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Na₃Co₂(AsO₄)(As₂O₇): a new sodium cobalt arsenate

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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{As}-\text{O}) = 0.004$ Å; disorder in main residue; R factor = 0.027; wR factor = 0.067; data-to-parameter ratio = 11.5.

In the title compound, trisodium dicobalt arsenate diarsenate, Na₃Co₂AsO₄As₂O₇, the two Co atoms, one of the two As and three of the seven O atoms lie on special positions, with site symmetries 2 and m for the Co, m for the As, and 2 and twice m for the O atoms. The two Na atoms are disordered over two general and special positions [occupancies 0.72 (3):0.28 (3) and 0.940 (6):0.060 (6), respectively]. The main structural feature is the association of the CoO₆ octahedra in the ab plane, forming Co₄O₂₀ units, which are corner- and edge-connected *via* AsO₄ and As₂O₇ arsenate groups, giving rise to a complex polyhedral connectivity with small tunnels, such as those running along the b - and c -axis directions, in which the Na⁺ ions reside. The structural model is validated by both bond-valence-sum and charge-distribution methods, and the distortion of the coordination polyhedra is analyzed by means of the effective coordination number.

Related literature

For related structures, see: Ruiz-Valero *et al.* (1996); Ben Smail & Jouini (2005); Guesmi & Driss (2002*a,b*). For bond-valence analysis, see: Brown (2002); Adams (2003). For the charge distribution method, see: Nespolo *et al.* (2001); Nespolo (2001); Guesmi *et al.* (2006).

Experimental

Crystal data

Na ₃ Co ₂ (AsO ₄)(As ₂ O ₇)	$V = 963.2$ (3) Å ³
$M_r = 587.59$	$Z = 4$
Monoclinic, $C2/m$	Mo $K\alpha$ radiation
$a = 10.484$ (3) Å	$\mu = 13.87$ mm ⁻¹
$b = 16.309$ (2) Å	$T = 293$ K
$c = 6.531$ (1) Å	$0.20 \times 0.10 \times 0.10$ mm
$\beta = 120.40$ (2)°	

Data collection

Enraf–Nonius CAD-4 diffractometer	1183 independent reflections
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	998 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.168$, $T_{\max} = 0.338$	$R_{\text{int}} = 0.041$
1765 measured reflections	2 standard reflections every 120 min
	intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$	103 parameters
$wR(F^2) = 0.067$	$\Delta\rho_{\text{max}} = 0.72$ e Å ⁻³
$S = 1.01$	$\Delta\rho_{\text{min}} = -0.80$ e Å ⁻³
1183 reflections	

Table 1

Bond-valence-sum and charge distribution analysis.

Cation	$q(i)$ - $sof(i)$	$V(i)$	$Q(i)$	CN(i)	ECOn(i)	$d_{\text{moy}}(i)$	$d_{\text{med}}(i)$
Co1	2.00	1.87	2.01	6	5.97	2.14	2.14
Co2	2.00	2.05	2.03	6	5.62	2.11	2.08
As1	5.00	4.99	5.13	4	3.99	1.69	1.69
As2	5.00	4.93	4.94	4	3.89	1.70	1.69
Na1A	0.72	0.65	0.72	8	7.33	2.67	2.63
Na1B	0.28	0.25	0.27	8	5.72	2.74	2.58
Na2A	0.94	1.02	0.92	7	5.52	2.58	2.45
Na2B	0.06	0.07	0.06	6	4.78	2.45	2.31

$q(i)$ = formal oxidation number; $sof(i)$ = site occupation factor; $d_{\text{moy}}(i)$ = arithmetic average distance (Å); $d_{\text{med}}(i)$ = weighted average distance (Å); sodium CNS for $d(\text{Na}-\text{O})_{\text{max}} = 3.10$ Å; σ_{cat} = dispersion factor on cationic charges measuring the deviation of the computed charges (Q) with respect to the formal oxidation numbers; $\sigma_{\text{cat}} = [\sum_i (q_i - Q_i)^2 / N - 1]^{1/2} = 0.055$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1995); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2142).

