inorganic compounds

Disodium tris(dioxidomolybdenum) bis(diarsenate)

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The asymmetric unit of the title compound, $Na_2(MoO_2)_3(As_2O_7)_2$, is composed of two cyclic $MoAs_2O_{11}$ units and an MoO_6 corner-sharing octahedron. The anionic framework can be decomposed into two types of layers, *viz.* $MoO_2As_2O_7$ and $Mo_2As_2O_{14}$, which use mixed Mo—O—As and As—O—Mo bridges to achieve a new three-dimensional structure with two types of large channels in which the Na^+ cations are located. Two O atoms are disordered and are located in two positions close to their initial positions with occupancy ratios of 0.612 (17):0.388 (17) and 0.703 (12):0.298 (12).

Related literature

For background to the search for new materials with open structures, see: Kierkegaard & Westerlund (1964); Lii *et al.* (1987); Guesdon *et al.* (1994); Masquelier *et al.* (1995). In these materials, the association of XO_4 (X = P, As) tetrahedra and MO_6 (M = transition metal) octahedra forms covalent hybrid structures that delimit tunnels, see: Linnros (1970); Hammond & Barbier (1996). For details of the preparation, see: Zid & Jouini (1996a,b); Zid *et al.* (1997, 1998); Hajji *et al.* (2004); Hajji & Zid (2006); Ben Hlila *et al.* (2009). For related structures, see: Averbuch-Pouchot (1988, 1989); Zid *et al.* (2003); Lii & Wang (1989); Benhamada *et al.* (1992). For the properties of related compounds, see: Marzouki *et al.* (2010); Ouerfelli *et al.* (2007). For background to the bond-valence method, see: Brown & Altermatt (1985).

Experimental

Crystal data

$Na_2(MoO_2)_3(As_2O_7)_2$	c = 9.258 (2) Å
M_r = 953.48	β = 94.51 (2)°
Monoclinic, $P2_1/c$	V = 1691.9 (6) Å^3
a = 14.571 (3) Å	Z = 4
b = 12.580 (2) Å	Mo $K\alpha$ radiation

μ = 10.11 mm^{-1}	0.26 × 0.18 × 0.14 mm
T = 298 K	

Data collection

Enraf–Nonius CAD-4 diffractometer	3676 independent reflections
Absorption correction: ψ scan (North *et al.*, 1968)	3128 reflections with $I > 2\sigma(I)$
T_{min} = 0.133, T_{max} = 0.246	R_{int} = 0.022
4271 measured reflections	2 standard reflections every 120 min intensity decay: 1.1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025	273 parameters
$wR(F^2)$ = 0.060	2 restraints
S = 1.06	$\Delta\rho_{max}$ = 0.98 e Å^{-3}
3676 reflections	$\Delta\rho_{min}$ = −0.75 e Å^{-3}

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); 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, 1998); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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supplementary materials

Disodium tris(dioxidomolybdenum) bis(diarsenate)

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Comment

The search for new materials with open structure has motivated many works (Kierkegaard & Westerlund, 1964; Lii *et al.*, 1987; Guesdon *et al.*, 1994; Masquelier *et al.*, 1995). In these materials, the association of XO_4 (*X*=P, As) tetrahedra and MO_6 (*M*= transition metal) octahedra forms covalent hybrid structures that delimit tunnels (Linnros, 1970; Hammond & Barbier, 1996) or interlayers favorable to the migration of cations. For instance, the junction between these polyhedra can develop new materials that could have properties associated with the ion mobility of metal cations. This area is far from being fully explored and represents a field of considerable activity including several disciplines. In this context the A—Mo—As—O (A = monovalent cation) systems were explored and several interesting phases: $K_2MoO_2As_2O_7$ (Zid & Jouini, 1996*a*), $Rb_2MoO_2As_2O_7$ (Zid *et al.*, 1998) and $K_2MoO_2(MoO_2As_2O_7)_2$ (Zid & Jouini, 1996*b*) were characterized. Herein, we describe the synthesis of a new material of monoclinic symmetry by solid state reaction. The method of preparation, structure determination by X-ray diffraction on single-crystal and physical properties are presented. The new $Na_2(MoO_2)_3(As_2O_7)_2$ phase presents a three-dimensional framework. The asymmetric unit is built from two cyclic $MoAs_2O_{11}$ units and MoO_6 corner-sharing octahedron (Fig. 1). The anionic framework of $Na_2(MoO_2)_3(As_2O_7)_2$ can be decomposed into two layers: the first found in the material $K_2MoO_2(MoO_2As_2O_7)_2$ (Zid & Jouini, 1996*b*), and the second $Mo_2As_2O_{14}$ layer which is new. Layers $(Mo2O_2As_2O_7)$ are constructed from conventional $Mo2As_2O_{11}$ units in which each diarsenate group shares two oxygen atoms with the $Mo2O_6$ octahedron. Units $Mo2As_2O_{11}$ are pairs connected to form double $[Mo2_2As_4O_{20}]$ units. The junction between the latter sharing corners with polyhedra of different nature, leads to the $(Mo2O_2As_2O_7)$ layer (Fig. 2) in which each $Mo2O_6$ octahedron shares four of its corners with only three As_2O_7 groups. The other two free remaining corners form the $Mo2O_2$ molybdyl group directed to large elongated parallel channels arranged to the [100] direction resulting from the combination of four double $[Mo2_2As_4O_{20}]$ units. The layers of the second type are built from new units of formula $Mo_2As_2O_{16}$ (Fig. 3). These are due to the association between a conventional $Mo1As_2O_{11}$ unit and $Mo3O_6$ octahedron by corner-sharing. In addition, each $Mo_2As_2O_{16}$ unit binds to its centrosymmetric according to [001] direction forming double $Mo_4As_4O_{30}$ units. The latter are connected by corner-sharing and form new layers of $Mo_2As_2O_{14}$ type, which are organized in a wavy arrangement according to the plans (010) (Fig. 4). Within these layers there are two types of channels, hexagonal or square sections, arranged in the [010] direction. The anionic framework is constructed by the two types of layers previously described $(Mo2O_2As_2O_7)$) (Fig. 2) and $Mo_2As_2O_{14}$ (Fig. 4) using mixte Mo—O—As and As—O—Mo bridges leading to a new three-dimensional framework with large channels of two types where the Na$^+$ cations are located (Fig. 5). The average Mo—O, As—O and Na—O distances in the structure are consistent with those found in the literature (Zid *et al.*, 1997; Hajji *et al.*, 2004; Hajji & Zid, 2006; Ben Hlila *et al.*, 2009). In addition, the calculation of the various valence bonds (BVS) using empirical formula of Brown (Brown & Altermatt, 1985) confirms the expected values of ion charges: Mo1(5,967), Mo2(6,017), Mo3(6,021), As1(4,966), As2(4,990), As3(5,016), As4(4,981), Na1(0,606) and Na2(0,902). The phase studied is new but the comparaison of the structure of $Na_2(MoO_2)_3(As_2O_7)_2$ with those found in the literature for related MX_2O_{11} (*X*=P, As) units

