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Crystal structure of rubidium peroxide ammonia disolvate

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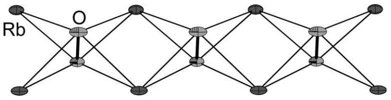
The title compound, $Rb_2O_2·2NH_3$, has been obtained as a reaction product of rubidium metal dissolved in liquid ammonia and glucuronic acid. As a result of the low-temperature crystallization, a disolvate was formed. To our knowledge, only one other solvate of an alkali metal peroxide is known: $Na_2O_2·8H_2O$ has been reported by Grehl *et al.* [*Acta Cryst.* (1995), C**51**, 1038–1040]. We determined the peroxide bond length to be 1.530 (11) Å, which is in accordance with the length reported by Bremm & Jansen [*Z. Anorg. Allg. Chem.* (1992), **610**, 64–66]. One of the ammonia solvate molecules is disordered relative to a mirror plane, with 0.5 occupancy for the corresponding nitrogen atom.

1. Chemical context

The crystal structure of the title compound was determined in the course of investigations regarding the reactivity of carbohydrates towards alkali metals and NH_3 in solutions where liquid ammonia itself is used as solvent. The source of the peroxide anion could not be explicitly traced back but it seems to have its origin in oxygen gas from intruding atmosphere due to undetected leakage in the reaction vessel.

2. Structural commentary

The asymmetric unit contains one peroxide anion, two charge-compensating rubidium cations and two ammonia molecules (Fig. 1). Except for one nitrogen atom (N1, showing half-occupancy) and one hydrogen atom (H2*B*), all other atoms are located on mirror planes. The anion is surrounded by four rubidium cations located around the girth of the peroxide ion (Fig. 2). This unit forms one-dimensional infinite strands by sharing one common edge of a distorted plane of four Rb ions



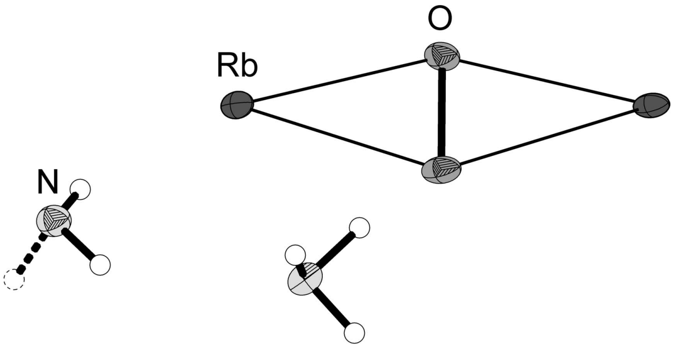


Figure 1
The asymmetric unit of the title compound, with the atom labeling and displacement ellipsoids drawn at the 50% probability level.

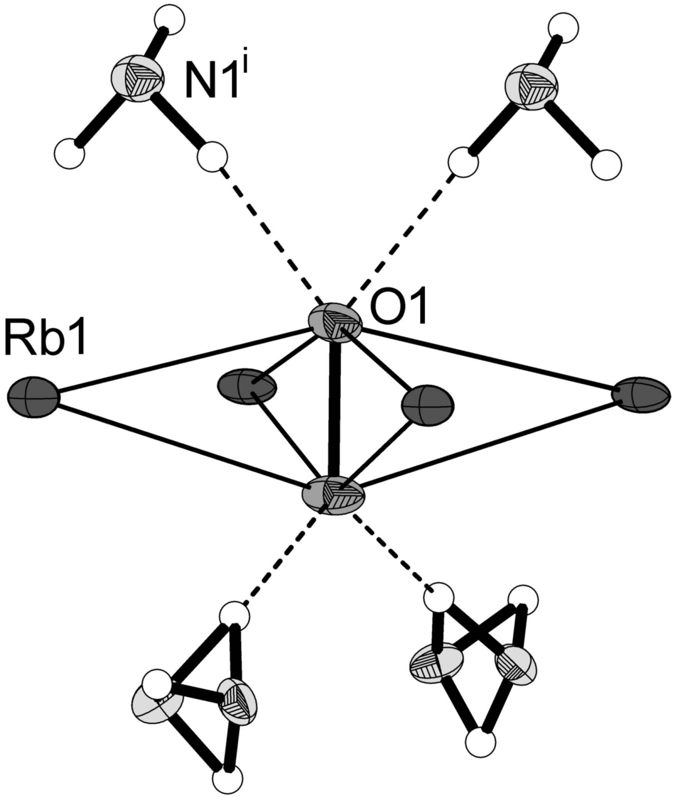


Figure 2
The environment of the peroxide anion. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $-1 + x, y, z$.]