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supporting information

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Na₃Co₂(AsO₄)(As₂O₇): a new sodium cobalt arsenate**Abderrahmen Guesmi and Ahmed Driss****S1. Comment**

The rich chemistry of the A–Co–P/As–O crystallographic systems (A is a monovalent cation), has been shown by the synthesis and crystal structures of several compounds with particular crystallographic properties such as:

Na₄Co₃(PO₄)₂P₂O₇, a phosphate containing a three-dimensional system of large intersecting tunnels (Ruiz-Valero *et al.* 1996), AgCo₃H₂(PO₄)₃, an alluaudite-like phosphate structure (Guesmi & Driss, 2002*a*), K₂CoP₂O₇, a layered tetrahedral phosphate with the mellilite structure (Guesmi & Driss, 2002*b*), *etc.*

For the case of arsenates, their main structural difference if compared to phosphates is that arsenic atoms can also adopt an octahedral coordination; it is the case for example of the oxygen-deficient layered sodium arsenate Na₇As₁₁O₃₁ (Guesmi *et al.* 2006). Continuous investigations on the crystal chemistry of the arsenates are performed because arsenic is at the top of the priority of the most hazardous substances, but less is known about its crystal structures.

We are interested in the present work in the crystal structure of the new compound Na₃Co₂AsO₄As₂O₇ (I). The crystal structure of the isostructural Na₃Ni₂(As_{0.1}P_{0.9})O₄(As_{1.3}P_{0.7})O₇ compound and ionic conductivity properties of its limiting arsenate has been studied (Ben Smail & Jouini, 2005). The chemical formula of (I) has been established as a result of the crystal structure determination and the obtained structural model is validated by means of charge distribution (CD) (Nespolo *et al.* 2001, Nespolo, 2001) and bond valence sum methods (BVS) (Brown, 2002; Adams, 2003) as the formal charges (Q) and valences (V) agree well with the expected values (Table 1).

The new compound (I) is an example of a mixed transition-metal arsenate, representing the first cobalt arsenate built up from mono- and diarsenate groups. In the asymmetric unit, the crystal structure is built up from corner and edge-sharing between cobalt octahedra and arsenate groups (Fig. 1). The two crystallographically distinct cobalt atoms exhibit a slightly distorted octahedral coordination with effective coordination numbers ECoN(Co1)=5.97 and ECoN(Co2)=5.62 and weighted average distances $d_{\text{med}}(\text{Co1})=2.14 \text{ \AA}$ and $d_{\text{med}}(\text{Co2})=2.08 \text{ \AA}$. The longest Co–O₆ bond distances in the two octahedra correspond to the three-coordinated oxygen atom, related also to As1.

The As1 tetrahedron, with a 2 + 2 coordination, shares its four corners with five octahedra. The As2 tetrahedron, a more precisely a trigonal pyramid (1 + 3 coordination), is more distorted with O5 as a bridging oxygen in the As(2)₂O₇ group (ECoN(As2)=3.89 and $d_{\text{med}}(\text{As2})=1.69 \text{ \AA}$). The other six corners in the diarsenate group are common with four Co1 and two Co2 octahedra. It is worth noting that the Co2 and As1 polyhedra share a common edge which induces a strong repulsion between positive charges; this type of connection was also observed in the structure of Na₄Co₃(PO₄)₂P₂O₇ (Ruiz-Valero *et al.* 1996).

The cobalt octahedra are associated in the *ab* plane to form the original octahedral metallic units Co₄O₂₀ which are corner- and edge-connected *via* As(1)O₄ and As(2)₂O₇ arsenate groups, giving rise to a complex polyhedral connectivity which produces small tunnels, such as those running along the *b* and *c* axis, where the sodium cations reside (Figs. 2–4).

The anionic framework can be decomposed in a succession of alternate layers in the *ac* plane, stacked along the crystallographic *b*-axis. They are built up of Co1 octahedra and As(2)₂O₇ groups in such a way that each octahedron is corner-shared to four diarsenate groups (Fig. 3). These layers are alternate by a chain type resulting from the connection between Co2 and As1 polyhedra and formed by the centrosymmetric cyclic units [Co₂As₂O₁₄] (Fig. 4), each one of these units is connected to two neighbours by means of mixed Co–O–As bridges.

The Na1 ions are split into two independent positions near *c*/2, Na1B has the more distorted polyhedron and the ECoN(Na1B) is as low as 5.72. The Na2 ions are also disordered with the Na2B polyhedron sandwiched by Na2A ones which are off-centred around the Na2B positions. The motion of sodium cations within the framework of (I) by means of theoretical studies and electrical measurements will be the subject of future works.