reveals some structural affiliation. In the diphosphates of formula $A_2MoO_2P_2O_7$ (A=Cs (Averbuch-Pouchot, 1988); A= NH_4 (Averbuch-Pouchot, 1989); A=K (Zid *et al.*, 2003)), MoP_2O_{11} units are connected by cornrer-sharing octahedra and tetrahedra to form ribbons leading to a one-dimensional framework. The combination of ribbons of type $MO_2X_2O_7$ (*M*= transition metal, *X*= P, As) by M—O—*X* mixed bridges leads to layered structures similar to those encountered in diphosphates of the formula $A_2VOP_2O_7$ (A=Rb (Lii & Wang, 1989); A=Na (Benhamada *et al.*, 1992)). The combination of such layers can lead to different three-dimensional frameworks. The structure of $K_2MoO_2(MoO_2As_2O_7)_2$ (Zid & Jouini, 1996*b*) was obtained by insertion of the MoO_2 group between $MoO_2As_2O_7$ layers, but in our case the two types of layers in $Na_2(MoO_2)_3(As_2O_7)_2$ are connected directly by mixed Mo—O—As and As—O—Mo bridges. In order to use the structural data obtained which are in favor of a good ion mobility and related to physicochemical properties and in particular ionic conduction, measurements of resistance as a function of temperature were carried out using an impedance analyzer of type HP4192A of a pure sample compacted in pellet and having a geometric factor of (e/s=0,017 cm^{-1}). The values of the conductivities obtained by rising temperatures correlate well with the Arrhenius law: $Ln(\sigma T) = f(10^4/T)$. These results show that this material has an activation energy of 0,79 eV and a conductivity of 4,294 10^{-5} S.cm^{-1} at 793 K indicating that the material can be classified as a mild ionic conductor. These results are comparable to those found in the literature for compounds with cobalt (Marzouki *et al.*, 2010) and iron (Ouerfelli *et al.*, 2007).

Experimental

The crystals of the $Na_2(MoO_2)_3(As_2O_7)_2$ phase were obtained from the reagents: $(NH_4)_2Mo_4O_{13}$ (Fluka, 69858), $NH_4H_2AsO_4$ (prepared in the laboratory, ASTM 01–775) and Na_2CO_3 (Prolabo, 27778) taken in Na: Mo: As molar ratio of 2: 3: 4. The finely ground mixture was preheated in air at 623 K to remove volatile compounds. It was then heated, in steps of 100 degrees followed by grinding to a temperature close to the melting point 828 K. The mixture was then left at this temperature for one week to promote germination and growth of crystals. The final residue was subjected to cooling first a slow (5°/half-day at 778 K) and a second faster (50°/h) to room temperature. Yellow crystals of sufficient size for the measurements of intensities, were separated from refluxing water. A qualitative analysis of a selected crystal by scanning electron microscopy (S.E.*M*) of the type FEI Quanta 200 under polarizing microscope, confirmed the presence of different chemical elements expected: As, Mo, Na and oxygen.

Refinement

In the final refinement, examination of the Fourier-Difference reveals the presence of two peaks which are close to the oxygen atoms O19 and O20. In addition, the thermal motion of these atoms is relatively high, so each oxygen atom is distribute between two positions with variable occupancy ratios satisfying the condition of electrical neutrality of the material. For this, the use of two conditions SUMP and EADP allowed by the *SHELX* program is necessary. The ellipsoids of the atoms considered are better defined.

