Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Ammonium and caesium carbonate peroxosolvates: supramolecular networks formed by hydrogen bonds

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Received 16 January 2012
Accepted 14 February 2012
Online 23 February 2012

Diammonium carbonate hydrogen peroxide monosolvate, $2NH_4^+\cdot CO_3^{2-}\cdot H_2O_2$, (I), and dicaesium carbonate hydrogen peroxide trisolvate, $2Cs^+\cdot CO_3^{2-}\cdot 3H_2O_2$, (II), were crystallized from 98% hydrogen peroxide. In (I), the carbonate anions and peroxide solvent molecules are arranged on twofold axes. The peroxide molecules act as donors in only two hydrogen bonds with carbonate groups, forming chains along the *a* and *c* axes. In the structure of (II), there are three independent Cs^+ ions, two of them residing on twofold axes, as are two of the four peroxide molecules, one of which is disordered. Both structures comprise complicated three-dimensional hydrogen-bonded networks.

Comment

The structures of peroxosolvates formed by simple inorganic salts have been intensively studied to establish correlations between crystal packing and stability. Significant interest has been shown in the oxalates (Pedersen, 1972*a*,*b*; Adams *et al.*, 1976, 1980*a*,*b*; Adams & Pritchard, 1976), phosphates (Adams & Ramdas, 1978; Oeckler & Montbrun, 2008) and sulfates (Adams *et al.*, 1978; Adams & Pritchard, 1978; Pritchard *et al.*, 2005) of alkali metals (Adams *et al.*, 1980*a*,*b*; Oeckler & Montbrun, 2008; Adams *et al.*, 1978; Adams & Pritchard, 1978; Pritchard *et al.*, 2005) and of ammonium (Pedersen, 1972*a*,*b*) and guanidinium (Adams *et al.*, 1976; Adams & Pritchard, 1976; Adams & Ramdas, 1978) cations. These substances were considered potential H_2O_2 carriers. Special attention has been paid to $Na_2CO_3\cdot 1.5H_2O_2$, one of the most important industrially produced bleaching and oxidation agents (Jakob *et al.*, 2005; McKillop & Sanderson, 2000). The structure of $Na_2CO_3\cdot 1.5H_2O_2$ has been determined several times under various conditions (Adams & Pritchard, 1977; Carrondo *et al.*, 1977; Pritchard & Islam, 2003). The unusual peroxosolvate potassium hydroxopercarbonate $\{K[H(O_2)CO_2]\cdot H_2O_2\}$ has been studied previously (Adam & Mehta, 1998). It should be noted that hydrogen bonding plays a predominant role in the formation of the structures of both organic and inorganic peroxosolvates.

However, in all the above-cited works, the target peroxosolvates were crystallized from dilute (30 or 50%) hydrogen peroxide. Some time ago, we introduced concentrated (96–98%) peroxide as a medium for peroxosolvate synthesis (Churakov *et al.*, 2005). This method allowed us to synthesize several peroxosolvates that were not accessible using less concentrated H_2O_2, namely $Ph_4AsCl\cdot 2H_2O_2$ (Churakov *et al.*, 2005), glycine hydrogen peroxide sesquisolvate (Churakov *et al.*, 2009), isoleucine hydrogen peroxide monosolvate and β-alanine hydrogen peroxide disolvate (Prikhodchenko *et al.*, 2011). Furthermore, the use of highly concentrated hydrogen peroxide solved the problem of partial substitution of H_2O_2 by water molecules in the crystal structures (Pedersen, 1972*b*; Churakov *et al.*, 2005, 2009; Prikhodchenko *et al.*, 2011). Herein, the structures of diammonium carbonate hydrogen peroxide monosolvate, (I), and dicaesium carbonate hydrogen peroxide trisolvate, (II), both crystallized from 98% hydrogen peroxide, are presented.

