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Crystal structure of silver [(propane-1,3-diyldinitrilo-κ^2N,N')tetraacetato-κ^4O,O',O'',O''']chromate(III) from synchrotron X-ray data

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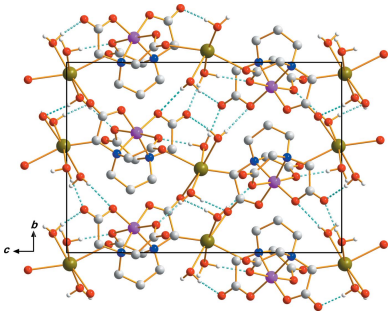
The asymmetric unit of the title compound, $Ag[Cr(C_{11}H_{14}N_2O_8)]\cdot 3H_2O$, contains one $[Cr(1,3\text{-pdta})]^-$ anion [1,3-pdta is (propane-1,3-diyldinitrilo)tetraacetate], one Ag^+ cation and three water molecules. The Cr^{3+} ion is coordinated to the four O and two N atoms of the 1,3-pdta ligand, displaying a distorted octahedral geometry. The mean Cr—N and Cr—O bond lengths are 2.0727 (17) and 1.9608 (15) Å, respectively. The conformations of the chelate rings were found to be envelope for the glycinates and twist-boat for the six-membered diamine (*T*) ring. The Ag^+ cation is surrounded by six O atoms from three non-coordinated carbonyl O atoms of neighbouring 1,3-pdta groups and three water molecules. The crystal structure is stabilized by intermolecular hydrogen bonding involving the water O—H group as donor and the carboxyl O atom as acceptor.

1. Chemical context

The hexadentate ligand, propane-1,3-diyldinitrilotetraacetate (abbreviated here as 1,3-pdta, $C_{11}H_{14}N_2O_8$) has been used for the preparation of complexes with many transition metal ions (Herak *et al.*, 1984; Yamamoto *et al.*, 1988; Douglas & Radanović, 1993). In the complex anion, $[M(1,3\text{-pdta})]^{n-}$, the six-membered propane-1,3-diamine ring is referred to as the *T* ring, the equatorially coordinated glycinate ring as the *G* ring, and the axially coordinated glycinate ring as the *R* ring (see Scheme). The counter-ion and metal-center oxidation state play a very important role in conformational isomerism. Upon coordination of 1,3-pdta by a metal center, the six-membered *T* ring can take twist-boat or half-chair conformers (Meier *et al.*, 2007). The twist-boat conformer was found in the crystal structures of $K[Co(1,3\text{-pdta})]\cdot 2H_2O$ (Nagao *et al.*, 1972), $Li[Fe(1,3\text{-pdta})]\cdot 3H_2O$ (Yamamoto *et al.*, 1988) and $Na[Cr(1,3\text{-pdta})]\cdot 3H_2O$ (Herak *et al.*, 1984), whereas the half-chair form was observed in structural studies of $[C(NH_2)_3][Fe(1,3\text{-pdta})]\cdot 2H_2O$ (Meier *et al.*, 2007) and $Li_2[Co(1,3\text{-pdta})]\cdot 3H_2O$ (Rychlewska *et al.*, 2008). The crystal structure of $Na[Cr(1,3\text{-pdta})]\cdot 3H_2O$ (Herak *et al.*, 1984) has also been reported previously. In this communication, we report the crystal structure of $Ag[Cr(1,3\text{-pdta})]\cdot 3H_2O$ in order to clarify unambiguously the bonding mode and the conformational geometry adopted by the Ag^+ salt.



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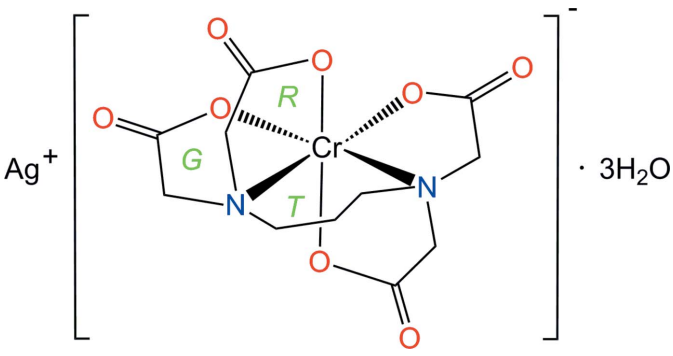