Computing details

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); cell refinement: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); 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, 1998); software used to prepare material for publication: *WinGX* (Farrugia, 1999).
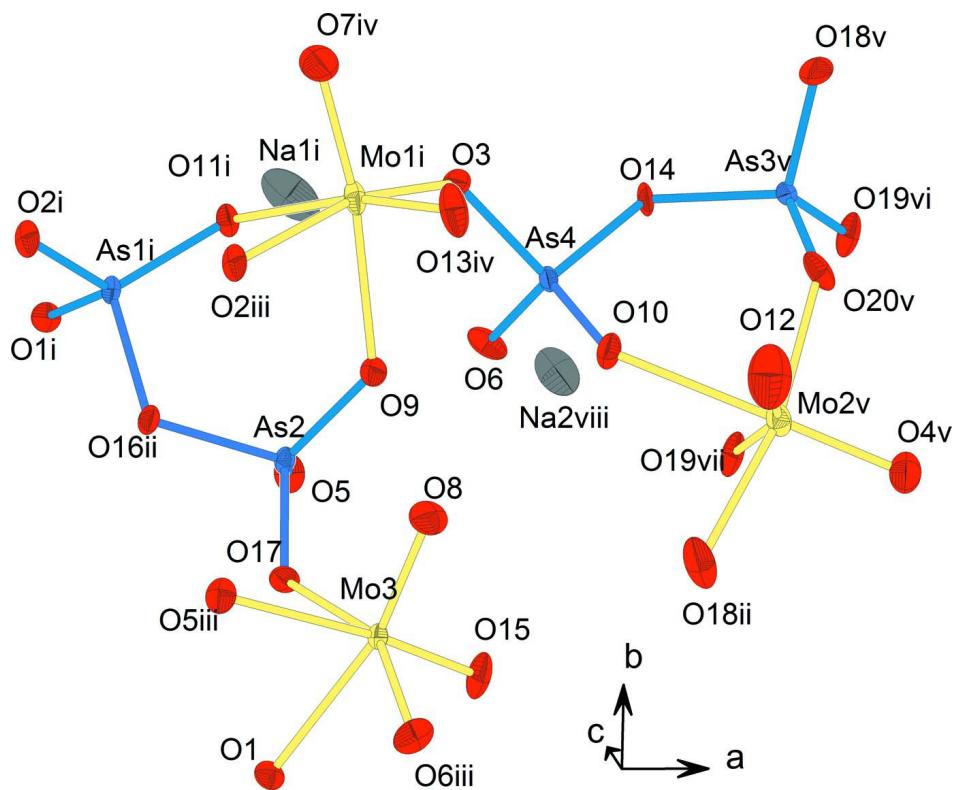
Figure 1

The asymmetric unit of $Na_2(MoO_2)_3(As_2O_7)_2$. Displacement ellipsoids are drawn at the 50% probability level. *symmetry codes*:(i) -*x* + 1, *y* + 1/2, -*z* + 1/2; (ii) -*x* + 1, *y* - 1/2, -*z* + 1/2; (iii) *x*, -*y* + 1/2, *z* + 1/2; (iv) -*x* + 1, *y* + 1/2, -*z* + 3/2; (v) *x* + 1, *y*, *z*; (vi) *x* + 1, *y*, *z* - 1; (vii) -*x* + 1, -*y* + 1, -*z* + 1; (viii) -*x* + 1, *y* - 1/2, -*z* + 3/2.
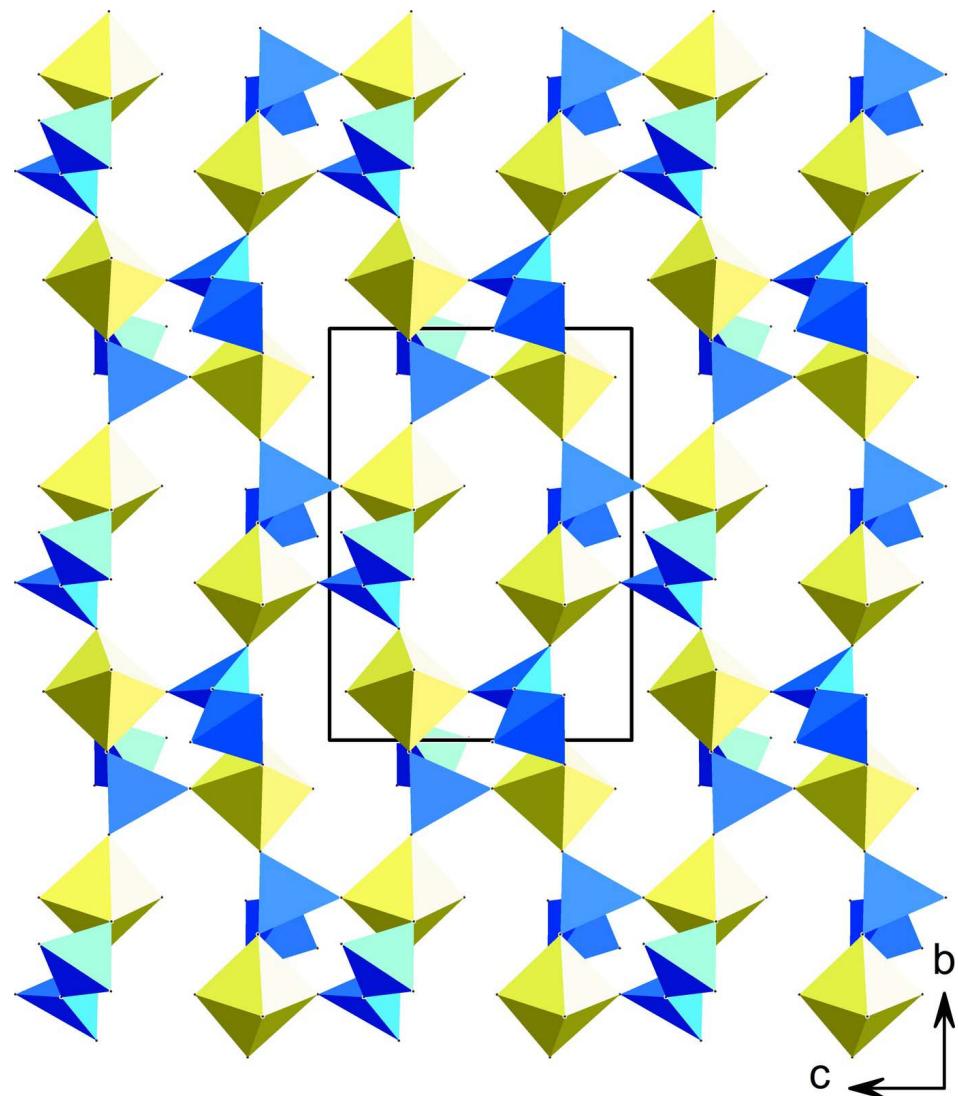
Figure 2