In the structure of (I), both independent carbonate anions and both independent H_2O_2 solvent molecules are arranged on twofold axes. In the peroxide molecules, the O—O distances (see Table 1) are close to those observed previously in the accurately determined structures of crystalline H_2O_2 [1.461 (3) Å; Savariault & Lehmann, 1980] and urea per-

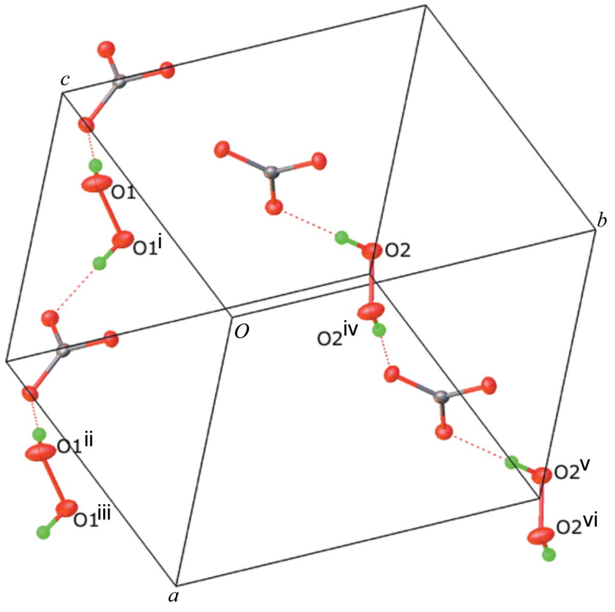

Figure 1
The hydrogen-bonded (dotted lines) chains formed by the hydrogen peroxide molecules and carbonate anions in (I). Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x + \frac{1}{2}, y, -z + \frac{3}{2}$; (ii) $x + 1, y, z$; (iii) $-x + \frac{3}{2}, y, -z + \frac{3}{2}$; (iv) $-x + \frac{1}{2}, y, -z + \frac{1}{2}$; (v) $x, y, -z + 1$; (vi) $-x + \frac{1}{2}, y, -z - \frac{1}{2}$.]

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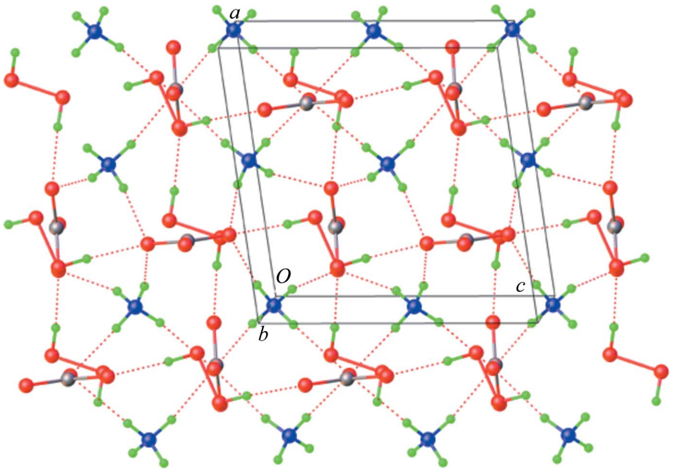

Figure 2
The crystal packing in the structure of (I), viewed along the *b* axis. Hydrogen bonds are shown as dotted lines.

hydrate [1.4573 (8) Å; Fritchie & McMullan, 1981]. Partial substitutional disorder of hydrogen peroxide by water molecules (Pedersen, 1972*b*) was not observed, as no residual peaks with intensities greater than 0.16 e Å^3 were present in the region of the hydrogen peroxide molecules (Churakov *et al.*, 2009; Prikhodchenko *et al.*, 2011). The hydrogen peroxide molecules containing atoms O1 and H1 (denoted molecule 1) and atoms O2 and H2 (denoted molecule 2) have a skew geometry (C_2 symmetry), with H—O—O—H torsion angles of −133 (2) and 137 (2)°, respectively. The carbonate anions lie on crystallographic twofold axes which pass through the C atom (C1 or C2) and one O atom (O11 or O21, respectively), and are therefore planar by symmetry. The ammonium cations are tetrahedral [H—N—H angles = 104.2 (11)–113.7 (12)°] and the N—H bond lengths range from 0.859 (15) to 0.893 (13) Å.