2. Structural commentary

This is another example of a $[Cr(1,3\text{-pdta})]^-$ anion but with a different cation. The crystal structure of the title compound is isotypic with $Na[M(1,3\text{-pdta})]\cdot 3H_2O$ (M = Fe, Cr or Rh; Okamoto *et al.*, 1990; Herak *et al.*, 1984) but it belongs to the orthorhombic space group $P2_12_12_1$ compared with the monoclinic space group $P2_1/n$ of $Li[Fe(1,3\text{-pdta})]\cdot 3H_2O$ (Yamamoto *et al.*, 1988) and orthorhombic space group $B22_12$ of $K[Co(1,3\text{-pdta})]\cdot 2H_2O$ (Nagao *et al.*, 1972). The structural analysis shows that the propane-1,3-diyldinitrilotetraacetate anion is coordinated octahedrally by the Cr metal center through four O and two N atoms. An ellipsoid plot of title complex showing the atomic numbering is given in Fig. 1. The Cr—O bond distances differ slightly, the mean equatorial and axial distances being 1.9672 (15) and 1.9544 (15) Å, respectively. The *cis* angles at the Cr^{III} ion range from 81.66 (6) to 99.41 (6)° and the *trans* angles are 173.07 (7), 175.01 (6) and 176.04 (7)°. The six-membered propane-1,3-diamine T ring (Fig. 1) adopts a flexible twist-boat conformation. The R rings are nearly planar and are in an envelope conformation. The G rings are much more puckered and are halfway between an envelope and a twist conformation. The Cr—O bond distances are greater in the G rings than in the R rings, and the average Cr—N bond length of 2.0727 (17) Å is 0.1119 Å longer than the average Cr—O bond distance. The Cr—N and Cr—O bond distances are in accordance with the values observed in $Na[Cr(1,3\text{-pdta})]\cdot 3H_2O$. However, the average Ag—O distance of 2.525 (2) Å is slightly longer than the Na—O distance of 2.437 Å in $Na[Cr(1,3\text{-pdta})]\cdot 3H_2O$ (Herak *et al.*, 1984).

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

D—H···A	D—H	H···A	D···A	D—H···A
O9—H1*O*1···O3^{i}	0.84 (1)	1.95 (1)	2.797 (2)	178 (3)
O9—H2*O*1···O8^{ii}	0.85 (1)	1.93 (1)	2.767 (3)	172 (4)
O10—H1*O*2···O5^{iii}	0.85 (1)	2.02 (1)	2.870 (2)	173 (4)
O10—H2*O*2···O2^{iv}	0.85 (1)	1.89 (1)	2.729 (3)	170 (4)
O11—H1*O*3···O7^{ii}	0.84 (1)	2.33 (2)	3.142 (3)	163 (4)
O11—H2*O*3···O8^{v}	0.83 (1)	1.99 (2)	2.791 (3)	161 (3)

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

3. Supramolecular features

The Ag^+ cation is surrounded octahedrally by three water molecules (O9, O10 and O11) and three carboxylate O atoms [O6, O2^{iii}($x+\frac{1}{2}, -y+\frac{1}{2}, 1-z$) and O4^{i}($-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$)] that are not directly coordinated to the Cr atom (Fig. 1). Hydrogen bonds exist between the water molecules and the O atoms in the 1,3-pdta moiety (Table 1). An extensive array of these contacts generate a three-dimensional network of molecules stacked along the a-axis direction (Fig. 2). Non-coordinating and coordinating carboxylate O atoms take part in the formation of O—H···O hydrogen bonds, which contribute to the crystal packing stabilization and give rise to an infinite three-dimensional framework.

