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A new phase of 7,16-dibenzyl-1,4,10,13-tetraoxa-7,16-diazacyclooctadecane, and 7,16-dibenzyl-1,4,10,13-tetraoxa-7,16-diazoniacyclooctadecane bis(tetrafluoroborate) monohydrate, both determined at 123 K

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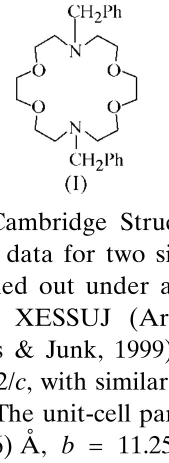
7,16-Dibenzyl-1,4,10,13-tetraoxa-7,16-diazacyclooctadecane, $C_{26}H_{38}N_2O_4$, (I), crystallizes in space group $P2_1/c$, with two independent molecules adopting different conformations. The 'free' crowns adopt a typical 'parallelogram' shape, in which two methylene groups are turned inward toward the center of the ring and the benzyl groups splay out from the ring. In 7,16-dibenzyl-1,4,10,13-tetraoxa-7,16-diazoniacyclooctadecane bis(tetrafluoroborate) monohydrate, $C_{26}H_{40}N_2O_4^{2+}\cdot 2BF_4^{-}\cdot H_2O$, (II), the macrocycle is centrosymmetric, and the protonated N atoms adopt an *endo–endo* orientation that is stabilized by a bifurcated N—H···O hydrogen bond, where the O atoms of the macrocycle act as hydrogen-bond acceptors. The phenyl groups of the benzyl side arms are turned above and below the macrocycle; C—H···π interactions between the phenyl substituents and two macrocyclic methylene H atoms govern the overall conformation of the macrocycle. Bridging tetrafluoroborate anions link the macrocyclic cations *via* weak C—H···F hydrogen bonds into channels running along [100], which are filled by the weakly hydrogen-bonded water molecules.

Comment

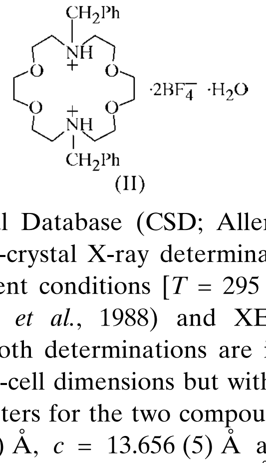
The ability of crown ethers to form host–guest complexes with normally unstable or volatile species is well known (Bott *et al.*, 1991; Chuit *et al.*, 1993; Feinberg *et al.*, 1993). We have previously reported that the classic crown ethers (18-crown-6 and isomers of dicyclohexyl-18-crown-6) provide the opportunity to hold harmful gaseous (SiF_4; Simonov *et al.*, 1994) or low-melting substances ($BF_3\cdot H_2O$; Fonari *et al.*, 1997) in the form of hydrogen-bonded molecular or ionic complexes.

A general method of synthesis of host–guest-type complexes of boron–fluoro complexes with crown ethers, based on the interaction of boron trifluoride etherate, $BF_3\cdot OEt_2$, with the corresponding ligand in the atmosphere not protected from moisture, has been developed (Gelmboldt *et al.*, 1995; Simonov *et al.*, 1995). This method was further elaborated for the preparation of complexes based on nitrogen-containing macrocycles (Fonari *et al.*, 1999; Lipkowski *et al.*, 2001). On the basis of these studies, it was shown that the composition of the 'guest', the product of hydrolytic transformations of $BF_3\cdot OEt_2$, is dictated by the nature of the macrocycle. Crown ethers offering different extents of shielding of the macrocyclic cavity stabilize either molecular ($BF_3\cdot H_2O$) or ionic ($H_3O^+\cdot BF_4^-$) complexes, while nitrogen-containing macrocycles, which are easily protonated in the acidic medium, form complexes exclusively with the tetrafluoroborate anions. The components are linked in the crystal structure *via* a diverse system of O—H···O and N—H···F hydrogen bonds.