(Fig. 3). This structural motif can also be observed in potassium acetylide K_2C_2 (Hamberger *et al.*, 2012). The peroxide bond length was determined to be 1.530 (11) Å. The anion–cation contacts range between 2.790 (5) Å and 2.917 (6) Å. The coordination number of the cations is 8.

The O—O bond length of the peroxide anion is longer than the value found in the literature based on the work of Föppl which is approximately 1.49 Å. In Fig. 4, a comparative view of bond lengths is presented based on the work of Bremm & Jansen (1992), Föppl (1954, 1955, 1957) and Grehl *et al.* (1995).

3. Supramolecular features

Despite the low ammonia content, numerous hydrogen bonds can be observed and the NH_3 molecules bridge the peroxide anions. The peroxide anion shows five contacts to ammonia molecules, forming a three-dimensional network in the packing. The distances between donor and acceptor atoms ranges from 2.926(15) Angstrom to 3.597(16) Angstrom, which is commonly observed in ammoniates. Numerical details of the hydrogen-bonding interactions are given in Table 1.

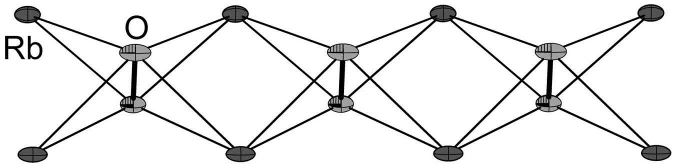


Figure 3
One-dimensional infinite strands formed by peroxide anions and rubidium cations. Displacement ellipsoids are drawn at the 50% probability level.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$A\cdots$O2	1.05 (14)	1.98 (15)	2.941 (16)	151 (8)
N1—H1$B\cdots$O2^{i}	0.97 (13)	2.04 (15)	2.926 (15)	151 (8)
N1—H1$C\cdots$O1^{ii}	0.82 (14)	3.07 (16)	3.597 (16)	125 (11)
N2—H2$A\cdots$N2^{iii}	0.74 (16)	3.03 (12)	3.57 (2)	131 (6)
N2—H2$A\cdots$N2^{iv}	0.74 (16)	3.03 (12)	3.57 (2)	131 (6)
N2—H2$A\cdots$N2^{v}	0.74 (16)	3.03 (12)	3.57 (2)	131 (6)
N2—H2$B\cdots$O1^{ii}	1.01 (11)	1.95 (11)	2.955 (10)	173 (8)

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

4. Synthesis and crystallization

500 mg (2.58 mmol) D-glucuronic acid and 880 mg (10.29 mmol) rubidium were placed under an argon atmo-

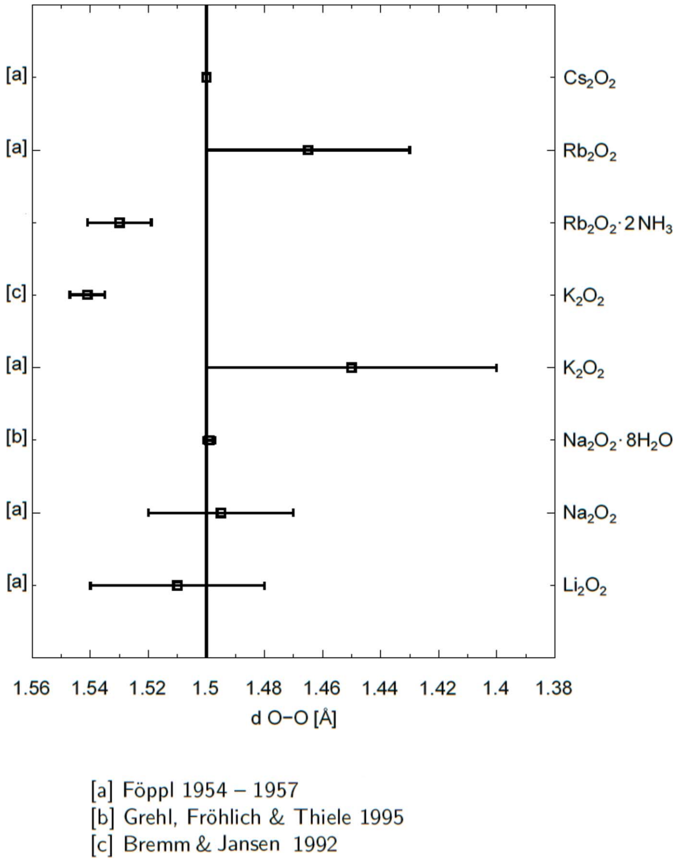


Figure 4
Comparison of peroxide bond lengths in different compounds. The vertical line shows the peroxide bond length commonly used in the literature. Each data point is shown with its standard uncertainties.