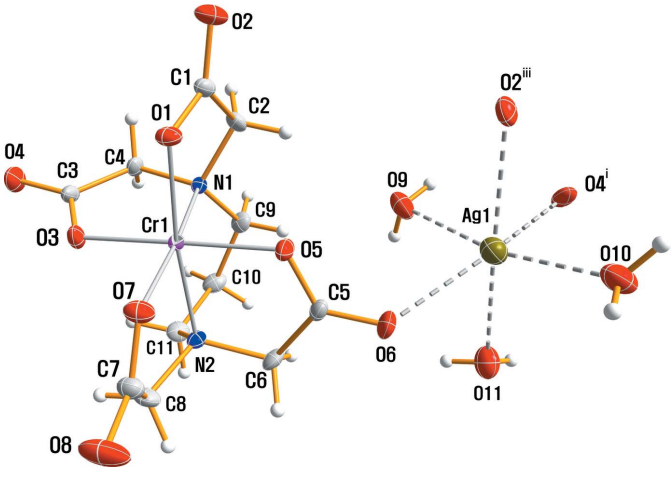

Figure 1
The structures of the molecular entities in compound (I), showing the atom-numbering scheme. Non-H atoms are shown with displacement ellipsoids at the 50% probability level. [Symmetry codes: (i) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, 1-z$.]

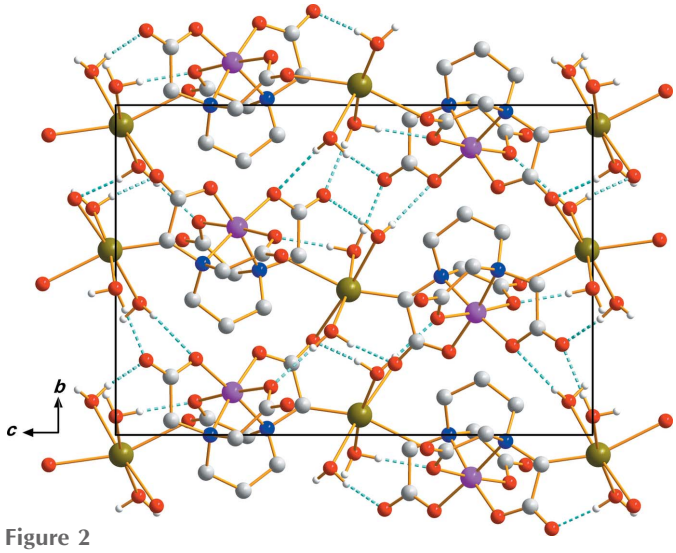

Figure 2
Crystal packing of $Ag[Cr(1,3\text{-pdta})]\cdot 3H_2O$, viewed perpendicular to the bc plane. Dashed lines represent O—H···O hydrogen-bonding interactions.

Table 2
Experimental details.

Crystal data	
Chemical formula	$Ag[Cr(C_{11}H_{14}N_2O_8)]\cdot 3H_2O$
M_r	516.16
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	260
a, b, c (Å)	8.7800 (18), 11.443 (2), 16.573 (3)
V (Å^3)	1665.1 (6)
Z	4
Radiation type	Synchrotron, λ = 0.610 Å
μ (mm^{-1})	1.25
Crystal size (mm)	0.17 × 0.13 × 0.07
Data collection	
Diffractometer	ADSC Q210 CCD area detector
Absorption correction	Empirical (using intensity measurements) (*HKL3000sm SCALEPACK*; Otwinowski & Minor, 1997)
T_{min}, T_{max}	0.843, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14937, 4807, 4738
R_{int}	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.706
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.020, 0.051, 1.07
No. of reflections	4807
No. of parameters	253
No. of restraints	9
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.41, −0.65
Absolute structure	Flack x determined using 2027 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter	−0.008 (6)

Computer programs: *PAL BL2D-SMDC* (Shin *et al.*, 2016), *HKL3000sm* (Otwinowski & Minor, 1997), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *DIAMOND* (Putz & Brandenburg, 2014) and *publCIF* (Westrip, 2010).

4. Database survey

A search of the Cambridge Structural Database (Version 5.38, May 2017 with three updates; Groom *et al.*, 2016) gave just three hits for a related complex anion, the $[Cr(C_{11}H_{14}N_2O_8)_2]^-$ unit. The crystal structure with an Na^+ counter-cation (Herak *et al.*, 1981, 1984) has been determined. The crystal structures of $Na[Cr(1,3\text{-pndta})]\cdot H_2O$, $K[Cr(1,3\text{-pndta})]\cdot H_2O$ and $Ca[Cr(1,3\text{-pndta})]_2\cdot 4H_2O$ (1,3-pndta = pentane-1,3-diyldinitrilotetraacetate; Warżajtis *et al.*, 2014) have been reported previously. However, no structure of a $[Cr(1,3\text{-pdta})]^-$ or $[Cr(1,3\text{-pndta})]^-$ complex with Ag^+ cation was found.

