooft, *R.* W. W., Schreurs, A. *M. M.* & Kroon, J. (2000). J. Appl. Cryst. 33, 893–898.

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Co1 | 0.83110 (5) | 0.35774 (5) | 0.19420 (6) | 0.0229 (2) |
| K1 | 0.75090 (11) | 0.57375 (9) | 0.42121 (13) | 0.0439 (3) |
| O1A | 0.8801 (3) | 0.3649 (2) | 0.3852 (3) | 0.0261 (7) |
| O2A | 1.0300 (3) | 0.3831 (3) | 0.5172 (3) | 0.0317 (8) |
| C1A | 0.9823 (4) | 0.3742 (3) | 0.4046 (5) | 0.0267 (10) |
| C2A | 1.0420 (4) | 0.3720 (4) | 0.2685 (5) | 0.0295 (10) |
| H21A | 1.1123 | 0.3343 | 0.2841 | 0.035* |
| N1A | 0.9735 (3) | 0.3124 (3) | 0.1647 (4) | 0.0264 (8) |
| H11A | 0.9804 | 0.2396 | 0.1777 | 0.032* |
| H12A | 0.9920 | 0.3289 | 0.0765 | 0.032* |
| C3A | 1.0608 (4) | 0.4847 (4) | 0.2154 (5) | 0.0326 (11) |
| H31A | 1.0981 | 0.5265 | 0.2902 | 0.039* |
| H32A | 1.1096 | 0.4801 | 0.1382 | 0.039* |
| C4A | 0.9615 (4) | 0.5457 (4) | 0.1658 (5) | 0.0274 (10) |
| O3A | 0.8679 (3) | 0.5034 (2) | 0.1644 (3) | 0.0264 (7) |
| O4A | 0.9747 (3) | 0.6394 (2) | 0.1257 (3) | 0.0330 (8) |
| O1B | 0.7903 (3) | 0.2133 (2) | 0.2163 (3) | 0.0270 (7) |
| O2B | 0.6924 (4) | 0.0825 (3) | 0.1201 (4) | 0.0545 (12) |
| C1B | 0.7245 (4) | 0.1754 (4) | 0.1234 (5) | 0.0354 (12) |
| C2B | 0.6882 (5) | 0.2621 (5) | 0.0140 (6) | 0.0491 (15) |
| H21B | 0.6743 | 0.2280 | −0.0785 | 0.059* |