As part of our investigation of the coordination abilities of boron trifluoride with crown ethers and their derivatives, we focus here on *N*,*N*′-dibenzyl-1,4,10,13-tetraoxa-7,16-diazacyclooctadecane, (I). Compound (I) contains a mixed N,O-atom donor function that could provide diverse possibilities for the realization of hydrogen bonds with neutral boron trifluoride molecules or with the tetrafluoroborate anion. C—H···π interactions may also contribute to the conformational stability of the macrocycle.



(I)



(II)

The Cambridge Structural Database (CSD; Allen, 2002) contains data for two single-crystal X-ray determinations of (I), carried out under ambient conditions [T = 295 K; CSD refcodes XESSUJ (Arnold *et al.*, 1988) and XESSUJ01 (Fewings & Junk, 1999)]. Both determinations are in space group $C2/c$, with similar unit-cell dimensions but with high R factors. The unit-cell parameters for the two compounds [a = 19.487 (6) Å, b = 11.259 (2) Å, c = 13.656 (5) Å and β = 121.10 (3)° for XESSUJ, and a = 19.479 (2) Å, b = 11.235 (3) Å, c = 17.038 (3) Å and β = 136.68 (0)° for XESSUJ01] can be easily transformed to the same unit cell [with dimensions a = 13.656 Å, b = 11.259 Å, c = 17.068 Å and β = 102.14° for XESSUJ, and a = 13.668 Å, b = 11.235 Å, c = 17.038 Å and β = 102.11° for XESSUJ01] using the transformation matrices (001/010/101) and ($\bar{1}0\bar{1}/0\bar{1}0/001$), respectively. In the new setting, both structures again belong to space group

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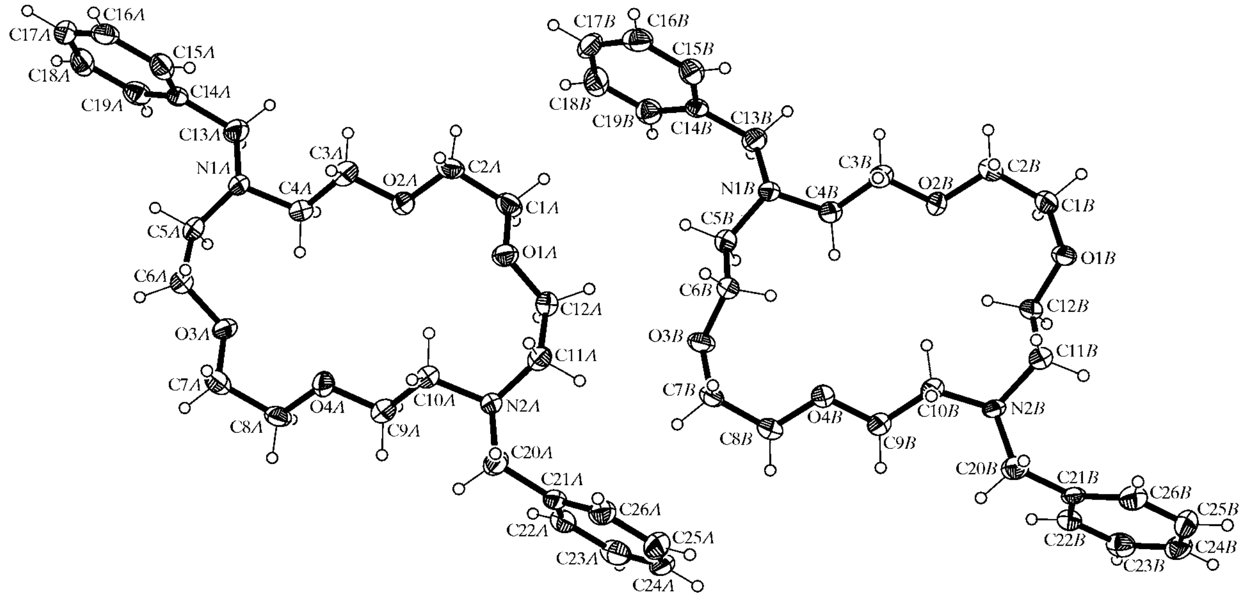

Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

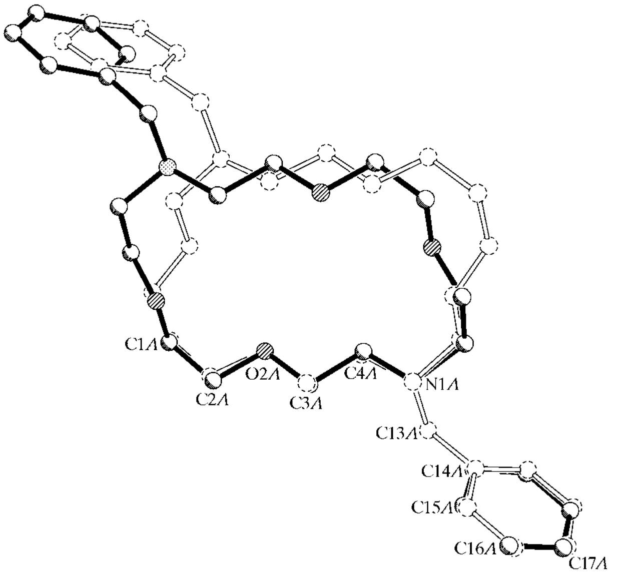

Figure 2
Superimposition of molecules *A* and *B* in (I), in projection on the labeled atoms.

*C*2/*c*, so XESSUJ and XESSUJ01 report the same structure, as confirmed by the practically ideal superposition of these molecules.

In our experiment, carried out at 123 K, the unit-cell dimensions [a = 16.993 (3) Å and c = 13.391 (3) Å] are close to the calculated c and a parameters of the new setting, although in an inverted order and with a reduction explained by the low-temperature conditions of the experiment, while the b parameter [b = 22.455 (5) Å] is about two times longer. We found a structure solution in space group $P2_1/c$. The phase transition and the structure of (I) are assumed to correspond to a low-temperature phase with lower symmetry. Fig. 1 shows the asymmetric unit, containing two independent crown molecules (molecules *A* and *B*), which differ essentially in their conformations. This difference is clearly evident from Fig. 2, which depicts the superposition of molecules *A* and *B*. The lack of inversion centers in the two molecules is evidenced by the values of the dihedral angles between the planes of the aromatic rings C14–C19 and C21–C26 [3.6 (2) and 4.4 (2)° in molecules *A* and *B*, respectively], which deviate from 0° as expected in a molecule imposed on an inversion center. The 'free' crowns adopt a typical 'parallelogram' shape, in which two methylene moieties turn inward toward the center of the ring. The shortest intramolecular distances across the cavity are C4···O4 and C10···O2, being 4.157 (3) and 4.151 (3) Å in molecule *A*, and 4.299 (3) and 4.306 (3) Å in molecule *B*; the longest distances are C1···C7 and C5···C11, being 7.384 (4) and 8.611 (4) Å in molecule *A*, and 8.518 (4) and 7.387 (4) Å in molecule *B*.

Each conformer in (I) is very close to being centrosymmetric, as shown by the torsion angles in Table 1. The torsion angles run in the sequences $g\bar{g}aagaaaa\bar{g}gaa\bar{g}aaaa$ and $gaa\bar{g}gaaaa\bar{g}aag\bar{g}aaaa$ in molecules *A* and *B*, respectively [in the anticlockwise direction, beginning from the C4—N1—C5—C6 angle, where g denotes *gauche* (+60 to +120°), $\bar{g}$ denotes *gauche* (−60 to −120°) and a denotes antiperiplanar (±120 to ±180°)]. The differences in the values of the angles that are situated in the macrocycles in the same positions relative to the centers of the rings (the point of the intersection of three transannular O···O distances) range from 0° for the C5—C6—O3—C7 and C11—C12—O1—C1 angles, up to 2.1° for the C6—O3—C7—C8 and C12—O1—C1—C2 angles in molecule *A*, and range from 0.2° for the C2—O2—C3—C4 and C8—O4—C9—C10 angles, up to 3.1° for the C6—O3—C7—C8 and C12—O1—C1—C2 angles in molecule *B*.