5. Synthesis and physical measurements

All chemicals were reagent-grade materials and were used without further purification. The UV–Vis absorption spectrum was recorded with a Cary 5000 UV–Vis–NIR Spectrophotometer. The FT–IR spectrum was obtained from a KBr pellet with a JASCO 460 plus series FT–IR spectrometer. Analyses for C, H, N were performed on a Carlo Erba 1108 Elemental Vario EL analyser. The precursor salt, $Na[Cr(1,3\text{-pdta})]\cdot 3H_2O$ was prepared as described previously (Weyh & Hamm, 1968; Herak *et al.*, 1984). The sodium salt (0.20 g) was dissolved in 15 mL of water at 323 K and added to 3 mL of water containing 0.5 g of $AgNO_3$. The resulting solution was filtered and left to stand at room temperature for several days to give purple block-shaped crystals of the silver salt, $Ag[Cr(1,3\text{-pdta})]\cdot 3H_2O$ suitable for X-ray structural analysis. Elemental analysis calculated for $Ag[Cr(C_{11}H_{14}N_2O_8)]\cdot 3H_2O$: C, 25.60; H, 3.91; N, 5.43%; found: C, 25.71; H, 3.23; N, 5.36%. UV–vis data (H_2O solution, nm): 201 (*vs*), 223 (*vs*), 245 (*sh*), 385 (*s*), 506 (*s*), 700 (*w*). IR spectrum (KBr, cm^{-1}) : 3447 (*vs, br*) (ν OH), 3232 (*sh*), 2977 (*vs*) and 2941 (*s*) (ν CH), 1643 (*s, br*) (ν_{as} COO), 1473 (*s*), 1428 (*m*), 1363 (*vs*) and 1327 (*vs*) (ν_s COO), 1271 (*sh*), 1222 (*s*), 1144 (*s*), 1099 (*vs*), 1061 (*m*), 1029 (*s*), 988 (*s*), 941 (*vs*), 916 (*vs*), 897 (*m*), 853 (*vs*), 746 (*vs*), 690 (*m*), 632 (*w*), 579 (*m*), 529 (*s*), 486 (*s*), 433 (*s*).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.97 Å with $U_{iso}(H) = 1.2U_{eq}(C)$. O-bound H atoms were assigned based on a difference-Fourier map, and were refined with distance restraints of 0.88 (2) Å (using DFIX and DANG commands), and $U_{iso}(H) = 1.2U_{eq}(O)$.

Funding information

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Crystal structure of silver [(propane-1,3-diyldinitrilo-κ^2N,N')tetraacetato-κ^4O,O',O'',O''']chromate(III) from synchrotron X-ray data

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Computing details

Data collection: *PAL BL2D-SMDC* (Shin *et al.*, 2016); cell refinement: *HKL3000sm* (Otwinowski & Minor, 1997); data reduction: *HKL3000sm* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXT2014* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Putz & Brandenburg, 2014); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Silver [(propane-1,3-diyldinitrilo-κ^2N,N')tetraacetato-κ^4O,O',O'',O''']chromate(III)

Crystal data

$Ag[Cr(C_{11}H_{14}N_2O_8)]\cdot 3H_2O$
$M_r = 516.16$
Orthorhombic, $P2_12_12_1$
$a = 8.7800$ (18) Å
$b = 11.443$ (2) Å
$c = 16.573$ (3) Å
$V = 1665.1$ (6) Å^3
$Z = 4$
$F(000) = 1036$
$D_x = 2.059$ Mg m^{-3}
Synchrotron radiation, $\lambda = 0.610$ Å
Cell parameters from 33074 reflections
$\theta = 0.4$–$33.7°$
$\mu = 1.25$ mm^{-1}
$T = 260$ K
Block, purple
0.17 × 0.13 × 0.07 mm