Representation of the (Mo2O$_2$As$_2$O$_7$) layer along the *a* axis.
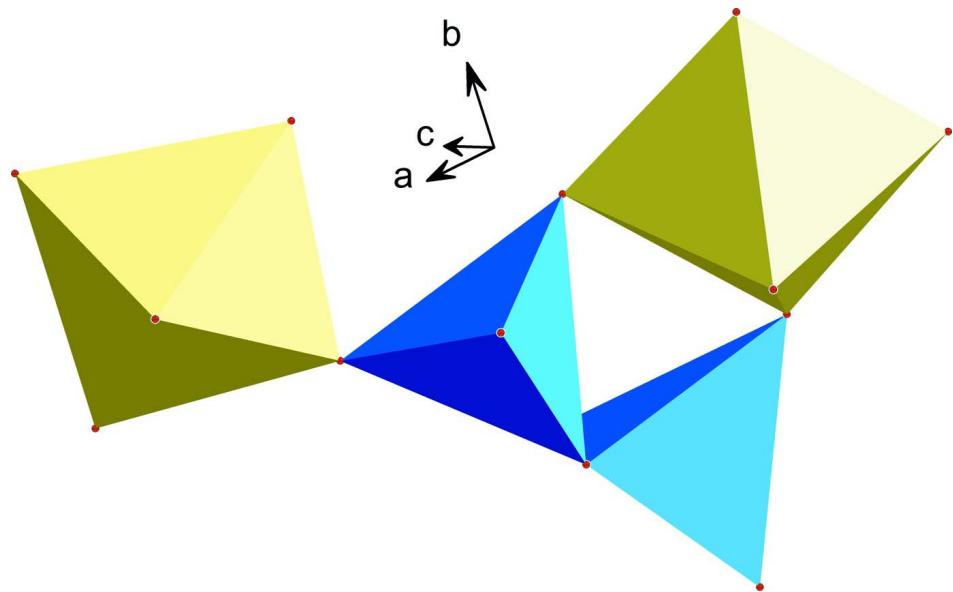
Figure 3

Representation of $Mo_2As_2O_{16}$ unit.
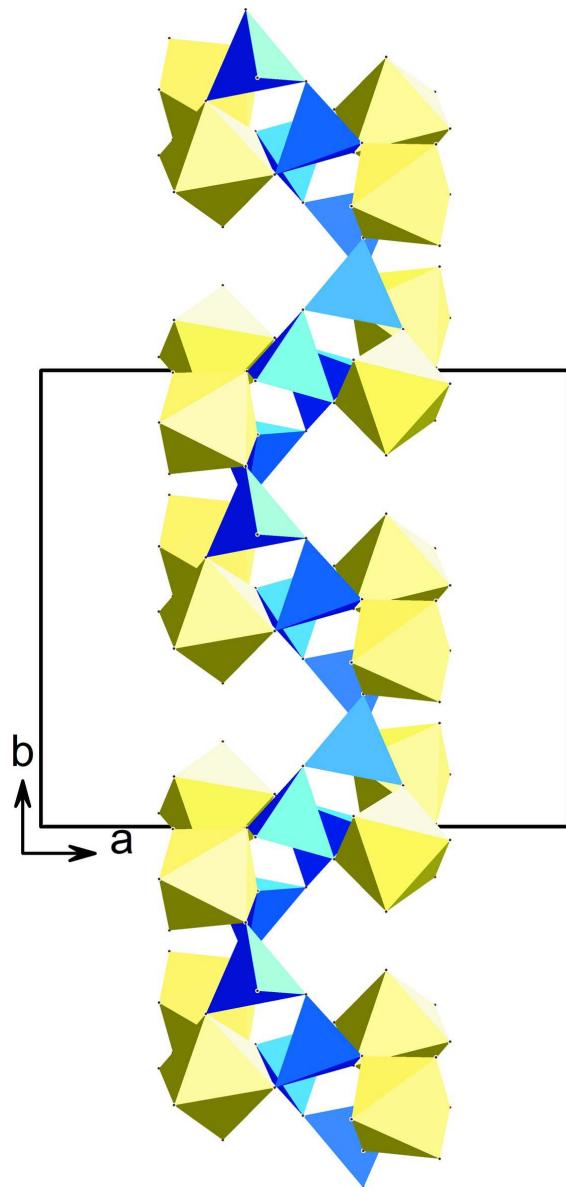
Figure 4