| | | | | |
|---|---|---|---|---|
| N1B | 0.7764 (3) | 0.3441 (3) | 0.0074 (4) | 0.0304 (9) |
| H11B | 0.7499 | 0.4085 | −0.0257 | 0.036* |
| H12B | 0.8290 | 0.3208 | −0.0486 | 0.036* |
| C3B | 0.5928 (6) | 0.3219 (5) | 0.0535 (6) | 0.0525 (16) |
| H31B | 0.5632 | 0.3603 | −0.0293 | 0.063* |
| H32B | 0.5381 | 0.2692 | 0.0797 | 0.063* |
| C4B | 0.6066 (5) | 0.3995 (5) | 0.1653 (6) | 0.0420 (13) |
| O3B | 0.6932 (3) | 0.4104 (3) | 0.2403 (3) | 0.0284 (7) |
| O4B | 0.5292 (4) | 0.4599 (5) | 0.1905 (6) | 0.0818 (17) |
| O5 | 0.3163 (3) | 0.4669 (3) | 0.1357 (5) | 0.0475 (10) |
| H5A | 0.3821 | 0.4518 | 0.1469 | 0.071* |
| H5B | 0.3056 | 0.4986 | 0.2107 | 0.071* |
| O6 | 0.7698 (4) | 0.7853 (3) | 0.5047 (6) | 0.0744 (17) |
| H6A | 0.7598 | 0.8298 | 0.4404 | 0.112* |
| H6B | 0.8282 | 0.8038 | 0.5449 | 0.112* |
| O7 | 0.6130 (6) | 0.6865 (6) | 0.2363 (8) | 0.117 (3) |
| H7A | 0.6556 | 0.6947 | 0.1728 | 0.175* |
| H7B | 0.5893 | 0.6240 | 0.2237 | 0.175* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Co1 | 0.0306 (4) | 0.0164 (3) | 0.0212 (3) | 0.0002 (2) | −0.0019 (2) | −0.0009 (2) |
| K1 | 0.0616 (9) | 0.0277 (6) | 0.0415 (7) | −0.0043 (5) | −0.0062 (6) | −0.0053 (5) |
| O1A | 0.035 (2) | 0.0226 (16) | 0.0199 (16) | 0.0018 (13) | −0.0024 (13) | −0.0001 (12) |
| O2A | 0.042 (2) | 0.0193 (16) | 0.0327 (19) | −0.0008 (14) | −0.0092 (15) | 0.0028 (13) |
| C1A | 0.032 (3) | 0.015 (2) | 0.032 (3) | −0.0003 (17) | −0.002 (2) | 0.0051 (17) |
| C2A | 0.031 (3) | 0.024 (2) | 0.033 (3) | 0.0026 (19) | −0.002 (2) | 0.0043 (19) |
| N1A | 0.032 (2) | 0.0168 (18) | 0.030 (2) | 0.0001 (15) | −0.0011 (16) | −0.0032 (15) |
| C3A | 0.039 (3) | 0.027 (3) | 0.032 (3) | −0.004 (2) | 0.004 (2) | 0.0046 (19) |
| C4A | 0.038 (3) | 0.024 (2) | 0.020 (2) | 0.0022 (19) | −0.0010 (19) | 0.0003 (17) |
| O3A | 0.0326 (18) | 0.0143 (15) | 0.0314 (17) | 0.0007 (13) | −0.0054 (13) | 0.0015 (12) |
| O4A | 0.050 (2) | 0.0215 (17) | 0.0269 (17) | −0.0100 (14) | −0.0040 (15) | 0.0047 (13) |
| O1B | 0.0363 (19) | 0.0179 (15) | 0.0264 (17) | −0.0028 (13) | −0.0012 (14) | 0.0012 (12) |
| O2B | 0.067 (3) | 0.049 (3) | 0.048 (2) | −0.031 (2) | 0.010 (2) | −0.0141 (19) |
| C1B | 0.036 (3) | 0.043 (3) | 0.027 (3) | −0.001 (2) | 0.003 (2) | −0.006 (2) |
| C2B | 0.053 (4) | 0.060 (4) | 0.034 (3) | 0.004 (3) | −0.003 (3) | −0.007 (3) |
| N1B | 0.034 (2) | 0.034 (2) | 0.023 (2) | −0.0099 (17) | 0.0005 (17) | 0.0001 (16) |
| C3B | 0.065 (4) | 0.044 (3) | 0.046 (4) | −0.001 (3) | −0.024 (3) | 0.001 (3) |
| C4B | 0.032 (3) | 0.055 (3) | 0.039 (3) | 0.000 (3) | −0.004 (2) | 0.004 (3) |
| O3B | 0.0293 (18) | 0.0288 (18) | 0.0268 (17) | 0.0038 (14) | −0.0012 (14) | −0.0045 (13) |
| O4B | 0.040 (3) | 0.123 (5) | 0.081 (4) | 0.019 (3) | −0.007 (2) | −0.016 (3) |
| O5 | 0.035 (2) | 0.055 (3) | 0.051 (2) | 0.0017 (18) | −0.0079 (18) | 0.0020 (19) |
| O6 | 0.066 (3) | 0.035 (2) | 0.116 (4) | 0.007 (2) | −0.046 (3) | −0.018 (3) |
| O7 | 0.117 (6) | 0.103 (5) | 0.124 (6) | 0.045 (4) | −0.050 (5) | −0.035 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Co1—O1B | 1.886 (3) | C3A—H32A | 0.9900 |
| Co1—O3A | 1.899 (3) | C4A—O4A | 1.245 (6) |
| Co1—N1A | 1.902 (4) | C4A—O3A | 1.282 (6) |
| Co1—N1B | 1.909 (4) | O1B—C1B | 1.278 (6) |
| Co1—O3B | 1.917 (3) | O2B—C1B | 1.226 (7) |
| Co1—O1A | 1.921 (3) | O2B—K1$^{iii}$ | 2.862 (4) |
| K1—O3B | 2.759 (3) | C1B—C2B | 1.565 (8) |
| K1—O6 | 2.766 (4) | C2B—C3B | 1.472 (9) |
| K1—O7 | 2.799 (6) | C2B—N1B | 1.506 (8) |
| K1—O2A$^{i}$ | 2.821 (4) | C2B—H21B | 1.0000 |
| K1—O2B$^{ii}$ | 2.862 (4) | N1B—H11B | 0.9200 |
| K1—O3A | 3.078 (4) | N1B—H12B | 0.9200 |
| K1—O1A | 3.092 (3) | C3B—C4B | 1.456 (8) |
| O1A—C1A | 1.286 (6) | C3B—H31B | 0.9900 |
| O2A—C1A | 1.221 (6) | C3B—H32B | 0.9900 |
| O2A—K1$^{i}$ | 2.821 (4) | C4B—O4B | 1.258 (8) |
| C1A—C2A | 1.549 (7) | C4B—O3B | 1.279 (6) |
| C2A—N1A | 1.486 (6) | O5—H5A | 0.8434 |
| C2A—C3A | 1.518 (6) | O5—H5B | 0.8445 |
| C2A—H21A | 1.0000 | O6—H6A | 0.8397 |
| N1A—H11A | 0.9200 | O6—H6B | 0.8406 |
| N1A—H12A | 0.9200 | O7—H7A | 0.8400 |
| C3A—C4A | 1.512 (7) | O7—H7B | 0.8400 |
| C3A—H31A | 0.9900 | Co1—K1 | 3.6502 (14) |