Data collection

ADSC Q210 CCD area detector diffractometer
Radiation source: PLSII 2D bending magnet
ω scan
Absorption correction: empirical (using intensity measurements) (*HKL3000sm Scalepack*; Otwinowski & Minor, 1997)
$T_{min} = 0.843$, $T_{max} = 1.000$
14937 measured reflections
4807 independent reflections
4738 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.1°$
$h = -12\rightarrow12$
$k = -16\rightarrow16$
$l = -23\rightarrow23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.051$
$S = 1.07$
4807 reflections
253 parameters
9 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0272P)^2 + 0.5713P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.65$ e Å^{-3}

Absolute structure: Flack *x* determined using 2027 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter: −0.008 (6)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cr1	0.24189 (3)	0.37102 (2)	0.24502 (2)	0.00967 (6)
O1	0.12226 (18)	0.25175 (12)	0.30121 (9)	0.0175 (3)
O2	−0.0055 (2)	0.21547 (15)	0.41423 (11)	0.0251 (3)
O3	0.08051 (18)	0.39909 (14)	0.16628 (9)	0.0186 (3)
O4	−0.16003 (19)	0.45764 (18)	0.15293 (11)	0.0282 (4)
O5	0.40372 (17)	0.35453 (13)	0.32531 (9)	0.0177 (3)
O6	0.63816 (19)	0.40881 (18)	0.35973 (11)	0.0286 (4)
O7	0.35275 (18)	0.27005 (12)	0.16811 (9)	0.0184 (3)
O8	0.5114 (3)	0.27410 (18)	0.06396 (14)	0.0398 (5)
N1	0.11336 (19)	0.48076 (14)	0.31737 (10)	0.0116 (3)
N2	0.38257 (19)	0.50019 (14)	0.19855 (11)	0.0132 (3)
C1	0.0637 (2)	0.28284 (17)	0.36905 (12)	0.0150 (3)
C2	0.0876 (2)	0.41046 (17)	0.39202 (12)	0.0165 (3)
H21	−0.001299	0.439533	0.420408	0.020*
H22	0.174899	0.417403	0.427556	0.020*
C3	−0.0428 (2)	0.45009 (19)	0.19226 (12)	0.0163 (3)
C4	−0.0348 (2)	0.50289 (17)	0.27589 (13)	0.0155 (3)
H41	−0.050662	0.586572	0.271860	0.019*
H42	−0.116537	0.470866	0.308456	0.019*
C5	0.5249 (2)	0.41833 (17)	0.31713 (12)	0.0159 (3)
C6	0.5206 (2)	0.50999 (17)	0.25100 (15)	0.0177 (3)
H61	0.522708	0.587001	0.275442	0.021*
H62	0.611006	0.502164	0.217770	0.021*
C7	0.4341 (3)	0.32390 (19)	0.11558 (13)	0.0192 (4)
C8	0.4251 (3)	0.45637 (18)	0.11673 (13)	0.0204 (4)
H81	0.522910	0.488802	0.101292	0.024*
H82	0.349954	0.482279	0.077731	0.024*
C9	0.1955 (2)	0.59037 (17)	0.33877 (13)	0.0183 (4)
H91	0.286801	0.569504	0.368393	0.022*
H92	0.131325	0.635424	0.374865	0.022*
C10	0.2416 (3)	0.66904 (17)	0.26797 (15)	0.0214 (4)
H10	0.153858	0.716501	0.254216	0.026*
H10B	0.319794	0.721905	0.287443	0.026*
C11	0.3006 (2)	0.61414 (17)	0.18957 (14)	0.0189 (4)
H11	0.214770	0.602452	0.153619	0.023*