Table 2
Experimental details.

Crystal data	
Chemical formula	$Rb_2O_2\cdot 2NH_3$
M_r	237.01
Crystal system, space group	Orthorhombic, *Pnma*
Temperature (K)	123
a, b, c (Å)	7.3957 (7), 4.0932 (6), 18.1873 (17)
V (Å^3)	550.57 (11)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	17.66
Crystal size (mm)	0.24 × 0.09 × 0.08
Data collection	
Diffractometer	Agilent SuperNova Dual Source diffractometer with an Eos detector
Absorption correction	Analytical [*CrysAlis PRO* (Agilent, 2012), based on expressions derived by Clark & Reid (1995)]
T_{min}, T_{max}	0.064, 0.354
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2921, 641, 570
R_{int}	0.057
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.118, 1.35
No. of reflections	641
No. of parameters	51
H-atom treatment	Only H-atom coordinates refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.12, −1.79

Computer programs: *CrysAlis PRO* (Agilent, 2012), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg & Putz, 2012) and *OLEX2* (Dolomanov *et al.*, 2009).

sphere in a reaction vessel and 25 ml of dry liquid ammonia were condensed. The mixture was stored at 237 K for five days. The flask was then stored at 161 K for several months. After that period, clear needle-shaped colorless crystals of the title compound could be found at the wall of the flask.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The nitrogen atom N1 is disordered with 0.5 as the site occupation factor. All hydrogen atoms could be located in difference map and and their positions were refined freely with a common U_{iso}(H) parameter. The isotropic displacement parameters were fixed to 0.025.

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Acta Cryst. (2017). E**73**, 200-202 [https://doi.org/10.1107/S2056989017000354]

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); program(s) used to solve structure: *olex2.solve* (Bourhis *et al.*, 2015); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Rubidium peroxide ammonia disolvate

Crystal data

$Rb_2O_2 \cdot 2NH_3$
$M_r = 237.01$
Orthorhombic, *Pnma*
$a = 7.3957\ (7)$ Å
$b = 4.0932\ (6)$ Å
$c = 18.1873\ (17)$ Å
$V = 550.57\ (11)$ Å^3
$Z = 4$
$F(000) = 440$

$D_x = 2.859$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1711 reflections
$\theta = 3.4–28.3°$
$\mu = 17.66$ mm^{-1}
$T = 123$ K
Block, clear colourless
0.24 × 0.09 × 0.08 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Eos detector
Mirror monochromator
Detector resolution: 7.9851 pixels mm^{-1}
phi and ω scans
Absorption correction: analytical [CrysAlis PRO (Agilent, 2012), based on expressions derived by Clark & Reid (1995)]
$T_{min} = 0.064$, $T_{max} = 0.354$

2921 measured reflections
641 independent reflections
570 reflections with $I > 2\sigma(I)$
$R_{int} = 0.057$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.6°$
$h = -9 \rightarrow 9$
$k = -5 \rightarrow 4$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.118$
$S = 1.35$
641 reflections
51 parameters
0 restraints