Both peroxide molecules 1 and 2 are involved as donors in only two almost linear hydrogen bonds with adjacent car-

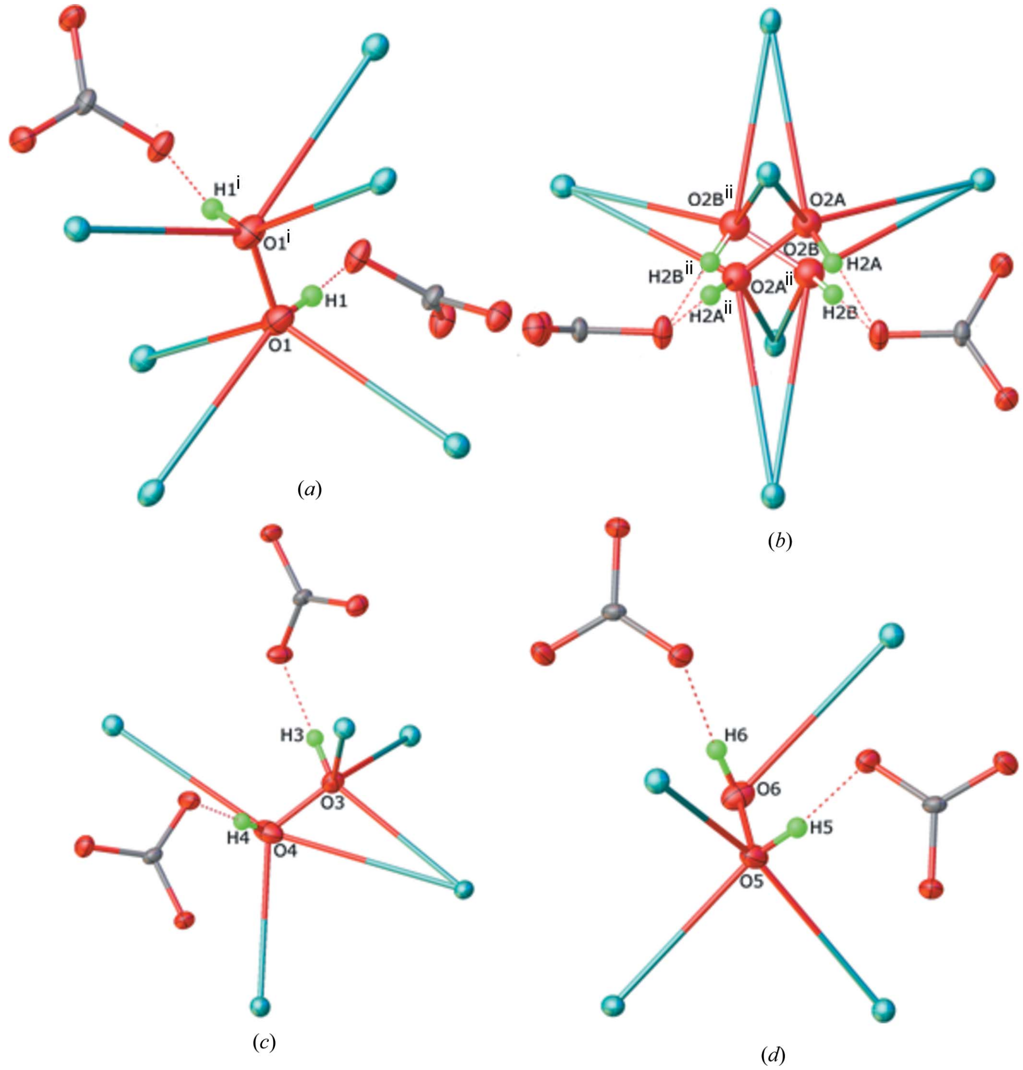

Figure 3
The coordination environment of the four independent hydrogen peroxide molecules in (II), showing (*a*) the H1—O1—O1^i—H1^i molecule, (*b*) the minor part of the disordered H2—O2—O2^{ii}—H2^{ii} site (open bonds), (*c*) the H3—O3—O4—H4 molecule and (*d*) the H5—O5—O6—H6 molecule. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x + \frac{3}{2}, -y - \frac{1}{2}, z$; (ii) $-x + \frac{3}{2}, -y + \frac{3}{2}, z$.]