S2. Experimental

The investigated compound was synthesized by a solid state reaction from a mixture of Na₂CO₃ (0.46 g, Fluka, 99.0%), cobalt (II and III) oxides (0.1 g, Fluka, 99.0%, Co 71% min.) and As₂O₅ (0.33 g, Prolabo). The reaction mixture was heated at 673 K for 24 h and progressively at 923 K and kept at this temperature for three days. Finally, it was slowly cooled to room temperature. The obtained pink crystals were separated from the excess flux by washing the product in boiling water.

S3. Refinement

The non-equivalent sodium ions are inserted in the anionic framework first in two full-occupied general and special crystallographic sites. The Na1 atoms are better described by a split model with two independent general positions, refined with the same thermal parameters. The highest Fourier peaks near the Na2A site suggests that the Na2A position deviates from the full occupancy and another partial-occupied position (Na2B) was introduced in the model, leading to a lowering of *R* values and residual electron density peaks.

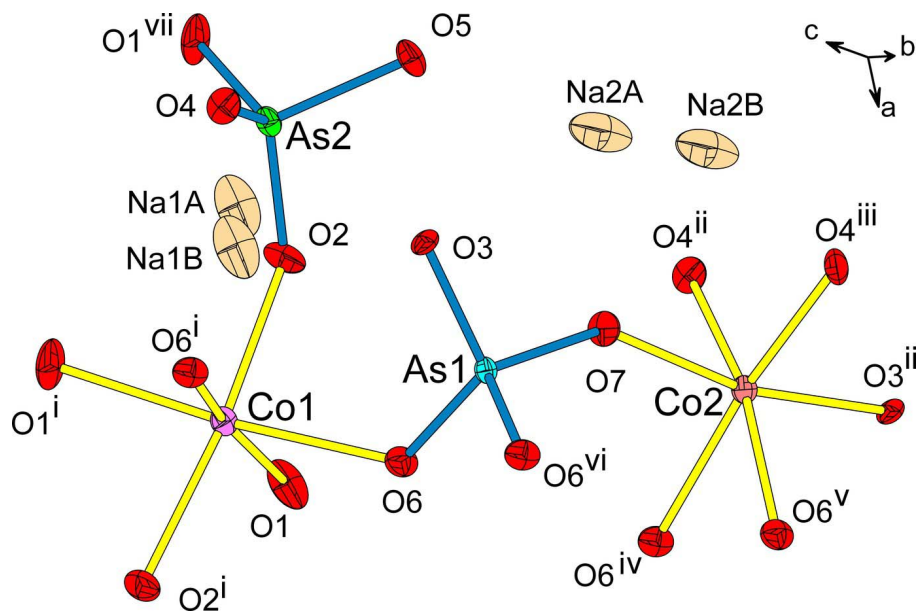


Figure 1

The asymmetric unit in (I) with atom-labelling scheme. Some symmetry-related O atoms are included to show the full coordination polyhedra around the Co and As atoms. Displacement ellipsoids are drawn at the 50% probability level [Symmetry codes: (i) $-x+1, y, -z+2$; (ii) $x, y, z-1$; (iii) $x, -y+1, z-1$; (iv) $-x+1, y, -z+1$; (v) $-x+1, -y+1, -z+1$; (vi) $x, -y+1, z$; (vii) $x-1/2, -y+1/2, z$.]