Projection of $Mo_2As_2O_{14}$ layer along the c axis.
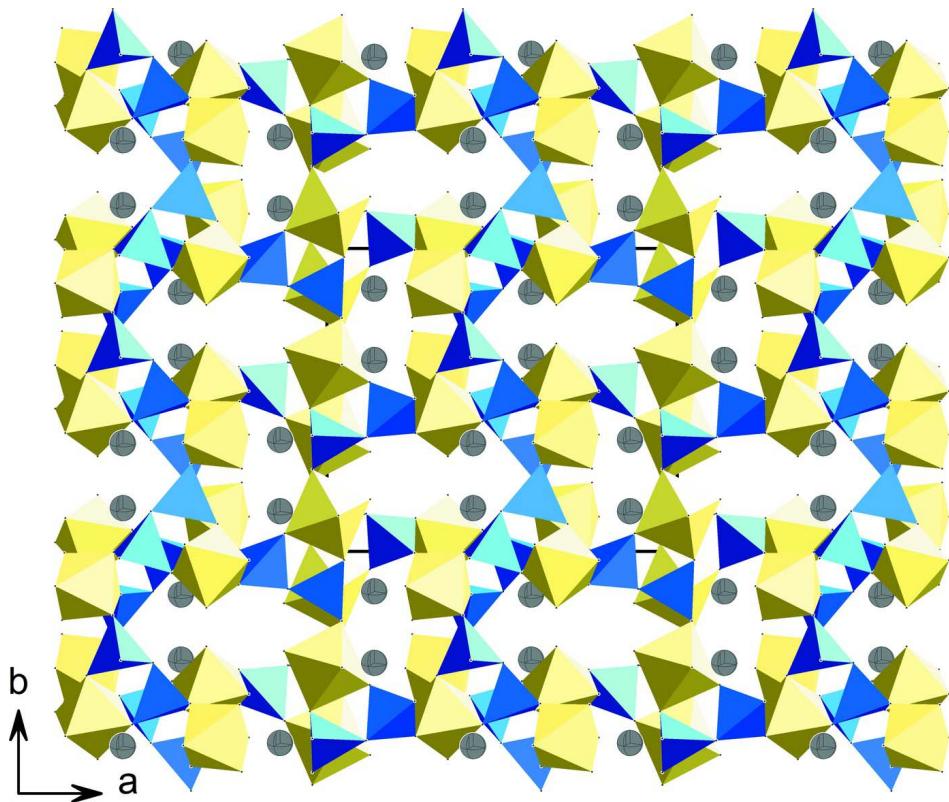
Figure 5

Projection of structure according to *c* axis, showing the channel where Na$^+$ cations are located.

Disodium tris(dioxidomolybdenum) bis(diarsenate)

Crystal data

Na$_2$(MoO$_2$)$_3$(As$_2$O$_7$)$_2$
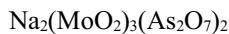
$M_r = 953.48$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 14.571$ (3) Å

$b = 12.580$ (2) Å

$c = 9.258$ (2) Å

$\beta = 94.51$ (2)°

$V = 1691.9$ (6) Å^3

$Z = 4$

$F(000) = 1760$

$D_x = 3.743$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 10–15°$

$\mu = 10.11$ mm^{-1}

$T = 298$ K

Prism, yellow

$0.26 \times 0.18 \times 0.14$ mm

Data collection

Enraf–Nonius CAD-4
    diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega/2\theta$ scans

Absorption correction: ψ scan
    (North *et al.*, 1968)