Our calculations of the torsion angles in the independent part of the centrosymmetric molecule of XESSUJ revealed the following sequence (anticlockwise, running from the C4—N1—C5—C6 angle to the C9—C10—N2—C11 angle): −73.1, 107.7, −179.1, 139.2, −69.4, 171.8, 173.2, 177.1 and −159.2°. These values are close to the inverse of the torsion-angle pattern given in Table 1. The common point is a sequence of four *anti* torsion angles that is typical for the pure phases of oxygen-containing crown ethers, for example 18-crown-6 (Maverick *et al.*, 1980) and *cis-trans-cis*-dicyclohexano-18-crown-6 (Kravtsov *et al.*, 2002). The benzyl side arms splay out from the crown rather than lying below and above the macrocyclic plane. Surprisingly, there does not appear to be any intermolecular π stacking in (I).

The centrosymmetric formula unit of compound 7,16-dibenzyl-1,4,10,13-tetraoxa-7,16-diazoniacyclooctadecane bis(tetrafluoroborate) monohydrate, (II), is shown in Fig. 3. The slight distortion of the BF_4^- tetrahedron is indicated by the B—F distances being in the range 1.384 (2)–1.399 (2) Å and the F—B—F angles being in the range 108.05 (15)–110.94 (14)°.

The twofold protonated macrocyclic cation exhibits crystallographically imposed C_i symmetry. The protonated N atoms adopt an *endo–endo* orientation that is stabilized by three-center N—H$\cdots$O hydrogen bonds of 2.709 (2) and 2.901 (2) Å, where the O atoms of the macrocycle act as hydrogen-bond acceptors (Table 3). The 18-membered macrocyclic cavity exhibits a degree of rigidity and adopts a chair conformation, with the O atoms lying exactly in one plane and the N atoms deviating 1.609 (2) Å above and below this plane. This rigidity probably stems from the intramolecular N—H$\cdots$O hydrogen bonds, and from the presence of C—H$\cdots\pi$ interactions between two methylene H atoms and the aromatic subunits, as well as a C—H$\cdots$O intramolecular hydrogen bond of 3.395 (2) Å (Table 3 and Fig. 3). These interactions effectively anchor the ring into its present conformation, and turn the phenyl groups of the benzyl side arms above and below the macrocycle in such a way that the dihedral angle between the plane of the aromatic ring and the plane of the crown O atoms is 42.70 (5)°. The C$\cdots$*Cg*1 and H$\cdots$*Cg*1 (*Cg*1 is the centroid of the C11–C16 ring) distances of 3.758 (2) and 2.82 Å, and the C—H$\cdots$*Cg*1 angle of 164°, suggest the presence of strong $\pi\cdots$H interactions at the center of the aromatic ring (Braga *et al.*, 1998). Both of these types of intramolecular interaction in this dication have been noted for a series of lanthanoid complexes (Evans *et al.*, 2002) and seem to be reasonable in $[(I)H_2]I_8$ (Gaballa *et al.*, 2004). The involvement of methylene H atoms in these interactions is unusual in the light of possible competition with, for example, water H atoms.

The conformation of the macrocyclic cavity strongly deviates from that of the 'free' macrocycle; the corresponding torsion angles are listed in Table 2. Inspection of the torsion angles (absolute values 50–87 and 152–176°) reveals that the macrocycle is characterized by partial conformations [(*anti*, *gauche*, *anti*), (*anti*, *gauche*, *anti*) and (*gauche*, *gauche*, *gauche*)] of the N1—C2—C3—O4, O4—C5—C6—O7 and O7—C8—C9—N1′ subunits. The shape and conformation of the macrocyclic cation in (II) is close to that found in its complex with octaiodide (Gaballa *et al.*, 2004) and in the neodymium complex $[(I)H_2][Nd(NO_3)_5(H_2O)]$ (Saleh *et al.*, 1998). Protonation of the aza-crown ether and the above-mentioned intramolecular interactions close the cavity against the inclusion of neutral (water) or charged (BF_4^-) species that appear to occupy the outer space of the crowns.