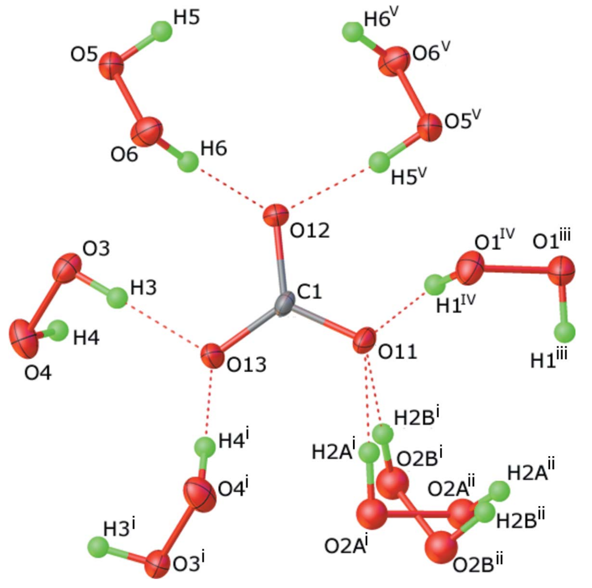

Figure 4
The hydrogen bonds (dashed lines) formed by the carbonate anions in (II). The H2—O2—O2—H2 molecule is disordered over two positions. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x - \frac{1}{2}, -y + 1, -z + \frac{1}{2}$; (iii) $x - \frac{1}{2}, y + \frac{1}{2}, -z$; (iv) $-x + 1, -y, -z$; (v) $-x + 1, -y + 1, -z$.]

bonate anions (Table 2). These hydrogen bonds are of medium strength and are somewhat longer than those found for similar hydrogen bonds in the structure of $Na_2CO_3{\cdot}1.5H_2O_2$ [2.5569 (10)–2.6022 (8) Å at 100 K; Pritchard & Islam, 2003]. Compound (I) is a rare example of a structure in which the hydrogen peroxide solvent molecules serve only as donors in hydrogen bonds (Churakov *et al.*, 2005; Thierbach *et al.*, 1980). Usually, hydrogen peroxide molecules are involved in both donor and acceptor interactions, forming six (two donors and four acceptors) (Fritchie & McMullan, 1981; Adams & Ramdas, 1979), five (two donors and three acceptors) (Adams & Ramdas, 1978; Prikhodchenko *et al.*, 2011), four (two donors and two acceptors) (Pedersen, 1972*a*; Churakov *et al.*, 2009; Prikhodchenko *et al.*, 2011) or three (two donors and one acceptor) (Mak & Lam, 1978; Prikhodchenko *et al.*, 2011) hydrogen bonds in the structures of peroxosolvates.

The two hydrogen bonds between peroxide molecules 1 and 2 and the carbonate groups form chains along the *a* and *c* axes, respectively (Fig. 1). These chains are crosslinked by ammonium cations *via* relatively weak ammonium–carbonate N—H···O hydrogen bonds, resulting in a three-dimensional network (Fig. 2). In (I), all H atoms are engaged in hydrogen bonding.

It should be noted that anhydrous $(NH_4)_2CO_3$ and its hydrates are not known. Commercially available 'ammonium carbonate' consists of a mixture of ammonium bicarbonate and ammonium carbamate. In contrast, the structure of ammonium bicarbonate is well known (Brooks & Alcock, 1950). Compound (I) may be considered as an environmentally friendly hydrogen-peroxide-containing reagent.