$T_{min} = 0.133$, $T_{max} = 0.246$

4271 measured reflections

3676 independent reflections

3128 reflections with $I > 2\sigma(I)$

$R_{int} = 0.022$

$\theta_{max} = 27.0°$, $\theta_{min} = 2.1°$

$h = -18 \rightarrow 18$

$k = -1 \rightarrow 16$

$l = -11 \rightarrow 1$

2 standard reflections every 120 min

intensity decay: 1.1%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.025$	$w = 1/[\sigma^2(F_o^2) + (0.0188P)^2 + 7.737P]$
$wR(F^2) = 0.060$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
3676 reflections	$\Delta\rho_{max} = 0.98$ e Å⁻³
273 parameters	$\Delta\rho_{min} = -0.75$ e Å⁻³
2 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc²λ³/sin(2θ)]⁻¹ᐟ⁴
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00106 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Mo1	0.31869 (3)	0.06243 (4)	0.05295 (4)	0.01284 (10)	
Mo2	0.02062 (3)	0.37400 (3)	0.26296 (5)	0.01470 (11)	
Mo3	0.70516 (3)	0.12821 (3)	0.42003 (4)	0.00864 (10)	
As1	0.53859 (3)	0.00042 (4)	0.20483 (5)	0.00852 (11)	
As2	0.60470 (3)	0.33825 (4)	0.24714 (5)	0.00885 (11)	
As3	0.00777 (3)	0.62745 (4)	0.12380 (6)	0.01163 (11)	
As4	0.81642 (3)	0.53068 (4)	0.18604 (5)	0.00924 (11)	
Na1	0.4171 (2)	0.1428 (2)	0.4537 (3)	0.0381 (7)	
Na2	0.13444 (18)	0.8679 (2)	0.9496 (3)	0.0343 (6)	
O1	0.6093 (2)	0.0020 (3)	0.3549 (4)	0.0129 (7)	
O2	0.5933 (2)	0.0231 (3)	0.0556 (4)	0.0146 (7)	
O3	0.7475 (2)	0.6100 (3)	0.2769 (4)	0.0150 (7)	
O4	0.1230 (3)	0.3287 (3)	0.2208 (5)	0.0291 (10)	
O5	0.5893 (2)	0.3594 (3)	0.0702 (4)	0.0163 (7)	
O6	0.7499 (3)	0.4947 (3)	0.0395 (4)	0.0189 (8)	
O7	0.3449 (3)	0.1862 (3)	0.9950 (4)	0.0276 (9)	
O8	0.7566 (3)	0.2271 (3)	0.5219 (4)	0.0225 (8)	
O9	0.6874 (2)	0.4098 (3)	0.3346 (4)	0.0136 (7)	
O10	0.8787 (2)	0.4405 (3)	0.2789 (4)	0.0161 (7)	
O11	0.4431 (2)	0.0715 (3)	0.2150 (4)	0.0125 (7)	
O12	0.0325 (3)	0.3820 (4)	0.4454 (5)	0.0425 (12)	
O13	0.2237 (3)	0.0292 (4)	0.9471 (4)	0.0259 (9)	
O14	0.8884 (2)	0.6242 (3)	0.1131 (4)	0.0149 (7)	
O15	0.7762 (2)	0.1145 (3)	0.2859 (4)	0.0222 (9)	
O16	0.5032 (2)	0.8672 (3)	0.1910 (4)	0.0123 (7)	
O17	0.6118 (2)	0.2098 (3)	0.2942 (4)	0.0147 (7)	