| | | | |
|---|---|---|---|
| O1B—Co1—O3A | 177.34 (14) | C2A—N1A—H12A | 110.7 |
| O1B—Co1—N1A | 89.62 (16) | Co1—N1A—H12A | 110.7 |
| O3A—Co1—N1A | 91.48 (16) | H11A—N1A—H12A | 108.8 |
| O1B—Co1—N1B | 86.38 (15) | C4A—C3A—C2A | 115.8 (4) |
| O3A—Co1—N1B | 91.08 (15) | C4A—C3A—H31A | 108.3 |
| N1A—Co1—N1B | 97.02 (18) | C2A—C3A—H31A | 108.3 |
| O1B—Co1—O3B | 92.85 (15) | C4A—C3A—H32A | 108.3 |
| O3A—Co1—O3B | 86.27 (14) | C2A—C3A—H32A | 108.3 |
| N1A—Co1—O3B | 174.29 (15) | H31A—C3A—H32A | 107.4 |
| N1B—Co1—O3B | 88.27 (16) | O4A—C4A—O3A | 121.2 (4) |
| O1B—Co1—O1A | 90.54 (13) | O4A—C4A—C3A | 116.9 (4) |
| O3A—Co1—O1A | 91.97 (13) | O3A—C4A—C3A | 121.9 (4) |
| N1A—Co1—O1A | 84.57 (15) | C4A—O3A—Co1 | 128.3 (3) |
| N1B—Co1—O1A | 176.52 (15) | C4A—O3A—K1 | 110.2 (3) |
| O3B—Co1—O1A | 90.26 (14) | Co1—O3A—K1 | 91.21 (12) |
| O3B—K1—O6 | 154.90 (14) | C1B—O1B—Co1 | 116.2 (3) |
| O3B—K1—O7 | 80.32 (15) | C1B—O2B—K1$^{iii}$ | 124.5 (3) |
| O6—K1—O7 | 75.45 (17) | O2B—C1B—O1B | 124.4 (5) |
| O3B—K1—O2A$^{i}$ | 119.60 (11) | O2B—C1B—C2B | 123.5 (5) |
| O6—K1—O2A$^{i}$ | 71.88 (12) | O1B—C1B—C2B | 112.0 (5) |
| O7—K1—O2A$^{i}$ | 126.7 (2) | C3B—C2B—N1B | 105.7 (5) |

| | | | |
|---|---|---|---|
| O3B—K1—O2B$^{ii}$ | 81.86 (12) | C3B—C2B—C1B | 112.6 (5) |
| O6—K1—O2B$^{ii}$ | 118.22 (16) | N1B—C2B—C1B | 108.1 (4) |
| O7—K1—O2B$^{ii}$ | 127.1 (2) | C3B—C2B—H21B | 110.1 |
| O2A$^{i}$—K1—O2B$^{ii}$ | 105.39 (12) | N1B—C2B—H21B | 110.1 |
| O3B—K1—O3A | 52.76 (9) | C1B—C2B—H21B | 110.1 |
| O6—K1—O3A | 118.15 (15) | C2B—N1B—Co1 | 104.3 (3) |
| O7—K1—O3A | 85.84 (18) | C2B—N1B—H11B | 110.9 |
| O2A$^{i}$—K1—O3A | 74.34 (10) | Co1—N1B—H11B | 110.9 |
| O2B$^{ii}$—K1—O3A | 119.93 (11) | C2B—N1B—H12B | 110.9 |
| O3B—K1—O1A | 55.06 (9) | Co1—N1B—H12B | 110.9 |
| O6—K1—O1A | 143.04 (13) | H11B—N1B—H12B | 108.9 |
| O7—K1—O1A | 131.14 (15) | C4B—C3B—C2B | 117.6 (5) |
| O2A$^{i}$—K1—O1A | 71.27 (9) | C4B—C3B—H31B | 107.9 |
| O2B$^{ii}$—K1—O1A | 69.84 (10) | C2B—C3B—H31B | 107.9 |
| O3A—K1—O1A | 52.89 (8) | C4B—C3B—H32B | 107.9 |
| C1A—O1A—Co1 | 114.1 (3) | C2B—C3B—H32B | 107.9 |
| C1A—O1A—K1 | 115.3 (3) | H31B—C3B—H32B | 107.2 |
| Co1—O1A—K1 | 90.35 (11) | O4B—C4B—O3B | 117.5 (5) |
| C1A—O2A—K1$^{i}$ | 129.0 (3) | O4B—C4B—C3B | 118.6 (5) |
| O2A—C1A—O1A | 125.0 (5) | O3B—C4B—C3B | 123.9 (5) |
| O2A—C1A—C2A | 121.9 (4) | C4B—O3B—Co1 | 125.1 (3) |
| O1A—C1A—C2A | 113.1 (4) | C4B—O3B—K1 | 128.7 (3) |
| N1A—C2A—C3A | 109.1 (4) | Co1—O3B—K1 | 101.11 (13) |
| N1A—C2A—C1A | 107.4 (4) | H5A—O5—H5B | 100.9 |
| C3A—C2A—C1A | 111.2 (4) | K1—O6—H6A | 114.0 |
| N1A—C2A—H21A | 109.7 | K1—O6—H6B | 117.1 |
| C3A—C2A—H21A | 109.7 | H6A—O6—H6B | 104.8 |
| C1A—C2A—H21A | 109.7 | K1—O7—H7A | 98.0 |
| C2A—N1A—Co1 | 105.1 (3) | K1—O7—H7B | 80.1 |
| C2A—N1A—H11A | 110.7 | H7A—O7—H7B | 103.8 |
| Co1—N1A—H11A | 110.7 | | |