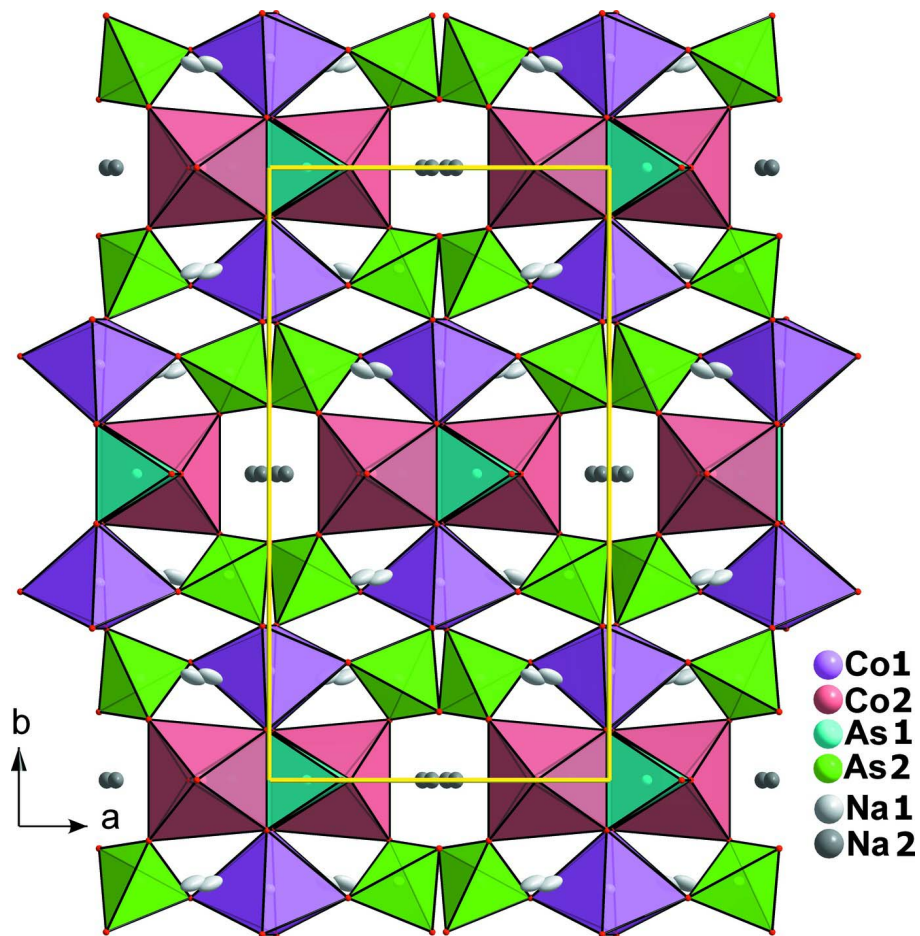


Figure 2
Polyhedron framework structure of (I) viewed along the c axis.

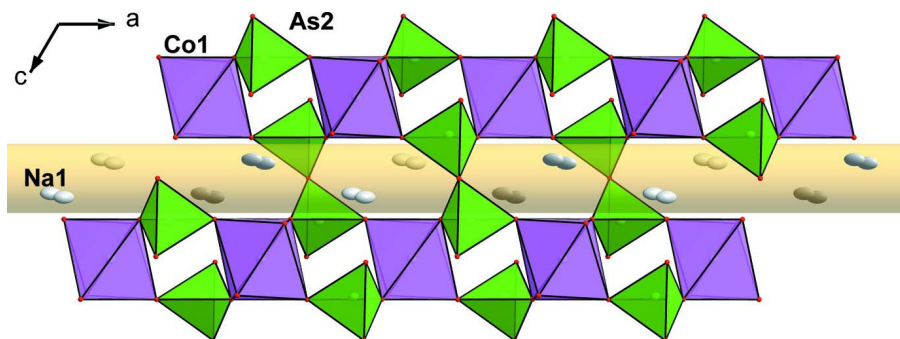


Figure 3
The polyhedral layers in the framework of (I); Na1 cations are on the periphery of tunnels parallel to $[100]$.