O18	0.0423 (3)	0.7296 (3)	0.2295 (5)	0.0366 (12)	
O19	0.0424 (7)	0.6173 (10)	0.9610 (11)	0.0191 (19)	0.612 (17)
O191	0.0395 (12)	0.6633 (14)	0.9569 (19)	0.0191 (19)	0.388 (17)
O20	0.0489 (4)	0.5281 (5)	0.2364 (9)	0.0181 (15)	0.703 (12)
O201	0.0481 (10)	0.5117 (13)	0.164 (2)	0.0181 (15)	0.298 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.00827 (19)	0.0211 (2)	0.0093 (2)	0.00355 (16)	0.00231 (15)	0.00338 (16)
Mo2	0.0095 (2)	0.0139 (2)	0.0205 (2)	−0.00034 (16)	0.00001 (17)	0.00670 (17)
Mo3	0.00620 (19)	0.0103 (2)	0.0094 (2)	0.00053 (15)	0.00019 (14)	0.00045 (15)
As1	0.0061 (2)	0.0123 (2)	0.0073 (2)	−0.00051 (17)	0.00123 (17)	−0.00062 (17)
As2	0.0072 (2)	0.0101 (2)	0.0095 (2)	0.00085 (17)	0.00259 (17)	0.00266 (18)
As3	0.0083 (2)	0.0091 (2)	0.0182 (3)	−0.00207 (18)	0.00470 (19)	−0.00203 (19)
As4	0.0057 (2)	0.0116 (2)	0.0105 (2)	−0.00142 (17)	0.00102 (17)	−0.00197 (18)
Na1	0.0537 (17)	0.0409 (15)	0.0194 (12)	0.0219 (13)	−0.0001 (11)	−0.0080 (11)
Na2	0.0316 (14)	0.0373 (15)	0.0341 (14)	0.0110 (11)	0.0024 (11)	0.0004 (11)
O1	0.0126 (17)	0.0139 (17)	0.0115 (17)	−0.0014 (14)	−0.0039 (13)	−0.0012 (13)
O2	0.0116 (17)	0.0240 (19)	0.0091 (17)	0.0022 (14)	0.0064 (13)	0.0021 (14)
O3	0.0155 (17)	0.0130 (17)	0.0179 (18)	−0.0009 (14)	0.0091 (14)	−0.0030 (14)
O4	0.017 (2)	0.023 (2)	0.048 (3)	0.0010 (17)	0.0076 (19)	0.0048 (19)
O5	0.0149 (18)	0.0234 (19)	0.0107 (17)	−0.0006 (15)	0.0011 (14)	0.0043 (14)
O6	0.023 (2)	0.0188 (19)	0.0137 (18)	−0.0089 (15)	−0.0053 (15)	−0.0032 (15)
O7	0.028 (2)	0.027 (2)	0.029 (2)	0.0067 (18)	0.0126 (18)	0.0129 (18)
O8	0.0198 (19)	0.021 (2)	0.025 (2)	−0.0005 (15)	−0.0092 (16)	−0.0054 (16)
O9	0.0109 (16)	0.0136 (17)	0.0163 (18)	0.0005 (13)	0.0003 (14)	0.0004 (14)
O10	0.0095 (16)	0.0187 (18)	0.0203 (19)	0.0036 (14)	0.0024 (14)	0.0041 (15)
O11	0.0084 (16)	0.0156 (18)	0.0135 (17)	0.0012 (13)	0.0012 (13)	−0.0039 (14)
O12	0.029 (2)	0.068 (4)	0.029 (3)	0.002 (2)	−0.001 (2)	0.003 (2)
O13	0.0128 (18)	0.047 (3)	0.017 (2)	0.0070 (18)	−0.0025 (15)	−0.0025 (18)
O14	0.0055 (16)	0.0152 (18)	0.025 (2)	−0.0020 (13)	0.0052 (14)	0.0046 (15)
O15	0.0131 (18)	0.032 (2)	0.023 (2)	0.0060 (16)	0.0096 (15)	0.0064 (17)
O16	0.0087 (16)	0.0129 (17)	0.0159 (17)	−0.0016 (13)	0.0049 (13)	−0.0016 (13)
O17	0.0126 (17)	0.0088 (16)	0.0218 (19)	0.0015 (13)	−0.0041 (14)	0.0032 (14)
O18	0.015 (2)	0.024 (2)	0.069 (3)	0.0017 (17)	−0.004 (2)	−0.030 (2)
O19	0.012 (2)	0.030 (6)	0.017 (2)	0.006 (5)	0.0075 (17)	0.005 (5)
O191	0.012 (2)	0.030 (6)	0.017 (2)	0.006 (5)	0.0075 (17)	0.005 (5)
O20	0.013 (2)	0.019 (3)	0.021 (4)	−0.0069 (19)	−0.009 (3)	0.009 (3)
O201	0.013 (2)	0.019 (3)	0.021 (4)	−0.0069 (19)	−0.009 (3)	0.009 (3)

Geometric parameters (Å, º)

Mo1—O13^{i}	1.684 (4)	As2—O9	1.663 (3)
Mo1—O7^{i}	1.700 (4)	As2—O17	1.675 (3)
Mo1—O3^{ii}	2.002 (3)	As2—O16^{ii}	1.753 (3)
Mo1—O2^{iii}	2.003 (3)	As3—O19^{i}	1.631 (10)
Mo1—O9^{ii}	2.189 (3)	As3—O18	1.669 (4)
Mo1—O11	2.264 (3)	As3—O20	1.706 (6)