BF_4^- anions fulfill bridging functions, and in the *bc* plane they bind the macrocyclic cations *via* multiple weak C—H$\cdots$F hydrogen bonds, forming channels propagated along the *a* direction. The shortest C—H$\cdots$F contacts range from 3.278 (2) to 3.395 (2) Å and are summarized in Table 2, both aromatic and aliphatic H atoms being involved in these interactions. Water molecules are held in the center of the channel, as seen in Fig. 4, and because of the weak O—H$\cdots$F

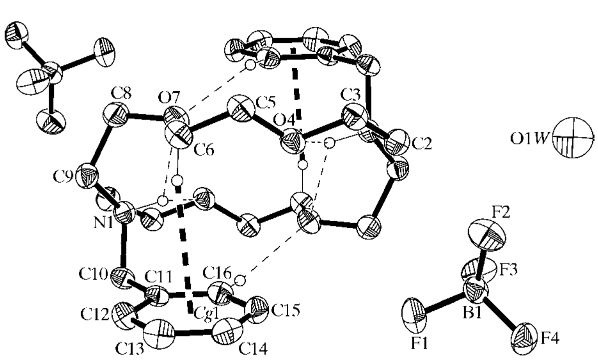

Figure 3
The structure of (II), showing the C—H$\cdots\pi$ interactions. *Cg*1 is the centroid of the C11–C16 ring. Displacement ellipsoids are drawn at the 50% probability level and only H atoms involved in specific contacts are shown.

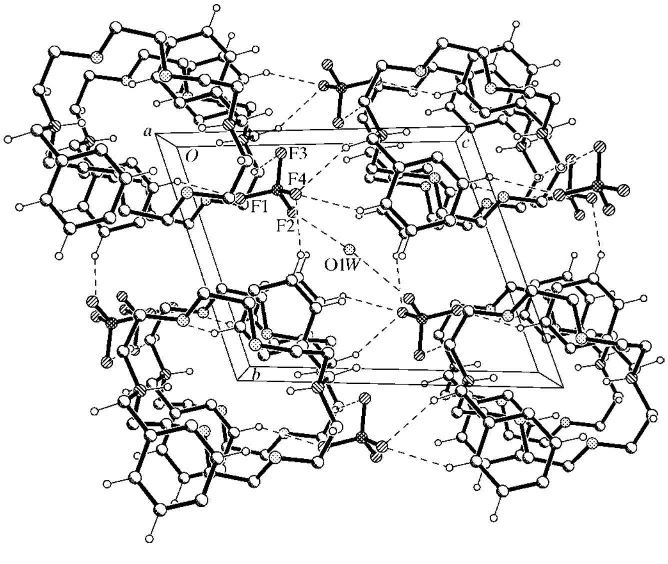

Figure 4
Part of the crystal structure of (II), showing the channels formed by hydrogen-bonded macrocyclic cations and BF_4^- anions, and the water molecules that occupy these channels. Only H atoms involved in specific contacts are shown.

hydrogen bond (O· · ·F = 2.83 Å) they are disordered around the inversion center. As shown above, the idealized stoichiometry of the $[(I)H_2](BF_4)_2\cdot H_2O$ complex is 1:2:1, whilst really only about 20% of the water content of each complex appears to be retained in the crystal lattice.