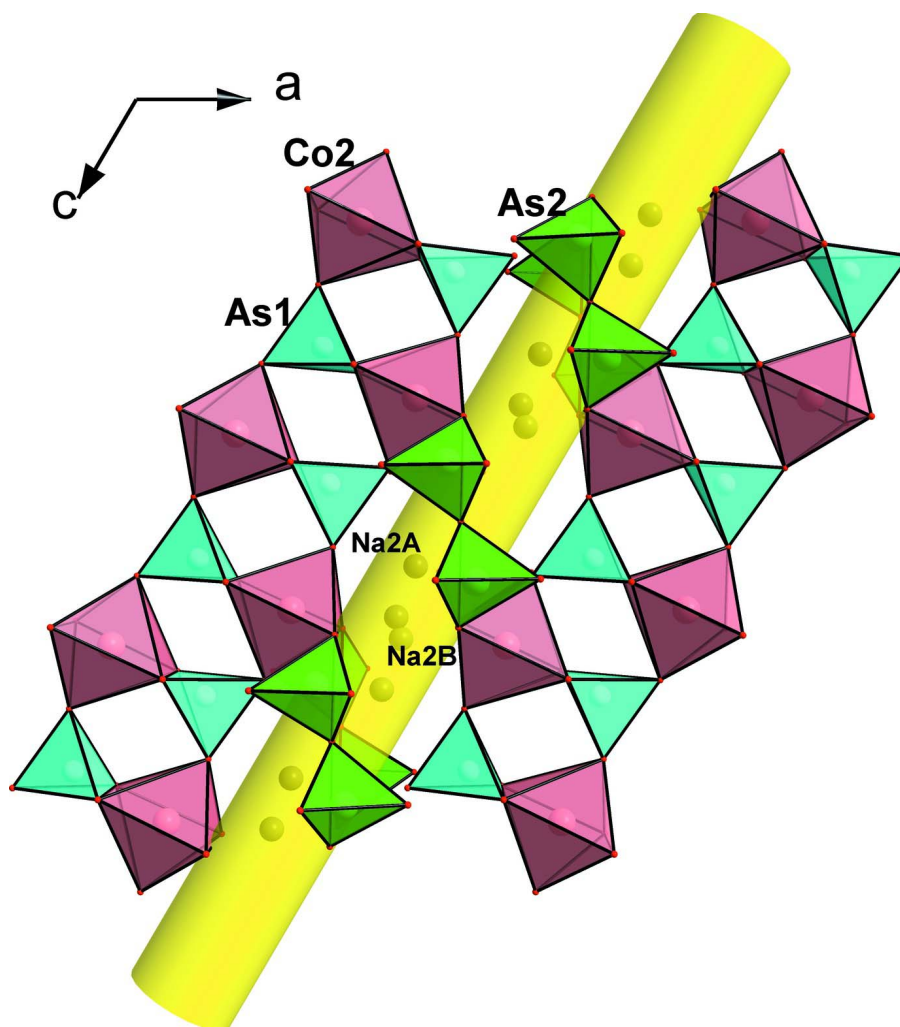


Figure 4

The connection between the chains parallel to [001]; Na2 cations are inside the resulted tunnels.

trisodium dicobalt arsenate diarsenate

Crystal data

$\text{Na}_3\text{Co}_2(\text{AsO}_4)(\text{As}_2\text{O}_7)$

$M_r = 587.59$

Monoclinic, $C2/m$

Hall symbol: $-C 2y$

$a = 10.484 (3) \text{ \AA}$

$b = 16.309 (2) \text{ \AA}$

$c = 6.531 (1) \text{ \AA}$

$\beta = 120.40 (2)^\circ$

$V = 963.2 (3) \text{ \AA}^3$

$Z = 4$

$F(000) = 1096$

$D_x = 4.052 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 11.7\text{--}14.5^\circ$

$\mu = 13.87 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Parallelepiped, pink

$0.20 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer	1183 independent reflections 998 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.041$
Graphite monochromator	$\theta_{\text{max}} = 28.0^\circ$, $\theta_{\text{min}} = 2.5^\circ$
$\omega/2\theta$ scans	$h = -13 \rightarrow 13$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$k = -1 \rightarrow 21$
$T_{\text{min}} = 0.168$, $T_{\text{max}} = 0.338$	$l = -8 \rightarrow 3$
1765 measured reflections	2 standard reflections every 120 min intensity decay: 1%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0261P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.027$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.067$	$(\Delta/\sigma)_{\text{max}} = 0.005$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.72 \text{ e } \text{\AA}^{-3}$
1183 reflections	$\Delta\rho_{\text{min}} = -0.80 \text{ e } \text{\AA}^{-3}$
103 parameters	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.00124 (17)
Primary atom site location: structure-invariant direct methods	

