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Hemipotassium hemirubidium digallium(III) manganese(II) tris(phosphate) dihydrate

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Key indicators: single-crystal X-ray study; T = 295 K; mean σ(Mn–O) = 0.002 Å; disorder in solvent or counterion; R factor = 0.023; wR factor = 0.065; data-to-parameter ratio = 13.1.

The title manganese(II) substituted gallophosphate, $K_{0.5}Rb_{0.5}[Ga_2Mn(PO_4)_3(H_2O)_2]$, features a three-dimensional network built of PO_4 tetrahedra, GaO_5 trigonal bipyramids and MnO_6 octahedra. The Rb^I and K^I ions, which are disordered with respect to each other in a 1:1 ratio, occupy sites within the channels of the framework. The Rb^I/K^I and Mn^{II} atoms occupy positions of 2 symmetry, as does one of the two P atoms. The Rb^I/K^I site is surrounded by six O atoms [2.996 (2)–3.178 (4) Å] in an irregularly-shaped coordination environment. O—H···O hydrogen bonds between the water molecules and phosphate O atoms consolidate the crystal packing.

Related literature

For isotypic $NH_4[Ga_2Mn(PO_4)_3(H_2O)_2]$, see: Chippindale *et al.* (1998).

Experimental

Crystal data

$K_{0.5}Rb_{0.5}[Ga_2Mn(PO_4)_3(H_2O)_2]$
M_r = 577.61
Monoclinic, $C2/c$
a = 13.5504 (12) Å
b = 10.2965 (9) Å
c = 8.9072 (8) Å
β = 108.527 (1)°
V = 1178.34 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.31 mm^{-1}
T = 295 K
0.45 × 0.40 × 0.35 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.118, T_{max} = 0.159
6267 measured reflections
1348 independent reflections
1239 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.065
S = 1.04
1348 reflections
103 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1w—H1···O3^{i}	0.84 (3)	1.97 (2)	2.790 (3)	166 (4)
O1w—H2···O6^{ii}	0.84 (3)	2.10 (2)	2.913 (3)	165 (4)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Shaanxi Normal University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2164).

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supplementary materials

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Hemipotassium hemirubidium digallium(III) manganese(II) tris(phosphate) dihydrate

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Comment

Microporous aluminium phosphates are readily synthesized by using the hydrothermal route; studies on these compounds have led to improvements in the synthesis of the related gallophosphates. The structure of $NH_4[Ga_2Mn(PO_4)_3(H_2O)_2]$ features PO_4 tetrahedra, GaO_5 trigonal bipyramids and MnO_6 octahedra that are linked together to form a three-dimensional network (Chippindale *et al.*, 1998). The title compound has a similar structure (Fig. 1); however, the rubidium and potassium atoms that occupy the channels within the network rattle in the cavities, as noted from the irregular nature of the polyhedron surrounding the atoms. The coordination number is much higher when longer interactions are considered.

Experimental

The compound was synthesized from a mixture of gallium oxide (0.037 g), boric acid (0.035 g), rubidium carbonate (0.023 g), potassium carbonate (0.138 g), manganese dichloride tetrahydrate (0.397 g), phosphoric acid (0.15 ml) and water (1.8 ml) (molar ratio of 2:5:1:10:20:20:1000). This mixture was sealed in 25 ml, Teflon-lined, stainless-steel Parr bomb. The bomb was heated at 468 K for 7 days. Colorless block-shaped crystals were isolated.

Refinement

The water H-atoms were located in a difference Fourier map, and were refined with a distance restraint of O–H 0.84±0.01 Å; their temperature factors were tied to those of the O atom by a factor of 1.5 times.

The potassium and rubidium atoms share the same site, a special position of *2* site symmetry. As the occupancy of each refined to nearly 1/2, the occupancies were then fixed as exactly 1/2. The temperature factors of K1 and Rb1 were restrained to be identical.

