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Sonochemical synthesis and crystal structure of dimethylammonium bis[3-carboxy-2-(dimethylamino)propanoato-κ^2N,O^1]chloridochromium(II) monohydrate

Meriem Saidi,[a] Michel Giorgi[b] and Leila Boukli-hacene[a]*

[a]Laboratoire de Chimie Inorganique et Environnement, Faculty of Science, University of Tlemcen, Tlemcen - 13000, Algeria, and [b]Aix-Marseille University, Spectropole, Campus St. Jerome, 52 av. Escadrille Normandie Niemen, 13013 Marseille, France. *Correspondence e-mail: leila.bouklihacene@univ-tlemcen.dz

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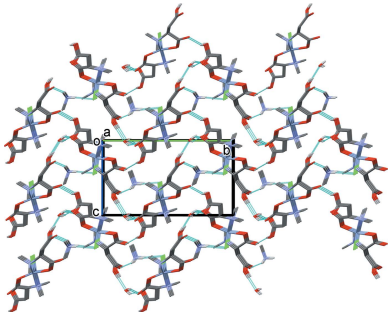
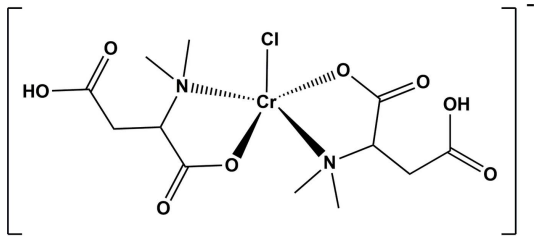
The title complex, $[(CH_3)_2NH_2][Cr(C_6H_{10}NO_4)_2Cl]\cdot H_2O$, was synthesized sonochemically. The complex anion consists of a chromium(II) ion ligated by two 3-carboxy-2-(dimethylamino)propanoate anions. They coordinate in a bidentate manner, with a carboxylate oxygen atom and the nitrogen atom *cis* to each other in the equatorial plane, while the apical position is occupied by a Cl^- ion. Hence, the chromium(II) ion is five-coordinate with a quasi-ideal square-pyramidal geometry; τ_5 parameter = 0.01. The complex crystallizes as a monohydrate and in the crystal, the water molecule and the dimethylammonium counter-ion link the complex cations *via* N—H···O, N—H···Cl, O_{water}—H···O, O—H···O_{water} and O—H···O hydrogen bonds, forming a supramolecular framework. There are also a number of C—H···O hydrogen bonds present that reinforce the framework structure. The crystal studied was refined as a racemic twin.

1. Chemical context

Fumaric acid, also known as *trans*-butenedioic acid, boletic acid, lichenic acid or allomaleic acid, occurs naturally in many plants and is named after *Fumaria officinalis*, a climbing annual plant (Felthouse *et al.*, 2001). Besides being 'practically non-toxic' (European Commission, 2003), it is used as an acidity regulator in the food industry (Linstrom & Mallard, 1998), in medicine (Gold *et al.*, 2012), and as a raw material in the manufacture of unsaturated polyester resins (Duty & Liu, 1980).

Since the beginning of the 21st century, fumaric acid has been used to synthesize one of the first metal–organic frameworks for commercial applications (Al-MOF: A520), presenting remarkable adsorption and mechanical properties, combined with low toxicity (Gaab *et al.*, 2012). In this context, the novel title compound was obtained during an attempt to synthesize a Cr–Fum MOF.





. $[(CH_3)_2NH_2]^+$. H_2O

The reaction of fumaric acid and chromium(II)acetate dihydrate in the presence of dimethylamine hydrochloride resulted in the hydroamination of fumaric acid to form *N*,*N*-dimethylaspartic acid, which coordinates in a bidentate fashion to the chromium(II) ion.

2. Structural commentary

The molecular structure of the title complex anion is illustrated in Fig. 1. The chromium(II) ion, atom Cr1, is coordinated to two 3-carboxy-2-(dimethylamino) propanoate anions in a bidentate manner with a carboxylate oxygen atom O1 and the nitrogen N1 *cis* to each other for one ligand and for the other ligand atoms O5 and N2 are *cis* to each other. The chloride anion, Cl1, occupies the apical position. The five-coordinate chromium ion is displaced by 0.3469 (7) Å from the mean plane through atoms O1, N1, O5 and N2. The equatorial Cu—O bond lengths are Cr1—O1 = 1.960 (5) Å and Cr1—O5 = 1.954 (5) Å, while the equatorial Cu—N bond lengths are slightly longer *viz*. Cr1—N1 = 2.025 (5) Å and Cr1—N2 = 2.030 (5) Å. The axial Cr1—Cl1 bond length is 2.5301 (16) Å. The C—C, C—O, and C—N bond lengths of the ligands are close to those reported for similar compounds (Zheng *et al.*, 2003; Devereux *et al.*, 2000; Kim *et al.*, 2002). The *cisoid* and *transoid* bond angles vary from 83.62 (19) to 100.88 (16)° and from 159.6 (2) to 160.3 (2)°, respectively. This leads to a quasi-ideal square-pyramidal geometry for atom Cr1 with a τ_5 parameter of 0.01 (τ_5 = 0 for an ideal square-pyramidal geometry and 1 for an ideal trigonal–bipyramidal geometry; Addison *et al.*, 1984). An intramolecular C6—H6*C*···O5 hydrogen bond (Table 1) occurs.