| | | | |
|---|---|---|---|
| O2A—C1A—C2A—N1A | 154.3 (4) | O2B—C1B—C2B—N1B | −153.2 (5) |
| O1A—C1A—C2A—N1A | −25.2 (5) | O1B—C1B—C2B—N1B | 27.4 (6) |
| O2A—C1A—C2A—C3A | −86.4 (5) | O2B—C1B—C2B—C3B | 90.5 (7) |
| O1A—C1A—C2A—C3A | 94.1 (5) | O1B—C1B—C2B—C3B | −89.0 (6) |
| N1A—C2A—C3A—C4A | 50.6 (6) | N1B—C2B—C3B—C4B | −45.0 (7) |
| C1A—C2A—C3A—C4A | −67.7 (5) | C1B—C2B—C3B—C4B | 72.8 (7) |
| C2A—C3A—C4A—O4A | 177.9 (4) | C2B—C3B—C4B—O4B | 172.2 (6) |
| C2A—C3A—C4A—O3A | −2.3 (7) | C2B—C3B—C4B—O3B | −7.7 (9) |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1*A*—H11*A*···O2*A*$^{iii}$ | 0.92 | 2.29 | 2.930 (5) | 126 |
| N1*A*—H11*A*···O4*A*$^{iv}$ | 0.92 | 2.32 | 3.011 (5) | 131 |

| | | | | |
|---|---|---|---|---|
| N1*A*—H12*A*···O4*A*$^{\rm v}$ | 0.92 | 2.06 | 2.981 (5) | 177 |
| N1*B*—H11*B*···O5$^{\rm vi}$ | 0.92 | 2.04 | 2.942 (6) | 168 |
| N1*B*—H12*B*···O1*B*$^{\rm iii}$ | 0.92 | 2.34 | 2.925 (5) | 121 |
| N1*B*—H12*B*···O1*A*$^{\rm iii}$ | 0.92 | 2.49 | 3.166 (5) | 130 |
| O5—H5*A*···O4*B* | 0.84 | 1.87 | 2.685 (6) | 163 |
| O5—H5*B*···O2*B*$^{\rm vii}$ | 0.84 | 1.94 | 2.777 (6) | 169 |
| O6—H6*A*···O5$^{\rm vii}$ | 0.84 | 2.07 | 2.826 (6) | 149 |
| O6—H6*B*···O4*A*$^{\rm viii}$ | 0.84 | 2.08 | 2.914 (6) | 174 |
| O7—H7*A*···O6$^{\rm ix}$ | 0.84 | 2.23 | 3.074 (11) | 17 |
| O7—H7*B*···O4*B* | 0.84 | 2.20 | 3.038 (10) | 179 |
| C2*B*—H21*B*···O3*B*$^{\rm iii}$ | 1.00 | 2.48 | 3.419 (7) | 156 |
| C3*A*—H32*A*···O5$^{\rm x}$ | 0.99 | 2.59 | 3.328 (7) | 132 |

Symmetry codes: (iii) *x*, −*y*+1/2, *z*−1/2; (iv) −*x*+2, *y*−1/2, −*z*+1/2; (v) −*x*+2, −*y*+1, −*z*; (vi) −*x*+1, −*y*+1, −*z*; (vii) −*x*+1, *y*+1/2, −*z*+1/2; (viii) *x*, −*y*+3/2, *z*+1/2; (ix) *x*, −*y*+3/2, *z*−1/2; (x) *x*+1, *y*, *z*.