Figures

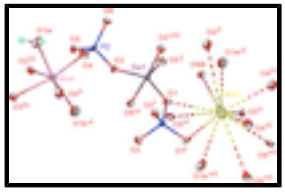


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of a portion of the polymeric structure of $K_{0.5}Rb_{0.5}[Ga_2Mn(PO_4)_3(H_2O)_2]$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The potassium atoms are disordered with respect to the rubidium atoms in a 1:1 ratio. Symmetry codes: (i) *x* + 1/2, -*y* + 3/2, *z* + 1/2; (ii) -*x* + 1/2, -*y* + 3/2, -*z* + 1; (iii) -*x* + 1, *y*, -*z* + 3/2; (iv) -*x* + 1/2, *y* + 1/2, -*z* + 1/2; (v) *x* + 1/2, *y* + 1/2, *z* + 1; (vi) *x*, -*y* + 1, *z* + 1/2; (vii) -*x* + 1, -*y* + 1, -*z* + 1; (viii) *x*, -*y* + 1, *z* - 1/2; (ix) -*x*, *y*, -*z* + 1/2; (*x*) *x* - 1/2, -*y* + 1/2, *z* - 1/2.

Hemipotassium hemirubidium digallium(III) manganese(II) tris(phosphate) dihydrate

Crystal data

$K_{0.5}Rb_{0.5}[Ga_2Mn(PO_4)_3(H_2O)_2]$ $F(000) = 1104$

M_r = 577.61	D_x = 3.256 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 4099 reflections
a = 13.5504 (12) Å	θ = 2.5–28.6°
b = 10.2965 (9) Å	μ = 8.31 mm^{-1}
c = 8.9072 (8) Å	*T* = 295 K
β = 108.527 (1)°	Block, colorless
V = 1178.34 (18) Å^3	0.45 × 0.40 × 0.35 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	1348 independent reflections
Radiation source: fine-focus sealed tube	1239 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.037
ω scans	θ_{max} = 27.5°, θ_{min} = 2.5°
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	*h* = −17→17
T_{min} = 0.118, T_{max} = 0.159	*k* = −13→13
6267 measured reflections	*l* = −11→11

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.023	Hydrogen site location: difference Fourier map
$wR(F^2)$ = 0.065	H-atom parameters constrained
S = 1.04	$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 2.8102P]$ where $P = (F_o^2 + 2F_c^2)/3$
1348 reflections	$(\Delta/\sigma)_{max}$ = 0.001
103 parameters	$\Delta\rho_{max}$ = 0.63 e Å^{-3}
2 restraints	$\Delta\rho_{min}$ = −0.75 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Rb1	0.5000	0.86132 (6)	0.7500	0.03306 (18)	0.50
Ga1	0.32950 (2)	0.57498 (3)	0.42741 (3)	0.01061 (11)	
Mn1	0.0000	0.28198 (6)	0.2500	0.01443 (15)	
K1	0.5000	0.86132 (6)	0.7500	0.03306 (18)	0.50
P1	0.5000	0.49997 (9)	0.7500	0.0106 (2)	
P2	0.21012 (6)	0.37336 (7)	0.17422 (8)	0.01171 (16)	
O1	0.44106 (16)	0.59068 (19)	0.6149 (2)	0.0150 (4)	
O2	0.42851 (16)	0.40472 (19)	0.8014 (2)	0.0154 (4)	
O3	0.29213 (16)	0.41300 (18)	0.3344 (2)	0.0146 (4)	