Hydrogen site location: difference Fourier map
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2 + 7.6682P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.12$ e Å^{-3}
$\Delta\rho_{min} = -1.79$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$	Occ. (<1)
Rb1	0.11196 (14)	0.2500	0.42075 (5)	0.0164 (4)	
Rb2	−0.25614 (15)	1.2500	0.28803 (5)	0.0182 (4)	
O1	−0.1266 (11)	0.7500	0.3825 (4)	0.019 (2)	
O2	0.0133 (11)	0.7500	0.3206 (4)	0.022 (2)	
N2	0.6843 (16)	0.2500	0.4710 (6)	0.020 (3)	
N1	0.4060 (18)	0.650 (4)	0.3327 (7)	0.017 (4)	0.5
H1A	0.28 (2)	0.7500	0.340 (8)	0.025*	
H1B	0.42 (2)	0.7500	0.285 (8)	0.025*	
H1C	0.46 (2)	0.7500	0.364 (8)	0.025*	
H2A	0.58 (2)	0.2500	0.472 (8)	0.025*	
H2B	0.739 (13)	0.43 (3)	0.441 (5)	0.025*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Rb1	0.0107 (5)	0.0268 (8)	0.0116 (5)	0.000	−0.0022 (4)	0.000
Rb2	0.0088 (5)	0.0345 (8)	0.0113 (5)	0.000	0.0003 (4)	0.000
O1	0.012 (4)	0.027 (6)	0.020 (4)	0.000	0.003 (3)	0.000
O2	0.011 (4)	0.040 (7)	0.014 (4)	0.000	0.000 (3)	0.000
N2	0.017 (5)	0.025 (7)	0.018 (5)	0.000	0.000 (4)	0.000
N1	0.016 (6)	0.019 (12)	0.016 (6)	−0.007 (6)	−0.002 (5)	0.000 (6)

Geometric parameters (Å, º)

Rb1—Rb2^{i}	3.6383 (15)	Rb2—N1^{x}	3.507 (15)
Rb1—O1	2.790 (5)	Rb2—N1^{xiii}	2.988 (14)
Rb1—O1^{ii}	3.579 (8)	Rb2—N1^{ix}	2.988 (14)
Rb1—O1^{i}	2.790 (6)	O1—Rb1^{ii}	3.579 (8)
Rb1—O2	2.836 (5)	O1—Rb1^{viii}	2.790 (5)
Rb1—O2^{i}	2.836 (5)	O1—Rb2^{i}	2.839 (6)
Rb1—N2^{iii}	3.292 (12)	O1—O2	1.530 (11)
Rb1—N2^{iv}	3.215 (8)	O2—Rb1^{viii}	2.836 (5)
Rb1—N2^{v}	3.215 (8)	O2—Rb2^{xiv}	3.316 (6)
Rb1—N2^{vi}	3.215 (8)	O2—Rb2^{xv}	3.316 (6)
Rb1—N1^{vii}	3.157 (14)	O2—Rb2^{i}	2.917 (6)
Rb1—N1	3.157 (14)	N2—Rb1^{iv}	3.215 (8)
Rb2—O1	2.839 (6)	N2—Rb1^{xvi}	3.292 (12)
Rb2—O1^{viii}	2.839 (6)	N2—Rb1^{xvii}	3.215 (8)
Rb2—O2^{viii}	2.917 (6)	N2—Rb2^{xviii}	3.356 (11)

Rb2—O2^{ix}	3.316 (6)	N1—Rb1^{viii}	3.652 (15)
Rb2—O2	2.917 (6)	N1—Rb2^{xiv}	2.988 (14)
Rb2—O2^{x}	3.316 (6)	N1—Rb2^{xv}	3.507 (15)
Rb2—N2^{xi}	3.356 (11)	N1—Rb2^{xvi}	3.598 (14)
Rb2—N1^{xi}	3.095 (15)	N1—Rb2^{xviii}	3.095 (15)
Rb2—N1^{xii}	3.095 (15)	N1—N1^{xix}	0.82 (3)