H11B	0.368853	0.669379	0.163804	0.023*
Ag1	0.66176 (2)	0.43997 (2)	0.51158 (2)	0.02983 (6)
O9	0.4276 (2)	0.55599 (18)	0.50044 (10)	0.0323 (4)
H1O1	0.426 (5)	0.571 (3)	0.5503 (8)	0.039*
H2O1	0.455 (4)	0.6206 (19)	0.4800 (19)	0.039*
O10	0.8668 (3)	0.31407 (19)	0.54800 (12)	0.0345 (4)
H1O2	0.869 (5)	0.264 (3)	0.5862 (15)	0.041*
H2O2	0.899 (4)	0.276 (3)	0.5075 (14)	0.041*
O11	0.8203 (3)	0.60117 (17)	0.44997 (13)	0.0333 (4)
H1O3	0.758 (4)	0.637 (3)	0.421 (2)	0.040*
H2O3	0.853 (4)	0.649 (3)	0.4838 (18)	0.040*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.00884 (12)	0.01014 (11)	0.01002 (11)	−0.00089 (9)	0.00008 (9)	0.00054 (9)
O1	0.0211 (7)	0.0136 (6)	0.0177 (6)	−0.0061 (5)	0.0050 (5)	−0.0013 (5)
O2	0.0265 (8)	0.0234 (7)	0.0253 (8)	−0.0017 (6)	0.0088 (7)	0.0093 (6)
O3	0.0137 (6)	0.0295 (7)	0.0127 (6)	0.0031 (5)	−0.0030 (5)	−0.0026 (5)
O4	0.0137 (6)	0.0496 (10)	0.0212 (7)	0.0015 (7)	−0.0065 (6)	0.0017 (7)
O5	0.0133 (6)	0.0219 (7)	0.0180 (6)	−0.0021 (5)	−0.0042 (5)	0.0052 (5)
O6	0.0154 (7)	0.0474 (10)	0.0230 (8)	−0.0030 (6)	−0.0080 (6)	0.0007 (7)
O7	0.0211 (7)	0.0143 (6)	0.0199 (7)	0.0007 (5)	0.0072 (6)	−0.0012 (5)
O8	0.0520 (13)	0.0286 (9)	0.0388 (11)	−0.0050 (9)	0.0299 (10)	−0.0106 (8)
N1	0.0116 (6)	0.0113 (6)	0.0119 (6)	−0.0005 (5)	−0.0009 (5)	−0.0003 (5)
N2	0.0117 (7)	0.0118 (7)	0.0160 (7)	−0.0009 (5)	0.0001 (5)	0.0022 (5)
C1	0.0140 (8)	0.0163 (8)	0.0147 (8)	−0.0005 (6)	0.0008 (6)	0.0039 (6)
C2	0.0206 (9)	0.0182 (8)	0.0108 (7)	0.0008 (7)	0.0019 (7)	0.0011 (6)
C3	0.0114 (8)	0.0233 (8)	0.0144 (8)	−0.0021 (7)	−0.0017 (6)	0.0044 (7)
C4	0.0110 (7)	0.0192 (9)	0.0163 (8)	0.0017 (6)	−0.0018 (6)	0.0003 (6)
C5	0.0110 (7)	0.0210 (8)	0.0157 (8)	−0.0002 (6)	−0.0018 (6)	−0.0034 (7)
C6	0.0108 (7)	0.0167 (7)	0.0258 (9)	−0.0031 (6)	−0.0016 (7)	0.0007 (7)
C7	0.0201 (9)	0.0205 (8)	0.0169 (9)	−0.0021 (7)	0.0049 (7)	−0.0025 (7)
C8	0.0253 (10)	0.0191 (9)	0.0167 (8)	−0.0036 (7)	0.0074 (8)	0.0019 (7)
C9	0.0203 (9)	0.0147 (7)	0.0200 (9)	−0.0028 (6)	−0.0008 (7)	−0.0056 (6)
C10	0.0201 (9)	0.0121 (7)	0.0319 (10)	−0.0014 (7)	0.0024 (8)	0.0002 (7)
C11	0.0215 (9)	0.0121 (8)	0.0230 (9)	0.0017 (6)	0.0009 (7)	0.0052 (7)
Ag1	0.03046 (10)	0.03290 (9)	0.02614 (9)	0.00309 (7)	0.00069 (7)	0.00446 (7)
O9	0.0440 (10)	0.0339 (8)	0.0191 (8)	−0.0016 (8)	−0.0045 (7)	0.0039 (7)
O10	0.0401 (11)	0.0378 (10)	0.0257 (9)	0.0108 (8)	0.0103 (8)	0.0094 (7)
O11	0.0376 (10)	0.0302 (8)	0.0320 (9)	0.0014 (8)	−0.0099 (8)	−0.0045 (7)

Geometric parameters (Å, º)

Cr1—O3	1.9530 (15)	C4—H41	0.9700
Cr1—O5	1.9558 (15)	C4—H42	0.9700
Cr1—O1	1.9578 (14)	C5—C6	1.517 (3)
Cr1—O7	1.9766 (15)	C6—H61	0.9700