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	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Co1	0.5000	0.32196 (5)	1.0000	0.0091 (2)	
Co2	0.29864 (9)	0.5000	0.08408 (15)	0.0090 (2)	
As1	0.38970 (6)	0.5000	0.67693 (11)	0.00692 (16)	
As2	0.11973 (5)	0.33430 (3)	0.76867 (8)	0.00892 (14)	
O1	0.5206 (4)	0.2480 (2)	0.7535 (6)	0.0219 (8)	
O2	0.2686 (3)	0.3079 (2)	0.7525 (6)	0.0173 (7)	
O3	0.2608 (5)	0.5000	0.7550 (8)	0.0097 (9)	
O4	0.1460 (3)	0.40124 (19)	0.9821 (6)	0.0136 (6)	
O5	0.0000	0.3884 (3)	0.5000	0.0133 (9)	
O6	0.5077 (3)	0.41858 (19)	0.7766 (6)	0.0126 (6)	
O7	0.2869 (5)	0.5000	0.3808 (8)	0.0193 (11)	
Na1A	0.1740 (18)	0.1692 (4)	0.5940 (15)	0.032 (2)	0.72 (3)
Na1B	0.225 (3)	0.1686 (13)	0.617 (4)	0.032 (2)	0.28 (3)
Na2A	0.0488 (3)	0.5000	0.2908 (8)	0.0353 (11)	0.940 (6)
Na2B	-0.021 (6)	0.5000	-0.054 (13)	0.0353 (11)	0.060 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0097 (4)	0.0075 (4)	0.0088 (4)	0.000	0.0037 (3)	0.000
Co2	0.0094 (4)	0.0105 (4)	0.0067 (4)	0.000	0.0038 (3)	0.000
As1	0.0077 (3)	0.0073 (3)	0.0047 (3)	0.000	0.0023 (2)	0.000
As2	0.0091 (2)	0.0071 (2)	0.0073 (2)	-0.00090 (15)	0.00172 (18)	0.00007 (17)
O1	0.0297 (19)	0.0157 (17)	0.015 (2)	0.0132 (15)	0.0073 (16)	-0.0024 (15)
O2	0.0091 (14)	0.0211 (18)	0.0157 (18)	0.0020 (13)	0.0018 (14)	-0.0030 (14)
O3	0.0093 (19)	0.015 (2)	0.008 (2)	0.000	0.0072 (18)	0.000
O4	0.0163 (14)	0.0152 (15)	0.0089 (15)	-0.0044 (13)	0.0061 (13)	-0.0053 (14)
O5	0.0128 (19)	0.011 (2)	0.008 (2)	0.000	-0.0008 (18)	0.000
O6	0.0114 (13)	0.0098 (14)	0.0158 (16)	0.0014 (12)	0.0064 (13)	0.0017 (13)
O7	0.014 (2)	0.040 (3)	0.004 (2)	0.000	0.005 (2)	0.000
Na1A	0.047 (6)	0.0192 (12)	0.021 (2)	0.012 (3)	0.011 (4)	-0.0034 (12)
Na1B	0.047 (6)	0.0192 (12)	0.021 (2)	0.012 (3)	0.011 (4)	-0.0034 (12)
Na2A	0.0179 (15)	0.0196 (17)	0.059 (3)	0.000	0.0126 (18)	0.000
Na2B	0.0179 (15)	0.0196 (17)	0.059 (3)	0.000	0.0126 (18)	0.000