O1^{ii}—Rb1—Rb2^{i}	133.30 (13)	O2—Rb2—N1^{x}	53.2 (3)
O1^{i}—Rb1—Rb2^{i}	50.32 (13)	O2—Rb2—N1^{xi}	150.4 (3)
O1—Rb1—Rb2^{i}	50.32 (13)	O2—Rb2—N1^{xii}	97.3 (3)
O1^{i}—Rb1—O1^{ii}	105.56 (17)	O2^{ix}—Rb2—N1^{x}	103.6 (3)
O1—Rb1—O1^{i}	94.4 (2)	O2^{viii}—Rb2—N1^{ix}	59.4 (3)
O1—Rb1—O1^{ii}	105.56 (17)	O2^{viii}—Rb2—N1^{x}	112.7 (3)
O1^{i}—Rb1—O2	101.92 (19)	O2^{viii}—Rb2—N1^{xi}	97.3 (3)
O1^{i}—Rb1—O2^{i}	31.5 (2)	O2^{x}—Rb2—N1^{x}	51.0 (3)
O1—Rb1—O2	31.5 (2)	O2—Rb2—N1^{xiii}	59.4 (3)
O1—Rb1—O2^{i}	101.92 (19)	O2^{viii}—Rb2—N1^{xiii}	105.0 (3)
O1^{i}—Rb1—N2^{v}	156.3 (3)	O2—Rb2—N1^{ix}	105.0 (3)
O1^{i}—Rb1—N2^{iii}	57.42 (17)	N2^{xi}—Rb2—N1^{x}	131.7 (3)
O1^{i}—Rb1—N2^{iv}	156.3 (3)	N1^{xi}—Rb2—O2^{x}	93.8 (3)
O1—Rb1—N2^{v}	89.0 (2)	N1^{xi}—Rb2—O2^{ix}	54.2 (3)
O1—Rb1—N2^{iv}	89.0 (2)	N1^{ix}—Rb2—O2^{ix}	55.3 (3)
O1—Rb1—N2^{vi}	156.3 (3)	N1^{xii}—Rb2—O2^{ix}	93.8 (3)
O1—Rb1—N2^{iii}	57.42 (17)	N1^{xiii}—Rb2—O2^{x}	55.3 (3)
O1^{i}—Rb1—N2^{vi}	88.98 (19)	N1^{xii}—Rb2—O2^{x}	54.2 (3)
O1—Rb1—N1	85.9 (3)	N1^{xiii}—Rb2—O2^{ix}	96.1 (3)
O1^{i}—Rb1—N1^{vii}	85.9 (3)	N1^{ix}—Rb2—O2^{x}	96.1 (3)
O1^{i}—Rb1—N1	133.5 (3)	N1^{xii}—Rb2—N2^{xi}	68.5 (3)
O1—Rb1—N1^{vii}	133.5 (3)	N1^{xiii}—Rb2—N2^{xi}	141.3 (3)
O2^{i}—Rb1—Rb2^{i}	51.77 (13)	N1^{xi}—Rb2—N2^{xi}	68.5 (3)
O2—Rb1—Rb2^{i}	51.77 (13)	N1^{ix}—Rb2—N2^{xi}	141.3 (3)
O2—Rb1—O1^{ii}	130.55 (13)	N1^{xii}—Rb2—N1^{xi}	63.8 (6)
O2^{i}—Rb1—O1^{ii}	130.55 (13)	N1^{ix}—Rb2—N1^{xi}	103.1 (5)
O2—Rb1—O2^{i}	92.4 (2)	N1^{ix}—Rb2—N1^{xii}	143.7 (3)
O2—Rb1—N2^{v}	93.14 (17)	N1^{xiii}—Rb2—N1^{x}	11.3 (4)
O2^{i}—Rb1—N2^{iv}	166.8 (2)	N1^{xiii}—Rb2—N1^{ix}	66.4 (6)
O2^{i}—Rb1—N2^{vi}	93.14 (17)	N1^{ix}—Rb2—N1^{x}	77.7 (3)
O2—Rb1—N2^{iii}	86.0 (2)	N1^{xiii}—Rb2—N1^{xii}	103.1 (5)
O2—Rb1—N2^{iv}	93.14 (17)	N1^{xii}—Rb2—N1^{x}	94.0 (2)
O2—Rb1—N2^{vi}	166.8 (2)	N1^{xiii}—Rb2—N1^{xi}	143.7 (3)
O2^{i}—Rb1—N2^{v}	166.8 (2)	N1^{xi}—Rb2—N1^{x}	144.2 (4)
O2^{i}—Rb1—N2^{iii}	86.0 (2)	Rb1^{viii}—O1—Rb1^{ii}	74.44 (17)
O2—Rb1—N1	58.5 (3)	Rb1—O1—Rb1^{viii}	94.4 (2)
O2—Rb1—N1^{vii}	103.0 (3)	Rb1—O1—Rb1^{ii}	74.44 (17)
O2^{i}—Rb1—N1	103.0 (3)	Rb1—O1—Rb2^{i}	80.53 (7)
O2^{i}—Rb1—N1^{vii}	58.5 (3)	Rb1^{viii}—O1—Rb2	80.53 (7)
N2^{iii}—Rb1—Rb2^{i}	57.66 (19)	Rb1^{viii}—O1—Rb2^{i}	153.3 (3)
N2^{vi}—Rb1—Rb2^{i}	139.21 (14)	Rb1—O1—Rb2	153.3 (3)