3. Supramolecular features

The crystal structure is stabilized by an extensive array of hydrogen bonds, forming a supramolecular framework (Fig. 2 and Table 1). Beyond metal coordination, the ligand has potential sites for hydrogen bonding. Ten of the thirteen heteroatoms are involved in strong and moderate hydrogen bonds (Fig. 2 and Table 1). The complex crystallizes as a monohydrate and in the crystal, the water molecule and the dimethylammonium counter-ion link the complex cations *via* N—H···O, N—H···Cl, O_{water}—H···O, O—H···O_{water} and O—H···O hydrogen bonds, forming a supramolecular framework. There are also a number of C—H···O hydrogen bonds present that reinforce the framework structure.

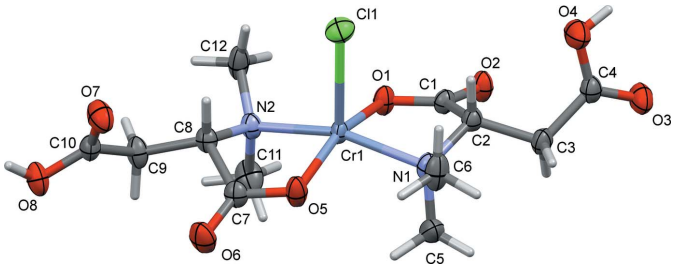


Figure 1
The molecular structure of the title complex anion, with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. For clarity, the dimethylammonium counter-ion and the water molecule of crystallization have been omitted.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*O*···O1*W*	0.82	1.77	2.591 (8)	180
O8—H8*O*···O2^{i}	0.82	1.91	2.585 (7)	139
N3—H3*C*···Cl1^{ii}	0.89	2.24	3.121 (7)	172
N3—H3*D*···O3^{iii}	0.89	1.94	2.763 (9)	153
O1*W*—H1*WA*···O7^{ii}	0.86 (3)	2.17 (8)	2.895 (8)	142 (12)
O1*W*—H1*WB*···O6^{iv}	0.86 (3)	2.18 (3)	3.006 (9)	160 (7)
C6—H6*B*···O8^{v}	0.96	2.51	3.351 (9)	146
C6—H6*C*···O5	0.96	2.46	3.038 (9)	119
C12—H12*C*···O6^{vi}	0.96	2.52	3.193 (10)	127
C13—H13*B*···O8^{vi}	0.96	2.56	3.451 (12)	154

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

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.40, update February 2019; Groom *et al.*, 2016) indicated that there are no reports of chromium complexes of fumaric acid and no reports of the structure of the title ligand, *N*,*N*-dimethylaspartic acid. There is only one report of a complex containing a similar ligand, *viz*. [(*R*,*S*)-dimethyl 3-(diphenylphosphino)-*N*,*N*-dimethylaspartate]dichloropalladium(II) [CASTIB; Chen *et al.*, 2012]. This chiral *P*,*N*-ligand was synthesized by hydrophosphination using diphenylphosphine followed by hydroamination with a secondary amine.

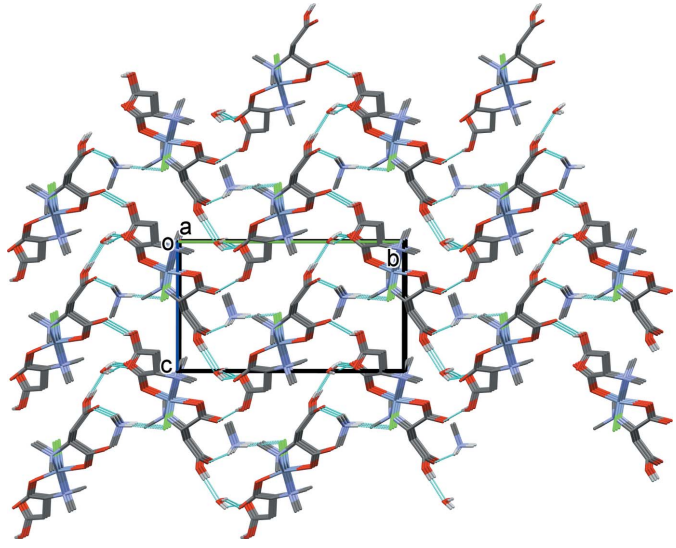


Figure 2
A view along the *a* axis of the crystal packing of the title complex. The hydrogen bonds (Table 1) are shown as dashed lines and, for clarity, all the C-bound H atoms have been omitted.