Experimental

Compound (I) was prepared according to the procedure of Gustowski *et al.* (1987). To obtain complex (II), compound (I) (244 mg, 0.1 mmol) was dissolved in methanol (5 ml). The reaction mixture was allowed to stand until crystals were deposited. These were filtered off and recrystallized from a 1:1 mixture of methanol and ethyl acetate to yield colorless transparent crystals (m.p. 441–443 K). Analysis calculated for $C_{26}H_{42}B_2F_8N_2O_5$: C 49.1, H 6.65, F 23.9, N 4.4%; found: C 49.0, H 6.7, F 23.9, N 4.4%.

Compound (I)

Crystal data

$C_{26}H_{38}N_2O_4$
$M_r = 442.58$
Monoclinic, $P2_1/c$
$a = 16.993\ (3)$ Å
$b = 22.455\ (5)$ Å
$c = 13.391\ (3)$ Å
$\beta = 103.48\ (3)^\circ$
$V = 4968.9\ (18)$ Å^3
$Z = 8$
$D_x = 1.183$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 15 860 reflections
$\theta = 1$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 123\ (2)$ K
Prism, colorless
$0.20 \times 0.20 \times 0.15$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
25 950 measured reflections
8591 independent reflections
5565 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.060$
$\theta_{\max} = 25.0^\circ$
$h = -20 \rightarrow 19$
$k = -26 \rightarrow 20$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.141$
$S = 1.10$
8591 reflections
577 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 2.1759P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected torsion angles (°) for (I).

O1*A*—C1*A*—C2*A*—O2*A*	−70.7 (3)	O1*B*—C1*B*—C2*B*—O2*B*	−74.7 (3)
C1*A*—C2*A*—O2*A*—C3*A*	174.2 (2)	C1*B*—C2*B*—O2*B*—C3*B*	178.7 (2)
C2*A*—O2*A*—C3*A*—C4*A*	174.4 (2)	C2*B*—O2*B*—C3*B*—C4*B*	171.7 (2)
O2*A*—C3*A*—C4*A*—N1*A*	178.1 (2)	O2*B*—C3*B*—C4*B*—N1*B*	178.0 (2)
C3*A*—C4*A*—N1*A*—C5*A*	−162.6 (2)	C3*B*—C4*B*—N1*B*—C5*B*	−156.1 (2)
C4*A*—N1*A*—C5*A*—C6*A*	68.6 (3)	C4*B*—N1B—C5*B*—C6B	76.6 (3)
N1*A*—C5*A*—C6*A*—O3*A*	−87.2 (3)	N1*B*—C5*B*—C6*B*—O3*B*	−161.3 (2)
C5*A*—C6*A*—O3*A*—C7*A*	178.1 (2)	C5*B*—C6*B*—O3*B*—C7*B*	167.3 (2)
C6*A*—O3*A*—C7*A*—C8*A*	−157.6 (2)	C6*B*—O3*B*—C7*B*—C8*B*	−88.1 (3)
O3*A*—C7*A*—C8*A*—O4*A*	72.4 (3)	O3*B*—C7*B*—C8*B*—O4*B*	72.1 (3)
C7*A*—C8*A*—O4*A*—C9*A*	−174.7 (2)	C7*B*—C8*B*—O4*B*—C9*B*	−177.4 (2)
C8*A*—O4*A*—C9*A*—C10*A*	−173.6 (2)	C8*B*—O4*B*—C9*B*—C10*B*	−171.9 (2)
O4*A*—C9*A*—C10*A*—N2*A*	177.9 (2)	O4*B*—C9*B*—C10*B*—N2*B*	−173.7 (2)
C9*A*—C10*A*—N2*A*—C11*A*	159.7 (2)	C9*B*—C10*B*—N2*B*—C11*B*	158.2 (2)
C10*A*—N2*A*—C11*A*—C12*A*	−68.9 (3)	C10*B*—N2*B*—C11*B*—C12*B*	−78.4 (3)
N2*A*—C11*A*—C12*A*—O1*A*	87.8 (3)	N2*B*—C11*B*—C12*B*—O1*B*	164.1 (2)
C11*A*—C12*A*—O1*A*—C1*A*	−178.1 (2)	C11*B*—C12*B*—O1*B*—C1*B*	−164.7 (2)
C12*A*—O1*A*—C1*A*—C2*A*	159.1 (2)	C12*B*—O1*B*—C1*B*—C2*B*	85.0 (3)