N2$^{\mathrm{iv}}$—Rb1—Rb2$^{\mathrm{i}}$	139.21 (14)	Rb2—O1—Rb1$^{\mathrm{ii}}$	127.97 (15)
N2$^{\mathrm{v}}$—Rb1—Rb2$^{\mathrm{i}}$	139.21 (14)	Rb2$^{\mathrm{i}}$—O1—Rb1$^{\mathrm{ii}}$	127.97 (15)
N2$^{\mathrm{iv}}$—Rb1—O1$^{\mathrm{ii}}$	51.21 (19)	Rb2$^{\mathrm{i}}$—O1—Rb2	92.3 (2)
N2$^{\mathrm{v}}$—Rb1—O1$^{\mathrm{ii}}$	51.21 (19)	O2—O1—Rb1$^{\mathrm{ii}}$	135.7 (5)
N2$^{\mathrm{iii}}$—Rb1—O1$^{\mathrm{ii}}$	75.6 (2)	O2—O1—Rb1	75.9 (3)
N2$^{\mathrm{vi}}$—Rb1—O1$^{\mathrm{ii}}$	51.21 (19)	O2—O1—Rb1$^{\mathrm{viii}}$	75.9 (3)
N2$^{\mathrm{v}}$—Rb1—N2$^{\mathrm{iii}}$	106.3 (2)	O2—O1—Rb2	77.4 (3)
N2$^{\mathrm{vi}}$—Rb1—N2$^{\mathrm{iii}}$	106.3 (2)	O2—O1—Rb2$^{\mathrm{i}}$	77.4 (3)
N2$^{\mathrm{iv}}$—Rb1—N2$^{\mathrm{iii}}$	106.3 (2)	Rb1—O2—Rb1$^{\mathrm{viii}}$	92.4 (2)
N2$^{\mathrm{v}}$—Rb1—N2$^{\mathrm{vi}}$	79.1 (2)	Rb1$^{\mathrm{viii}}$—O2—Rb2	78.45 (8)
N2$^{\mathrm{v}}$—Rb1—N2$^{\mathrm{iv}}$	0.0 (5)	Rb1—O2—Rb2	144.3 (3)
N2$^{\mathrm{iv}}$—Rb1—N2$^{\mathrm{vi}}$	79.1 (2)	Rb1$^{\mathrm{viii}}$—O2—Rb2$^{\mathrm{xiv}}$	134.1 (3)
N1$^{\mathrm{vii}}$—Rb1—Rb2$^{\mathrm{i}}$	100.3 (2)	Rb1—O2—Rb2$^{\mathrm{xiv}}$	78.75 (10)
N1—Rb1—Rb2$^{\mathrm{i}}$	100.3 (2)	Rb1—O2—Rb2$^{\mathrm{i}}$	78.45 (8)
N1—Rb1—O1$^{\mathrm{ii}}$	119.1 (3)	Rb1—O2—Rb2$^{\mathrm{xv}}$	134.1 (3)
N1$^{\mathrm{vii}}$—Rb1—O1$^{\mathrm{ii}}$	119.1 (3)	Rb1$^{\mathrm{viii}}$—O2—Rb2$^{\mathrm{i}}$	144.3 (3)
N1—Rb1—N2$^{\mathrm{v}}$	70.0 (3)	Rb1$^{\mathrm{viii}}$—O2—Rb2$^{\mathrm{xv}}$	78.75 (10)
N1$^{\mathrm{vii}}$—Rb1—N2$^{\mathrm{vi}}$	70.0 (3)	Rb2$^{\mathrm{i}}$—O2—Rb2	89.1 (2)
N1—Rb1—N2$^{\mathrm{iv}}$	70.0 (3)	Rb2$^{\mathrm{xiv}}$—O2—Rb2$^{\mathrm{xv}}$	76.22 (17)
N1$^{\mathrm{vii}}$—Rb1—N2$^{\mathrm{iv}}$	108.5 (3)	Rb2—O2—Rb2$^{\mathrm{xv}}$	78.31 (10)
N1—Rb1—N2$^{\mathrm{vi}}$	108.5 (3)	Rb2$^{\mathrm{i}}$—O2—Rb2$^{\mathrm{xiv}}$	78.32 (10)
N1—Rb1—N2$^{\mathrm{iii}}$	143.4 (3)	Rb2—O2—Rb2$^{\mathrm{xiv}}$	131.6 (3)
N1$^{\mathrm{vii}}$—Rb1—N2$^{\mathrm{iii}}$	143.4 (3)	Rb2$^{\mathrm{i}}$—O2—Rb2$^{\mathrm{xv}}$	131.6 (3)
N1$^{\mathrm{vii}}$—Rb1—N2$^{\mathrm{v}}$	108.5 (3)	O1—O2—Rb1	72.6 (3)
N1—Rb1—N1$^{\mathrm{vii}}$	62.4 (6)	O1—O2—Rb1$^{\mathrm{viii}}$	72.6 (3)
O1—Rb2—O1$^{\mathrm{viii}}$	92.3 (2)	O1—O2—Rb2	71.8 (3)
O1$^{\mathrm{viii}}$—Rb2—O2$^{\mathrm{ix}}$	95.11 (15)	O1—O2—Rb2$^{\mathrm{xv}}$	141.86 (9)
O1—Rb2—O2$^{\mathrm{viii}}$	98.78 (18)	O1—O2—Rb2$^{\mathrm{xiv}}$	141.86 (9)
O1—Rb2—O2$^{\mathrm{x}}$	95.11 (15)	O1—O2—Rb2$^{\mathrm{i}}$	71.8 (3)
O1$^{\mathrm{viii}}$—Rb2—O2$^{\mathrm{x}}$	168.22 (18)	Rb1$^{\mathrm{iv}}$—N2—Rb1$^{\mathrm{xvi}}$	73.7 (2)
O1—Rb2—O2$^{\mathrm{ix}}$	168.22 (18)	Rb1$^{\mathrm{iv}}$—N2—Rb1$^{\mathrm{xvii}}$	79.1 (2)
O1$^{\mathrm{viii}}$—Rb2—O2$^{\mathrm{viii}}$	30.8 (2)	Rb1$^{\mathrm{xvii}}$—N2—Rb1$^{\mathrm{xvi}}$	73.7 (2)
O1$^{\mathrm{viii}}$—Rb2—O2	98.77 (18)	Rb1$^{\mathrm{xvi}}$—N2—Rb2$^{\mathrm{xviii}}$	66.4 (2)
O1—Rb2—O2	30.8 (2)	Rb1$^{\mathrm{xvii}}$—N2—Rb2$^{\mathrm{xviii}}$	123.1 (3)
O1—Rb2—N2$^{\mathrm{xi}}$	56.23 (17)	Rb1$^{\mathrm{iv}}$—N2—Rb2$^{\mathrm{xviii}}$	123.1 (3)
O1$^{\mathrm{viii}}$—Rb2—N2$^{\mathrm{xi}}$	56.23 (17)	Rb1—N1—Rb1$^{\mathrm{viii}}$	73.5 (3)
O1$^{\mathrm{viii}}$—Rb2—N1$^{\mathrm{ix}}$	85.1 (3)	Rb1—N1—Rb2$^{\mathrm{xvi}}$	161.2 (5)
O1—Rb2—N1$^{\mathrm{ix}}$	134.7 (3)	Rb1—N1—Rb2$^{\mathrm{xv}}$	116.4 (4)
O1—Rb2—N1$^{\mathrm{x}}$	76.0 (3)	Rb2$^{\mathrm{xiv}}$—N1—Rb1	79.2 (4)
O1—Rb2—N1$^{\mathrm{xi}}$	119.6 (3)	Rb2$^{\mathrm{xvi}}$—N1—Rb1$^{\mathrm{viii}}$	93.0 (4)
O1—Rb2—N1$^{\mathrm{xii}}$	74.5 (3)	Rb2$^{\mathrm{xv}}$—N1—Rb1$^{\mathrm{viii}}$	66.4 (3)
O1$^{\mathrm{viii}}$—Rb2—N1$^{\mathrm{xiii}}$	134.7 (3)	Rb2$^{\mathrm{xviii}}$—N1—Rb1	114.5 (5)
O1$^{\mathrm{viii}}$—Rb2—N1$^{\mathrm{xii}}$	119.6 (3)	Rb2$^{\mathrm{xiv}}$—N1—Rb1$^{\mathrm{viii}}$	116.9 (4)
O1$^{\mathrm{viii}}$—Rb2—N1$^{\mathrm{xi}}$	74.5 (3)	Rb2$^{\mathrm{xviii}}$—N1—Rb1$^{\mathrm{viii}}$	162.1 (5)
O1$^{\mathrm{viii}}$—Rb2—N1$^{\mathrm{x}}$	140.3 (3)	Rb2$^{\mathrm{xviii}}$—N1—Rb2$^{\mathrm{xvi}}$	75.0 (3)
O1—Rb2—N1$^{\mathrm{xiii}}$	85.1 (3)	Rb2$^{\mathrm{xviii}}$—N1—Rb2$^{\mathrm{xv}}$	118.8 (4)
O2$^{\mathrm{x}}$—Rb2—O2$^{\mathrm{ix}}$	76.22 (17)	Rb2$^{\mathrm{xv}}$—N1—Rb2$^{\mathrm{xvi}}$	67.5 (3)
O2—Rb2—O2$^{\mathrm{ix}}$	154.83 (12)	Rb2$^{\mathrm{xiv}}$—N1—Rb2$^{\mathrm{xv}}$	77.7 (3)