Cr1—N1	2.0708 (17)	C6—H62	0.9700
Cr1—N2	2.0745 (16)	C7—C8	1.518 (3)
O1—C1	1.287 (2)	C8—H81	0.9700
O2—C1	1.234 (2)	C8—H82	0.9700
O3—C3	1.303 (2)	C9—C10	1.533 (3)
O4—C3	1.222 (2)	C9—H91	0.9700
O5—C5	1.298 (2)	C9—H92	0.9700
O6—C5	1.224 (3)	C10—C11	1.533 (3)
O6—Ag1	2.5501 (19)	C10—H10	0.9700
O7—C7	1.284 (3)	C10—H10B	0.9700
O8—C7	1.232 (3)	C11—H11	0.9700
N1—C9	1.490 (2)	C11—H11B	0.9700
N1—C4	1.493 (2)	Ag1—O10	2.383 (2)
N1—C2	1.493 (2)	Ag1—O9	2.455 (2)
N2—C8	1.493 (3)	Ag1—O11	2.526 (2)
N2—C6	1.496 (3)	O9—H1O1	0.844 (13)
N2—C11	1.497 (3)	O9—H2O1	0.848 (13)
C1—C2	1.524 (3)	O10—H1O2	0.854 (13)
C2—H21	0.9700	O10—H2O2	0.847 (13)
C2—H22	0.9700	O11—H1O3	0.839 (13)
C3—C4	1.514 (3)	O11—H2O3	0.834 (13)

O3—Cr1—O5	176.04 (7)	O6—C5—C6	119.87 (19)
O3—Cr1—O1	92.48 (7)	O5—C5—C6	116.36 (17)
O5—Cr1—O1	89.94 (7)	N2—C6—C5	112.83 (16)
O3—Cr1—O7	91.29 (7)	N2—C6—H61	109.0
O5—Cr1—O7	91.41 (7)	C5—C6—H61	109.0
O1—Cr1—O7	99.41 (6)	N2—C6—H62	109.0
O3—Cr1—N1	83.79 (7)	C5—C6—H62	109.0
O5—Cr1—N1	93.47 (7)	H61—C6—H62	107.8
O1—Cr1—N1	81.66 (6)	O8—C7—O7	123.7 (2)
O7—Cr1—N1	175.01 (7)	O8—C7—C8	120.0 (2)
O3—Cr1—N2	93.82 (7)	O7—C7—C8	116.21 (18)
O5—Cr1—N2	83.61 (7)	N2—C8—C7	111.09 (16)
O1—Cr1—N2	173.07 (7)	N2—C8—H81	109.4
O7—Cr1—N2	83.33 (7)	C7—C8—H81	109.4
N1—Cr1—N2	96.16 (7)	N2—C8—H82	109.4
C1—O1—Cr1	115.94 (12)	C7—C8—H82	109.4
C3—O3—Cr1	117.10 (13)	H81—C8—H82	108.0
C5—O5—Cr1	118.06 (13)	N1—C9—C10	116.11 (17)
C5—O6—Ag1	128.51 (15)	N1—C9—H91	108.3
C7—O7—Cr1	115.52 (13)	C10—C9—H91	108.3
C9—N1—C4	112.86 (15)	N1—C9—H92	108.3
C9—N1—C2	109.26 (16)	C10—C9—H92	108.3
C4—N1—C2	109.93 (16)	H91—C9—H92	107.4
C9—N1—Cr1	112.63 (12)	C11—C10—C9	119.81 (16)
C4—N1—Cr1	108.12 (12)	C11—C10—H10	107.4
C2—N1—Cr1	103.64 (11)	C9—C10—H10	107.4