O4	0.10038 (17)	0.3988 (2)	0.1749 (3)	0.0198 (4)
O5	0.23416 (16)	0.22845 (19)	0.1603 (2)	0.0156 (4)
O6	0.22740 (16)	0.45222 (19)	0.0373 (2)	0.0148 (4)
O1w	−0.11223 (19)	0.3043 (2)	0.0069 (3)	0.0263 (5)
H1	−0.146 (3)	0.237 (3)	−0.033 (5)	0.039*
H2	−0.154 (3)	0.366 (3)	−0.001 (5)	0.039*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Rb1	0.0418 (4)	0.0181 (3)	0.0425 (4)	0.000	0.0179 (3)	0.000
Ga1	0.01323 (18)	0.01036 (17)	0.00716 (17)	0.00043 (10)	0.00172 (12)	0.00000 (10)
Mn1	0.0166 (3)	0.0129 (3)	0.0145 (3)	0.000	0.0059 (2)	0.000
K1	0.0418 (4)	0.0181 (3)	0.0425 (4)	0.000	0.0179 (3)	0.000
P1	0.0127 (4)	0.0117 (4)	0.0067 (4)	0.000	0.0021 (3)	0.000
P2	0.0142 (4)	0.0121 (3)	0.0088 (3)	−0.0013 (3)	0.0036 (3)	−0.0006 (2)
O1	0.0181 (10)	0.0163 (10)	0.0072 (9)	−0.0007 (8)	−0.0010 (7)	0.0015 (7)
O2	0.0182 (10)	0.0134 (9)	0.0165 (10)	−0.0009 (8)	0.0081 (8)	0.0005 (8)
O3	0.0213 (11)	0.0110 (9)	0.0101 (9)	−0.0030 (8)	0.0028 (8)	−0.0027 (7)
O4	0.0176 (10)	0.0203 (10)	0.0232 (11)	0.0004 (8)	0.0088 (9)	0.0042 (9)
O5	0.0202 (10)	0.0115 (9)	0.0147 (9)	−0.0038 (8)	0.0048 (8)	−0.0038 (7)
O6	0.0196 (10)	0.0146 (9)	0.0118 (9)	0.0006 (8)	0.0072 (8)	0.0026 (7)
O1w	0.0286 (13)	0.0226 (11)	0.0204 (11)	0.0018 (10)	−0.0025 (9)	−0.0040 (9)

Geometric parameters (Å, °)

Rb1—O1	3.040 (2)	Mn1—O2^{ix}	2.264 (2)
Rb1—O1^{i}	3.040 (2)	Mn1—O2^{x}	2.264 (2)
Rb1—O4^{ii}	3.613 (2)	Mn1—O4	2.079 (2)
Rb1—O4^{iii}	2.996 (2)	Mn1—O4^{xi}	2.079 (2)
Rb1—O4^{iv}	2.996 (2)	Mn1—O1w	2.228 (2)
Rb1—O5^{v}	3.555 (2)	Mn1—O1w^{xi}	2.228 (2)
Rb1—O5^{vi}	3.555 (2)	P1—O1	1.532 (2)
Rb1—O6^{vii}	3.451 (2)	P1—O1^{i}	1.532 (2)
Rb1—O6^{ii}	3.451 (2)	P1—O2	1.546 (2)
Rb1—O4^{vii}	3.613 (2)	P1—O2^{i}	1.546 (2)
Rb1—O1w^{ii}	3.178 (3)	P2—O4	1.512 (2)
Rb1—O1w^{vii}	3.178 (3)	P2—O5	1.541 (2)
Ga1—O1	1.870 (2)	P2—O6	1.543 (2)
Ga1—O2^{viii}	2.015 (2)	P2—O3	1.558 (2)
Ga1—O3	1.860 (2)	O1w—H1	0.84 (3)
Ga1—O5^{ii}	1.850 (2)	O1w—H2	0.84 (3)
Ga1—O6^{vi}	1.952 (2)		
O4^{iv}—Rb1—O4^{iii}	68.95 (8)	O4^{iii}—Rb1—O4^{ii}	95.69 (5)
O4^{iv}—Rb1—O1	138.43 (6)	O1—Rb1—O4^{ii}	73.69 (5)