The crystals of (II) were obtained by cooling a saturated solution (room temperature) of caesium carbonate in 98% hydrogen peroxide to 255 K. Compound (II) also crystallizes from 30 or 50% hydrogen peroxide (Dobrynina & Dzyatkevich, 1967; Jones & Griffith, 1980). However, the crystals of (II) grown from dilute H_2O_2 solutions were of poor quality and not suitable for single-crystal X-ray structural investigations. A preliminary study of (II) revealed an orthorhombic unit cell with dimensions $a = 5.926$, $b = 8.444$ and $c = 17.827$ Å, in good agreement with the values previously found for $Rb_2CO_3{\cdot}3H_2O_2$ (a = 5.65, b = 8.15 and c = 18.01 Å) from powder diffraction data (Bakulina *et al.*, 1972). However, all attempts to solve the structure of (II) using that unit cell failed. Further detailed examination showed that the actual unit cell is doubled along the *b* axis. The axes were then transformed to the standard setting for the space group *Pccn*.

Compound (II) is an example of a structure in which hydrogen peroxide simultaneously coordinates an alkali metal and serves as a donor in hydrogen bonds to oxyacid ions. In the structure of (II), the three Cs^+ ions are crystallographically independent. Cation Cs1 occupies a general position, and the other two Cs^+ ions lie on twofold axes. Cations Cs1, Cs2 and Cs3 possess irregular coordination polyhedra with coordination numbers 13, 10, and 14, respectively. The Cs—O distances vary within the range 3.0558 (18)–3.5127 (18) Å. There are four independent peroxide molecules in (II), two of them arranged on twofold axes. One of these is orientationally disordered over two positions with an occupancy ratio of 0.812 (7):0.188 (7) (Fig. 3*b*). Both the major and minor components of the disordered molecule are anchored to the same carbonate and Cs^+ ions. The same type of disorder was observed previously in both polymorphs of $Na_2CO_3{\cdot}1.5H_2O_2$ (Adams & Pritchard, 1977; Carrondo *et al.*, 1977; Pritchard &

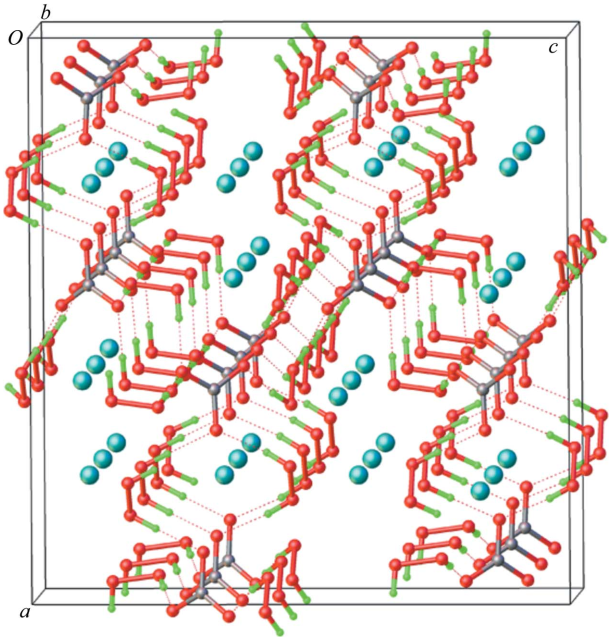

Figure 5
The structure of (II), showing how the Cs^+ ions fill the cavities in the hydrogen-bonded three-dimensional network formed by the carbonate ions and hydrogen peroxide molecules.

Islam, 2003). The O—O bond lengths are in the range 1.464 (3)–1.470 (3) Å (Table 3). All of the peroxide O atoms (except O6) coordinate three Cs^+ ions (Fig. 3). Atom O6 is bonded to only one Cs^+ ion.

All peroxide molecules are involved as donors in only two hydrogen bonds with adjacent carbonate anions (Table 4). No hydrogen bonds were observed between peroxide molecules. The maximum allowed number of hydrogen bonds (six) are formed by the carbonate ion (Fig. 4). These hydrogen bonds are almost linear [167 (3)–179 (3)°] and the O···O separations are in the range 2.596 (2)–2.656 (3) Å. As expected, the carbonate–peroxide C—O···O angles are close to the ideal value of 120° for sp^2-hybridized carbonate O atoms serving as donors of lone electron pairs.