O2$^{\rm viii}$—Rb2—O2$^{\rm ix}$	92.24 (11)	Rb2$^{\rm xiv}$—N1—Rb2$^{\rm xvi}$	119.1 (4)
O2$^{\rm viii}$—Rb2—O2	89.1 (2)	Rb2$^{\rm xiv}$—N1—Rb2$^{\rm xviii}$	80.9 (4)
O2$^{\rm viii}$—Rb2—O2$^{\rm x}$	154.83 (12)	N1$^{\rm xix}$—N1—Rb1$^{\rm viii}$	47.7 (2)
O2—Rb2—O2$^{\rm x}$	92.24 (11)	N1$^{\rm xix}$—N1—Rb1	121.2 (3)
O2$^{\rm viii}$—Rb2—N2$^{\rm xi}$	83.6 (2)	N1$^{\rm xix}$—N1—Rb2$^{\rm xv}$	45.5 (2)
O2$^{\rm ix}$—Rb2—N2$^{\rm xi}$	121.53 (19)	N1$^{\rm xix}$—N1—Rb2$^{\rm xvi}$	46.9 (3)
O2$^{\rm x}$—Rb2—N2$^{\rm xi}$	121.53 (19)	N1$^{\rm xix}$—N1—Rb2$^{\rm xiv}$	123.2 (3)
O2—Rb2—N2$^{\rm xi}$	83.6 (2)	N1$^{\rm xix}$—N1—Rb2$^{\rm xviii}$	121.9 (3)
O2$^{\rm viii}$—Rb2—N1$^{\rm xii}$	150.4 (3)		