O4^{iii}—Rb1—O1	139.75 (6)	O1^{i}—Rb1—O4^{ii}	118.48 (5)
O4^{iv}—Rb1—O1^{i}	139.75 (6)	O1w^{ii}—Rb1—O4^{ii}	51.14 (5)
O4^{iii}—Rb1—O1^{i}	138.43 (6)	O1w^{vii}—Rb1—O4^{ii}	131.83 (5)
O1—Rb1—O1^{i}	47.11 (7)	O6^{vii}—Rb1—O4^{ii}	134.28 (5)
O4^{iv}—Rb1—O1w^{ii}	68.68 (6)	O6^{ii}—Rb1—O4^{ii}	40.88 (5)
O4^{iii}—Rb1—O1w^{ii}	131.90 (6)	O5^{vi}—Rb1—O4^{ii}	77.00 (5)
O1—Rb1—O1w^{ii}	70.81 (6)	O5^{v}—Rb1—O4^{ii}	106.30 (5)
O1^{i}—Rb1—O1w^{ii}	89.44 (6)	O4^{iv}—Rb1—O4^{vii}	95.69 (5)
O4^{iv}—Rb1—O1w^{vii}	131.90 (6)	O4^{iii}—Rb1—O4^{vii}	74.01 (6)
O4^{iii}—Rb1—O1w^{vii}	68.68 (6)	O1—Rb1—O4^{vii}	118.48 (5)
O1—Rb1—O1w^{vii}	89.44 (6)	O1^{i}—Rb1—O4^{vii}	73.69 (5)
O1^{i}—Rb1—O1w^{vii}	70.81 (6)	O1w^{ii}—Rb1—O4^{vii}	131.83 (5)
O1w^{ii}—Rb1—O1w^{vii}	158.73 (9)	O1w^{vii}—Rb1—O4^{vii}	51.14 (5)
O4^{iv}—Rb1—O6^{vii}	65.17 (5)	O6^{vii}—Rb1—O4^{vii}	40.88 (5)
O4^{iii}—Rb1—O6^{vii}	88.43 (6)	O6^{ii}—Rb1—O4^{vii}	134.28 (5)
O1—Rb1—O6^{vii}	127.06 (5)	O5^{vi}—Rb1—O4^{vii}	106.30 (5)
O1^{i}—Rb1—O6^{vii}	83.95 (5)	O5^{v}—Rb1—O4^{vii}	77.00 (5)
O1w^{ii}—Rb1—O6^{vii}	93.74 (5)	O4^{ii}—Rb1—O4^{vii}	167.72 (7)
O1w^{vii}—Rb1—O6^{vii}	92.00 (5)	O5^{ii}—Ga1—O3	123.61 (9)
O4^{iv}—Rb1—O6^{ii}	88.43 (6)	O5^{ii}—Ga1—O1	116.06 (9)
O4^{iii}—Rb1—O6^{ii}	65.17 (5)	O3—Ga1—O1	120.26 (9)
O1—Rb1—O6^{ii}	83.95 (5)	O5^{ii}—Ga1—O6^{vi}	91.40 (9)
O1^{i}—Rb1—O6^{ii}	127.06 (5)	O3—Ga1—O6^{vi}	87.64 (9)
O1w^{ii}—Rb1—O6^{ii}	92.00 (5)	O1—Ga1—O6^{vi}	93.74 (9)
O1w^{vii}—Rb1—O6^{ii}	93.74 (5)	O5^{ii}—Ga1—O2^{viii}	88.85 (8)
O6^{vii}—Rb1—O6^{ii}	148.53 (7)	O3—Ga1—O2^{viii}	88.85 (9)
O4^{iv}—Rb1—O5^{vi}	131.58 (5)	O1—Ga1—O2^{viii}	89.78 (9)
O4^{iii}—Rb1—O5^{vi}	76.41 (5)	O6^{vi}—Ga1—O2^{viii}	175.95 (8)
O1—Rb1—O5^{vi}	63.43 (5)	O4—Mn1—O4^{xi}	109.28 (12)
O1^{i}—Rb1—O5^{vi}	88.32 (5)	O4—Mn1—O1w	86.67 (9)
O1w^{ii}—Rb1—O5^{vi}	118.20 (5)	O4^{xi}—Mn1—O1w	86.48 (9)
O1w^{vii}—Rb1—O5^{vi}	55.36 (5)	O4—Mn1—O1w^{xi}	86.48 (9)
O6^{vii}—Rb1—O5^{vi}	147.08 (4)	O4^{xi}—Mn1—O1w^{xi}	86.67 (9)
O6^{ii}—Rb1—O5^{vi}	45.70 (4)	O1w—Mn1—O1w^{xi}	168.14 (13)
O4^{iv}—Rb1—O5^{v}	76.41 (5)	O4—Mn1—O2^{x}	157.23 (8)
O4^{iii}—Rb1—O5^{v}	131.58 (6)	O4^{xi}—Mn1—O2^{x}	93.49 (8)
O1—Rb1—O5^{v}	88.32 (5)	O1w—Mn1—O2^{x}	94.61 (8)
O1^{i}—Rb1—O5^{v}	63.43 (5)	O1w^{xi}—Mn1—O2^{x}	95.46 (8)
O1w^{ii}—Rb1—O5^{v}	55.36 (5)	O4—Mn1—O2^{ix}	93.49 (8)
O1w^{vii}—Rb1—O5^{v}	118.20 (5)	O4^{xi}—Mn1—O2^{ix}	157.23 (8)
O6^{vii}—Rb1—O5^{v}	45.70 (4)	O1w—Mn1—O2^{ix}	95.46 (8)