In the crystal structure, the hydrogen peroxide molecules and carbonate anions are linked by hydrogen bonds in a complicated three-dimensional-network (Fig. 5). Cs^+ ions fill the cavities in this extended framework.

Experimental

Ammonium carbamate, ammonium hydrocarbonate, caesium carbonate and 50% hydrogen peroxide were purchased from Sigma–Aldrich (CAS No. 506-87-6). The 98% hydrogen peroxide was prepared by an extraction method from serine peroxosolvate (Wolanov *et al.*, 2010). **CAUTION–HAZARD:** concentrated hydrogen peroxide is corrosive and its use carries significant fire and explosion risks. Consult full current safety documentation before use. Handling procedures are described in detail elsewhere (Schumb *et al.*, 1955; Maass & Hatcher, 1920).

Colourless crystals of (I) and (II) were obtained by cooling saturated solutions (room temperature) of anhydrous ammonium carbamate, $(NH_4)(COONH_2)$, and anhydrous caesium carbonate, Cs_2CO_3, in 98% H_2O_2 (H_2O_2–H_2O molar ratio of approximately 26:1) to 255 K. Tiny crystals of (I) also crystallize from a saturated solution (room temperature) of ammonium hydrocarbonate NH_4HCO_3 in 30% H_2O_2 at 255 K. The crystals of (I) and (II) are stable for several hours in air.

The crystals were extracted from the mother liquor using a plastic spatula and covered immediately with inert oil to prevent contact with atmospheric moisture. They were then rapidly mounted on the top of a hair fibre and transferred to a cold nitrogen stream on the diffractometer.

Compound (I)

Crystal data

$2NH_4^+\cdot CO_3^{2-}\cdot H_2O_2$
$M_r = 130.11$
Monoclinic, $P2/n$
$a = 7.5657\ (9)$ Å
$b = 9.9027\ (12)$ Å
$c = 7.5819\ (9)$ Å
$\beta = 98.050\ (2)°$
$V = 562.45\ (12)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.16$ mm^{-1}
$T = 150$ K
0.40 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.940$, $T_{max} = 0.970$
5575 measured reflections
1349 independent reflections
1256 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$

Table 1
Selected geometric parameters (Å, °) for (I).

C1—O12	1.2846 (8)	O1—O1^i	1.4652 (14)
C1—O11	1.2928 (14)	O1—H1	0.855 (15)
C2—O22	1.2835 (8)	O2—O2^{ii}	1.4685 (13)
C2—O21	1.2965 (14)	O2—H2	0.843 (15)
O12^i—C1—O12	120.15 (11)	O22—C2—O21	119.79 (5)
O12^i—C1—O11	119.92 (5)	O22^{ii}—C2—O21	119.79 (5)
O12—C1—O11	119.92 (5)	O1^i—O1—H1	98.9 (9)
O22—C2—O22^{ii}	120.42 (10)	O2^{ii}—O2—H2	98.3 (10)
H1—O1—O1^i—H1^i	−133 (2)	H2—O2—O2^{ii}—H2^{ii}	137 (2)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O22^{iii}	0.855 (15)	1.816 (15)	2.6683 (10)	175.0 (14)
O2—H2···O12	0.843 (15)	1.823 (16)	2.6648 (10)	175.9 (15)
N1—H11···O12^{iii}	0.871 (14)	1.948 (14)	2.7965 (10)	164.4 (12)
N1—H12···O11	0.881 (13)	1.961 (13)	2.8376 (9)	173.7 (11)
N1—H13···O21	0.859 (15)	2.029 (15)	2.8795 (8)	170.7 (13)
N1—H14···O22^{iv}	0.893 (13)	1.989 (13)	2.8429 (10)	159.5 (12)
N2—H21···O11^{v}	0.871 (15)	2.096 (15)	2.9361 (9)	161.8 (13)
N2—H22···O22^{vi}	0.881 (13)	1.921 (13)	2.7817 (10)	165.0 (12)
N2—H23···O12	0.886 (13)	1.934 (14)	2.8136 (10)	171.9 (12)
N2—H24···O21^{v}	0.893 (14)	1.974 (14)	2.8615 (9)	172.0 (12)