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*, −*y*+1, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) −*x*+1, −*y*+1, −*z*+1; (v) −*x*+1, *y*+1/2, −*z*+1; (vi) −*x*+1, *y*−1/2, −*z*+1; (vii) *x*, −*y*+1/2, *z*; (viii) *x*, *y*+1, *z*; (ix) *x*−1/2, *y*+1, −*z*+1/2; (x) *x*−1/2, *y*, −*z*+1/2; (xi) *x*−1, *y*+1, *z*; (xii) *x*−1, −*y*+3/2, *z*; (xiii) *x*−1/2, −*y*+3/2, −*z*+1/2; (xiv) *x*+1/2, *y*−1, −*z*+1/2; (xv) *x*+1/2, *y*, −*z*+1/2; (xvi) *x*+1, *y*, *z*; (xvii) −*x*+1, −*y*, −*z*+1; (xviii) *x*+1, *y*−1, *z*; (xix) *x*, −*y*+3/2, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2	1.05 (14)	1.98 (15)	2.941 (16)	151 (8)
N1—H1*B*···O2$^{\rm xx}$	0.97 (13)	2.04 (15)	2.926 (15)	151 (8)
N1—H1*C*···O1$^{\rm xvi}$	0.82 (14)	3.07 (16)	3.597 (16)	125 (11)
N2—H2*A*···N2$^{\rm iv}$	0.74 (16)	3.03 (12)	3.57 (2)	131 (6)
N2—H2*A*···N2$^{\rm vi}$	0.74 (16)	3.03 (12)	3.57 (2)	131 (6)
N2—H2*A*···N2$^{\rm v}$	0.74 (16)	3.03 (12)	3.57 (2)	131 (6)
N2—H2*B*···O1$^{\rm xvi}$	1.01 (11)	1.95 (11)	2.955 (10)	173 (8)

Symmetry codes: (iv) −*x*+1, −*y*+1, −*z*+1; (v) −*x*+1, *y*+1/2, −*z*+1; (vi) −*x*+1, *y*−1/2, −*z*+1; (xvi) *x*+1, *y*, *z*; (xx) *x*+1/2, −*y*+3/2, −*z*+1/2.