Mo2—O4	1.672 (4)	As3—O14vi	1.735 (3)
Mo2—O12	1.688 (5)	As4—O10	1.651 (3)
Mo2—O20	2.002 (6)	As4—O6	1.666 (3)
Mo2—O18iv	2.038 (4)	As4—O3	1.686 (3)
Mo2—O19v	2.204 (10)	As4—O14	1.747 (3)
Mo2—O10vi	2.246 (3)	Na1—O7ix	2.438 (5)
Mo3—O15	1.686 (4)	Na1—O11	2.442 (4)
Mo3—O8	1.699 (4)	Na1—O1x	2.592 (4)
Mo3—O6vii	1.982 (3)	Na1—O5vii	2.652 (4)
Mo3—O17	2.003 (3)	Na2—O8xi	2.379 (5)
Mo3—O1	2.168 (3)	Na2—O13xii	2.412 (5)
Mo3—O5vii	2.275 (4)	Na2—O20xiii	2.603 (8)
As1—O11	1.663 (3)	Na2—O15xiv	2.636 (5)
As1—O1	1.664 (3)	Na2—O18xiii	2.651 (6)
As1—O2	1.673 (3)	Na2—O10xi	2.695 (4)
As1—O16viii	1.755 (3)	Na2—O12xv	2.695 (5)
As2—O5	1.657 (3)		
O13i—Mo1—O7i	103.8 (2)	O11—As1—O16viii	105.99 (16)
O13i—Mo1—O3ii	96.13 (17)	O1—As1—O16viii	103.40 (16)
O7i—Mo1—O3ii	96.25 (17)	O2—As1—O16viii	105.00 (17)
O13i—Mo1—O2iii	95.99 (17)	O5—As2—O9	115.52 (17)
O7i—Mo1—O2iii	99.49 (17)	O5—As2—O17	114.38 (18)
O3ii—Mo1—O2iii	157.22 (14)	O9—As2—O17	111.62 (17)
O13i—Mo1—O9ii	89.84 (17)	O5—As2—O16ii	103.55 (17)
O7i—Mo1—O9ii	166.34 (17)	O9—As2—O16ii	111.25 (16)
O3ii—Mo1—O9ii	81.23 (13)	O17—As2—O16ii	98.86 (16)
O2iii—Mo1—O9ii	79.59 (13)	O19i—As3—O18	120.1 (5)
O13i—Mo1—O11	167.66 (18)	O19i—As3—O20	112.8 (5)
O7i—Mo1—O11	88.54 (17)	O18—As3—O20	97.5 (3)
O3ii—Mo1—O11	82.75 (13)	O19i—As3—O14vi	109.1 (4)
O2iii—Mo1—O11	81.30 (13)	O18—As3—O14vi	107.80 (18)
O9ii—Mo1—O11	77.84 (12)	O20—As3—O14vi	108.7 (2)
O4—Mo2—O12	103.3 (2)	O10—As4—O6	119.85 (18)
O4—Mo2—O20	96.0 (2)	O10—As4—O3	118.10 (18)
O12—Mo2—O20	93.4 (3)	O6—As4—O3	103.65 (18)
O4—Mo2—O18iv	96.68 (19)	O10—As4—O14	110.01 (17)
O12—Mo2—O18iv	91.7 (2)	O6—As4—O14	101.34 (18)
O20—Mo2—O18iv	164.8 (2)	O3—As4—O14	101.14 (16)
O4—Mo2—O19v	96.4 (3)	O7ix—Na1—O11	124.45 (17)
O12—Mo2—O19v	160.3 (3)	O7ix—Na1—O1x	115.09 (16)
O20—Mo2—O19v	84.9 (4)	O11—Na1—O1x	113.72 (15)
O18iv—Mo2—O19v	85.5 (3)	O7ix—Na1—O5vii	110.59 (16)
O4—Mo2—O10vi	170.19 (18)	O11—Na1—O5vii	98.91 (14)
O12—Mo2—O10vi	86.26 (19)	O1x—Na1—O5vii	84.34 (13)
O20—Mo2—O10vi	81.20 (19)	O8xi—Na2—O13xii	105.79 (16)
O18iv—Mo2—O10vi	84.89 (15)	O8xi—Na2—O20xiii	137.2 (2)
O19v—Mo2—O10vi	74.1 (3)	O13xii—Na2—O20xiii	78.22 (17)
O15—Mo3—O8	102.3 (2)	O8xi—Na2—O15xiv	77.62 (15)

O15—Mo3—O6[vii]	97.89 (17)	O13[xii]—Na2—O15[xiv]	67.62 (15)
O8—Mo3—O6[vii]	98.61 (17)	O20[xiii]—Na2—O15[xiv]	64.43 (18)
O15—Mo3—O17	93.01 (17)	O8[xi]—Na2—O18[xiii]	91.96 (15)
O8—Mo3—O17	101.38 (16)	O13[xii]—Na2—O18[xiii]	128.51 (16)
O6[vii]—Mo3—O17	154.59 (15)	O20[xiii]—Na2—O18[xiii]	57.74 (16)
O15—Mo3—O1	98.02 (17)	O15[xiv]—Na2—O18[xiii]	69.91 (14)
O8—Mo3—O1	159.50 (17)	O8[xi]—Na2—O10[xi]	104.17 (15)
O6[vii]—Mo3—O1	76.15 (14)	O13[xii]—Na2—O10[xi]	78.55 (14)
O17—Mo3—O1	79.65 (13)	O20[xiii]—Na2—O10[xi]	118.17 (18)
O15—Mo3—O5[vii]	169.94 (16)	O15[xiv]—Na2—O10[xi]	144.98 (16)
O8—Mo3—O5[vii]	85.81 (16)	O18[xiii]—Na2—O10[xi]	143.58 (16)
O6[vii]—Mo3—O5[vii]	86.56 (15)	O8[xi]—Na2—O12[xv]	128.48 (19)
O17—Mo3—O5[vii]	79.47 (14)	O13[xii]—Na2—O12[xv]	116.79 (18)
O1—Mo3—O5[vii]	74.19 (13)	O20[xiii]—Na2—O12[xv]	81.29 (19)
O11—As1—O1	114.32 (17)	O15[xiv]—Na2—O12[xv]	144.23 (17)
O11—As1—O2	114.23 (17)	O18[xiii]—Na2—O12[xv]	83.71 (16)
O1—As1—O2	112.56 (17)	O10[xi]—Na2—O12[xv]	60.61 (13)

Symmetry codes: (i) $x, y, z-1$; (ii) $-x+1, y-1/2, -z+1/2$; (iii) $-x+1, -y, -z$; (iv) $-x, y-1/2, -z+1/2$; (v) $-x, -y+1, -z+1$; (vi) $x-1, y, z$; (vii) $x, -y+1/2, z+1/2$; (viii) $x, y-1, z$; (ix) $x, -y+1/2, z-1/2$; (x) $-x+1, -y, -z+1$; (xi) $-x+1, y+1/2, -z+3/2$; (xii) $x, y+1, z$; (xiii) $x, -y+3/2, z+1/2$; (xiv) $-x+1, -y+1, -z+1$; (xv) $-x, y+1/2, -z+3/2$.