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

Co1—O1	2.108 (3)	Na1A—O6 ^{viii}	2.621 (11)
Co1—O1 ⁱ	2.108 (3)	Na1A—O2 ^{viii}	2.646 (11)
Co1—O2	2.141 (3)	Na1A—O1 ^{vii}	2.682 (13)
Co1—O2 ⁱ	2.141 (3)	Na1A—O4 ^{ix}	2.694 (10)
Co1—O6	2.177 (3)	Na1A—O7 ^{viii}	2.782 (7)
Co1—O6 ⁱ	2.177 (3)	Na1A—O6 ^{vii}	2.936 (13)
Co2—O3 ⁱⁱ	1.978 (4)	Na1B—O2	2.39 (2)
Co2—O7	2.003 (5)	Na1B—O2 ^{viii}	2.47 (2)
Co2—O4 ⁱⁱ	2.126 (3)	Na1B—O4 ^{ix}	2.53 (2)
Co2—O4 ⁱⁱⁱ	2.126 (3)	Na1B—O7 ^{viii}	2.75 (2)
Co2—O6 ^{iv}	2.201 (3)	Na1B—O1 ^{viii}	2.83 (2)
Co2—O6 ^v	2.201 (3)	Na1B—O6 ^{viii}	2.87 (2)
As1—O3	1.669 (4)	Na2A—O7	2.257 (5)
As1—O7	1.671 (5)	Na2A—O5	2.480 (4)
As1—O6 ^{vi}	1.704 (3)	Na2A—O5 ^x	2.480 (4)
As1—O6	1.704 (3)	Na2A—O4 ^{xi}	2.501 (4)
As2—O1 ^{vii}	1.670 (3)	Na2A—O4 ^x	2.501 (4)
As2—O2	1.673 (3)	Na2B—O4 ^{xi}	2.27 (6)
As2—O4	1.680 (3)	Na2B—O4 ^x	2.27 (6)
As2—O5	1.790 (2)	Na2B—O4 ⁱⁱ	2.30 (5)
Na1A—O2	2.475 (9)	Na2B—O4 ⁱⁱⁱ	2.30 (5)
Na1A—O1 ^{viii}	2.540 (12)	Na2B—O7 ^{xii}	2.51 (5)
O1—Co1—O1 ⁱ	110.2 (2)	O3 ⁱⁱ —Co2—O6 ^{iv}	94.87 (13)
O1—Co1—O2	82.94 (14)	O7—Co2—O6 ^{iv}	95.48 (14)
O1 ⁱ —Co1—O2	90.01 (14)	O4 ⁱⁱ —Co2—O6 ^{iv}	93.63 (12)
O1—Co1—O2 ⁱ	90.01 (14)	O4 ⁱⁱⁱ —Co2—O6 ^{iv}	167.77 (12)

O1 ⁱ —Co1—O2 ⁱ	82.94 (14)	O3 ⁱⁱ —Co2—O6 ^v	94.87 (13)
O2—Co1—O2 ⁱ	167.7 (2)	O7—Co2—O6 ^v	95.48 (14)
O1—Co1—O6	81.33 (13)	O4 ⁱⁱ —Co2—O6 ^v	167.77 (12)
O1 ⁱ —Co1—O6	168.19 (14)	O4 ⁱⁱⁱ —Co2—O6 ^v	93.63 (12)
O2—Co1—O6	89.07 (13)	O6 ^{iv} —Co2—O6 ^v	74.22 (16)
O2 ⁱ —Co1—O6	99.88 (13)	O3—As1—O7	101.9 (2)
O1—Co1—O6 ⁱ	168.19 (14)	O3—As1—O6 ^{vi}	115.27 (14)
O1 ⁱ —Co1—O6 ⁱ	81.33 (13)	O7—As1—O6 ^{vi}	111.14 (15)
O2—Co1—O6 ⁱ	99.88 (13)	O3—As1—O6	115.27 (14)
O2 ⁱ —Co1—O6 ⁱ	89.07 (13)	O7—As1—O6	111.14 (14)
O6—Co1—O6 ⁱ	87.23 (18)	O6 ^{vi} —As1—O6	102.4 (2)
O3 ⁱⁱ —Co2—O7	167.01 (18)	O1 ^{vii} —As2—O2	111.18 (19)
O3 ⁱⁱ —Co2—O4 ⁱⁱ	87.38 (12)	O1 ^{vii} —As2—O4	114.04 (17)
O7—Co2—O4 ⁱⁱ	84.15 (12)	O2—As2—O4	116.98 (16)
O3 ⁱⁱ —Co2—O4 ⁱⁱⁱ	87.38 (12)	O1 ^{vii} —As2—O5	103.39 (16)
O7—Co2—O4 ⁱⁱⁱ	84.15 (12)	O2—As2—O5	106.19 (13)
O4 ⁱⁱ —Co2—O4 ⁱⁱⁱ	98.49 (17)	O4—As2—O5	103.43 (16)

Symmetry codes: (i) $-x+1, y, -z+2$; (ii) $x, y, z-1$; (iii) $x, -y+1, z-1$; (iv) $-x+1, y, -z+1$; (v) $-x+1, -y+1, -z+1$; (vi) $x, -y+1, z$; (vii) $x-1/2, -y+1/2, z$; (viii) $-x+1/2, -y+1/2, -z+1$; (ix) $-x+1/2, -y+1/2, -z+2$; (x) $-x, -y+1, -z+1$; (xi) $-x, y, -z+1$; (xii) $-x, -y+1, -z$.