O6^{ii}—Rb1—O5^{v}	147.08 (4)	O1w^{xi}—Mn1—O2^{ix}	94.61 (8)
O5^{vi}—Rb1—O5^{v}	149.86 (6)	O2^{x}—Mn1—O2^{ix}	63.75 (10)
O4^{iv}—Rb1—O4^{ii}	74.01 (6)		

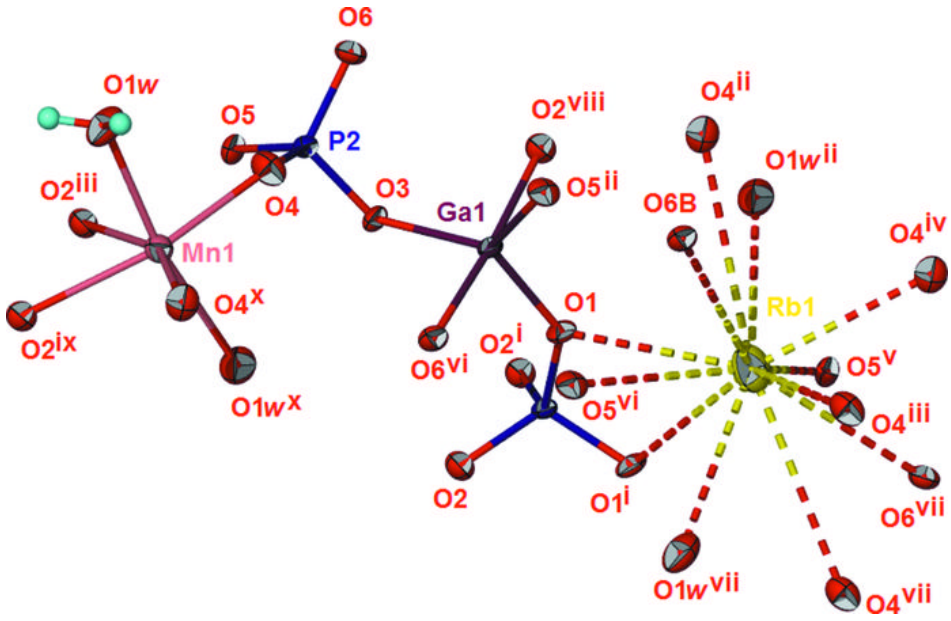
Symmetry codes: (i) −*x*+1, *y*, −*z*+3/2; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2; (iii) −*x*+1/2, −*y*+3/2, −*z*+1; (iv) *x*+1/2, −*y*+3/2, *z*+1/2; (v) −*x*+1, −*y*+1, −*z*+1; (vi) *x*, −*y*+1, *z*+1/2; (vii) *x*+1/2, *y*+1/2, *z*+1; (viii) *x*, −*y*+1, *z*−1/2; (ix) −*x*+1/2, −*y*+1/2, −*z*+1; (x) *x*−1/2,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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1w—H1···O3^{x}	0.84 (3)	1.97 (2)	2.790 (3)	166 (4)
O1w—H2···O6^{xii}	0.84 (3)	2.10 (2)	2.913 (3)	165 (4)

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

Fig. 1


O6
O1w
O5
P2
O2iii
O4
O3
Ga1
O2viii
O5ii
O4ii
O1wii
O6B
O4iv
Mn1
O4x
O1
Rb1
O2ix
O6vi
O2i
O5v
O5vi
O4iii
O2
O1i
O6vii
O1wvii
O4vii
O1wx