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

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.074$
$S = 1.12$
1349 reflections
115 parameters
All H-atom parameters refined
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Compound (II)

Crystal data

$2Cs^+\cdot CO_3^{2-}\cdot 3H_2O_2$
$M_r = 427.88$
Orthorhombic, $Pccn$
$a = 17.820\ (2)$ Å
$b = 5.9357\ (8)$ Å
$c = 16.888\ (2)$ Å
$V = 1786.3\ (4)$ Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 8.18$ mm^{-1}
$T = 173$ K
0.25 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.234$, $T_{max} = 0.373$
16916 measured reflections
2154 independent reflections
1841 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.018$
$wR(F^2) = 0.047$
$S = 1.12$
2154 reflections
134 parameters
16 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.88$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (II).

O1—O1^{i}	1.465 (4)	O5—O6	1.470 (3)
O2*A*—O2*A*^{ii}	1.469 (5)	C1—O11	1.281 (4)
O2*A*—H2*A*	0.89 (2)	C1—O12	1.285 (3)
O2*B*—O2*B*^{ii}	1.470 (15)	C1—O13	1.290 (3)
O3—O4	1.464 (3)		
O1^{i}—O1—H1	99 (2)	O6—O5—H5	100 (2)
O2*A*^{ii}—O2*A*—H2*A*	99 (3)	O5—O6—H6	100 (2)
O2*B*^{ii}—O2*B*—H2*B*	102	O11—C1—O12	120.5 (2)
O4—O3—H3	102 (2)	O11—C1—O13	119.9 (2)
O3—O4—H4	98 (2)	O12—C1—O13	119.7 (2)
H1—O1—O1^{i}—H1^{i}	67 (5)	H3—O3—O4—H4	−83 (3)
H2*A*—O2*A*—O2*A*^{ii}—H2*A*^{ii}	58 (6)	H5—O5—O6—H6	−65 (3)
H2*B*—O2*B*—O2*B*^{ii}—H2*B*^{ii}	−48		

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

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O11^{iii}	0.88 (2)	1.77 (2)	2.642 (2)	167 (3)
O2*A*—H2*A*···O11^{iv}	0.89 (2)	1.74 (2)	2.628 (3)	175 (4)
O2*B*—H2*B*···O11^{iv}	0.90	1.72	2.619 (10)	178
O3—H3···O13	0.87 (2)	1.72 (2)	2.596 (2)	179 (3)
O4—H4···O13^{iv}	0.89 (2)	1.75 (2)	2.637 (3)	177 (3)
O5—H5···O12^{v}	0.88 (2)	1.73 (2)	2.603 (2)	173 (3)
O6—H6···O12	0.87 (2)	1.79 (2)	2.656 (3)	177 (3)

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

In (I), all H atoms were found in a difference Fourier synthesis and refined isotropically. In (II), atom O2*B* belonging to the minor disorder component [occupancy 0.188 (7)] was refined isotropically, with a similarity restraint (weight 0.02 Å) between the O—O distances in the two components. All H atoms (except disordered atom H2*B*) were found in a difference Fourier synthesis and refined with $U_{iso}(H) = 1.5U_{eq}(O)$ and O—H distances restrained to be similar with a weight of 0.02 Å. Atom H2*B* was placed on a line between atoms O2*B* and O11 at a distance of 0.90 Å from O2*B*, and was refined using a riding model with $U_{iso}(H2B) = 1.5U_{eq}(O2B)$.

For both compounds, data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *SHELXTL*.

The authors thank the Israel Ministry of Science (MOS) and the Russian Foundation for Basic Research (grant No. 11-03-92478-a) for financial support. AVC is grateful to the Royal Society of Chemistry for an international travel grant. This research is supported by the Singapore National Research Foundation under CREATE programme: Nanomaterials for Energy and Water Management.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LG3077). Services for accessing these data are described at the back of the journal.

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