Compound (II)

Crystal data

$C_{26}H_{40}N_2O_4^{2+}\cdot 2BF_4^-\cdot H_2O$
$M_r = 621.48$
Triclinic, $P\overline{1}$
$a = 7.9290\ (6)$ Å
$b = 9.3870\ (6)$ Å
$c = 11.6070\ (7)$ Å
$\alpha = 69.342\ (4)^\circ$
$\beta = 72.226\ (4)^\circ$
$\gamma = 81.480\ (4)^\circ$
$V = 768.98\ (9)$ Å^3

$Z = 1$
$D_x = 1.342$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3035 reflections
$\theta = 1$–27.5°
$\mu = 0.12$ mm^{-1}
$T = 123\ (2)$ K
Prism, colorless
$0.25 \times 0.20 \times 0.20$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
4974 measured reflections
2660 independent reflections
2159 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.027$
$\theta_{\max} = 25.0^\circ$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.102$
$S = 1.04$
2660 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 0.1724P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 2
Selected torsion angles (°) for (II).

N1$^{\rm i}$—C2—C3—O4	−50.1 (2)	C6—O7—C8—C9	74.0 (2)
C2—C3—O4—C5	165.4 (1)	O7—C8—C9—N1	60.3 (2)
C3—O4—C5—C6	−176.2 (1)	C8—C9—N1—C2$^{\rm i}$	86.8 (2)
O4—C5—C6—O7	64.6 (2)	C9—N1—C2$^{\rm i}$—C3$^{\rm i}$	−164.0 (1)
C5—C6—O7—C8	152.6 (1)		

Symmetry code: (i) $1 - x, -y, -z$.

Table 3
Hydrogen-bonding geometry (Å, °) for (II).

D—H· · ·*A*	*D*—H	H· · ·*A*	*D*· · ·*A*	*D*—H· · ·*A*
N1—H1N· · ·O4$^{\rm i}$	0.91 (2)	2.25 (2)	2.709 (2)	110.2 (14)
N1—H1N· · ·O7	0.91 (2)	2.43 (2)	2.901 (2)	112.6 (14)
C2—H2*B*· · ·F3	0.99	2.43	3.278 (2)	143
C10—H10*B*· · ·F4$^{\rm ii}$	0.99	2.43	3.345 (2)	154
C12—H12*A*· · ·F4$^{\rm ii}$	0.95	2.53	3.383 (2)	150
C13—H13*A*· · ·F4$^{\rm iii}$	0.95	2.41	3.279 (2)	152
C15—H15*A*· · ·F1	0.95	2.51	3.368 (2)	151
C16—H16*A*· · ·O7$^{\rm i}$	0.95	2.52	3.395 (2)	154

Symmetry codes: (i) $1 - x, -y, -z$; (ii) $x, y, z - 1$; (iii) $2 - x, 1 - y, -z$.

All non-H atoms, except O1*W*, were refined with anisotropic displacement parameters. C-bound H atoms in (I) and in the $[(I)H_2]^{2+}$ ion in (II) were placed in calculated positions and treated using a riding model. The N-bound H atom in (II) was located in a difference Fourier map and refined $[U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm N})]$. After the refinement, the difference Fourier map revealed a peak of 0.8 e Å^3. The reasonable O· · ·F distances permit the conclusion that this is caused by a water molecule partially occupying a disordered position around an inversion center. The refined site occupancy is 0.102 (6). Because this value is so low, atom O1*W* was refined with an isotropic displacement parameter and its H atoms were not localized.

For both compounds, data collection: *COLLECT* (Hooft, 1998) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *COLLECT* and *DENZO*; data reduction: *COLLECT* and *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1362). Services for accessing these data are described at the back of the journal.

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