C8—N2—C6	110.51 (16)	C11—C10—H10B	107.4
C8—N2—C11	108.81 (16)	C9—C10—H10B	107.4
C6—N2—C11	112.46 (16)	H10—C10—H10B	106.9
C8—N2—Cr1	104.29 (12)	N2—C11—C10	115.79 (17)
C6—N2—Cr1	108.67 (12)	N2—C11—H11	108.3
C11—N2—Cr1	111.80 (12)	C10—C11—H11	108.3
O2—C1—O1	123.65 (19)	N2—C11—H11B	108.3
O2—C1—C2	120.98 (19)	C10—C11—H11B	108.3
O1—C1—C2	115.36 (16)	H11—C11—H11B	107.4
N1—C2—C1	109.28 (15)	O10—Ag1—O9	168.15 (6)
N1—C2—H21	109.8	O10—Ag1—O11	97.34 (7)
C1—C2—H21	109.8	O9—Ag1—O11	92.08 (7)
N1—C2—H22	109.8	O10—Ag1—O6	103.11 (7)
C1—C2—H22	109.8	O9—Ag1—O6	86.18 (6)
H21—C2—H22	108.3	O11—Ag1—O6	75.40 (6)
O4—C3—O3	123.7 (2)	Ag1—O9—H1O1	93 (3)
O4—C3—C4	119.96 (19)	Ag1—O9—H2O1	105 (3)
O3—C3—C4	116.29 (16)	H1O1—O9—H2O1	103 (3)
N1—C4—C3	113.23 (15)	Ag1—O10—H1O2	128 (3)
N1—C4—H41	108.9	Ag1—O10—H2O2	111 (3)
C3—C4—H41	108.9	H1O2—O10—H2O2	104 (3)
N1—C4—H42	108.9	Ag1—O11—H1O3	103 (3)
C3—C4—H42	108.9	Ag1—O11—H2O3	113 (3)
H41—C4—H42	107.7	H1O3—O11—H2O3	107 (3)
O6—C5—O5	123.8 (2)		

Cr1—O1—C1—O2	−174.74 (17)	C11—N2—C6—C5	129.15 (18)
Cr1—O1—C1—C2	4.1 (2)	Cr1—N2—C6—C5	4.8 (2)
C9—N1—C2—C1	−157.68 (16)	O6—C5—C6—N2	173.04 (19)
C4—N1—C2—C1	77.96 (19)	O5—C5—C6—N2	−7.8 (3)
Cr1—N1—C2—C1	−37.41 (17)	Cr1—O7—C7—O8	179.2 (2)
O2—C1—C2—N1	−156.79 (19)	Cr1—O7—C7—C8	−3.9 (3)
O1—C1—C2—N1	24.3 (2)	C6—N2—C8—C7	84.3 (2)
Cr1—O3—C3—O4	−168.99 (18)	C11—N2—C8—C7	−151.72 (18)
Cr1—O3—C3—C4	11.7 (2)	Cr1—N2—C8—C7	−32.3 (2)
C9—N1—C4—C3	120.00 (18)	O8—C7—C8—N2	−157.0 (2)
C2—N1—C4—C3	−117.75 (18)	O7—C7—C8—N2	25.9 (3)
Cr1—N1—C4—C3	−5.26 (18)	C4—N1—C9—C10	−61.7 (2)
O4—C3—C4—N1	177.0 (2)	C2—N1—C9—C10	175.70 (17)
O3—C3—C4—N1	−3.6 (2)	Cr1—N1—C9—C10	61.1 (2)
Ag1—O6—C5—O5	−62.2 (3)	N1—C9—C10—C11	−39.4 (3)
Ag1—O6—C5—C6	117.0 (2)	C8—N2—C11—C10	177.20 (17)
Cr1—O5—C5—O6	−173.92 (17)	C6—N2—C11—C10	−60.0 (2)
Cr1—O5—C5—C6	6.9 (2)	Cr1—N2—C11—C10	62.6 (2)
C8—N2—C6—C5	−109.04 (19)	C9—C10—C11—N2	−30.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9—H1*O*1···O3^{i}	0.84 (1)	1.95 (1)	2.797 (2)	178 (3)
O9—H2*O*1···O8^{ii}	0.85 (1)	1.93 (1)	2.767 (3)	172 (4)
O10—H1*O*2···O5^{iii}	0.85 (1)	2.02 (1)	2.870 (2)	173 (4)
O10—H2*O*2···O2^{iv}	0.85 (1)	1.89 (1)	2.729 (3)	170 (4)
O11—H1*O*3···O7^{ii}	0.84 (1)	2.33 (2)	3.142 (3)	163 (4)
O11—H2*O*3···O8^{v}	0.83 (1)	1.99 (2)	2.791 (3)	161 (3)

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