Table 2
Experimental details.

Crystal data	
Chemical formula	$(C_2H_8N)[Cr(C_6H_{10}NO_4)_2Cl]\cdot H_2O$
M_r	471.86
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	298
a, b, c (Å)	8.2246 (2), 15.1419 (4), 8.6851 (2)
β (°)	93.339 (2)
V (Å^3)	1079.77 (5)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	5.94
Crystal size (mm)	0.16 × 0.10 × 0.06
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Cu at home/near, AtlasS2
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)
T_{min}, T_{max}	0.917, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11333, 3930, 3864
R_{int}	0.039
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.605
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.053, 0.146, 1.08
No. of reflections	3930
No. of parameters	268
No. of restraints	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.14, −0.36
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.422 (11)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009), *OLEX2* (Dolomanov *et al.*, 2009), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

5. Synthesis and crystallization

A mixture of fumaric acid (25 mg, 0.22 mmol) and dimethylamine hydrochloride (0.09 ml) dissolved in 20 ml methanol was stirred for 1 h. Chromium(II) acetate dihydrate [$Cr_2(OAc)_4\cdot 2H_2O$; 25.2 mg, 0.11 mmol] in 10 ml of water was added with magnetic stirring for a further 30 min. The mixture was then put in an ultrasonic bath (353 K, 45 KHz, 90 W) for 2h. The solution was then left to evaporate slowly and blue prismatic crystals were collected after two months.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal was refined as a racemic twin [BASF = 0.422 (11)]. The water H atoms were located in a difference-Fourier map and refined with a distance restraint of O—H = 0.85 (2) Å with U_{iso}(H) = 1.5U_{eq}(O). All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms: O—H = 0.82 Å, N—H = 0.89 Å, C—H = 0.96–0.99 Å with U_{iso}(H) = 1.5U_{eq}(O-hydroxyl, C-methyl) and 1.2U_{eq}(N, C) for other H atoms.

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Sonochemical synthesis and crystal structure of dimethylammonium bis[3-carboxy-2-(dimethylamino)propanoato-κ^2N,O^1]chloridochromium(II) monohydrate

Meriem Saidi, Michel Giorgi and Leila Boukli-hacene

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Dimethylammonium bis[3-carboxy-2-(dimethylamino)propanoato-κ^2N,O^1]\ chloridochromium(II) monohydrate

Crystal data

$(C_2H_8N)[Cr(C_6H_{10}NO_4)_2Cl]\cdot H_2O$
$M_r = 471.86$
Monoclinic, $P2_1$
$a = 8.2246$ (2) Å
$b = 15.1419$ (4) Å
$c = 8.6851$ (2) Å
$\beta = 93.339$ (2)°
$V = 1079.77$ (5) Å^3
$Z = 2$
$F(000) = 496$
$D_x = 1.451$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 7960 reflections
$\theta = 5.4$–$68.8°$
$\mu = 5.94$ mm^{-1}
$T = 298$ K
Prism, blue
0.16 × 0.10 × 0.06 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Cu at home/near, AtlasS2 diffractometer
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 5.3048 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
$T_{min} = 0.917$, $T_{max} = 1.000$
11333 measured reflections
3930 independent reflections
3864 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 69.0°$, $\theta_{min} = 5.1°$
$h = -8\rightarrow9$
$k = -18\rightarrow18$
$l = -10\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.146$
$S = 1.08$
3930 reflections
268 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1071P)^2 + 0.4201P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.14$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.422 (11)

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.

Refinement. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cr1	0.77728 (9)	0.46008 (6)	0.77478 (8)	0.0331 (3)
Cl1	0.96891 (19)	0.44269 (11)	0.55923 (19)	0.0567 (4)
O1	0.7158 (5)	0.5842 (3)	0.7439 (5)	0.0467 (10)
O2	0.5397 (6)	0.6689 (3)	0.6095 (6)	0.0507 (10)
O3	0.2468 (7)	0.6241 (5)	0.2902 (6)	0.0647 (16)
O4	0.4927 (7)	0.5814 (5)	0.2465 (7)	0.0727 (16)
H4O	0.473620	0.609797	0.167158	0.109*
O5	0.7816 (6)	0.3408 (3)	0.8608 (6)	0.0568 (12)
O6	0.8881 (8)	0.2659 (4)	1.0581 (7)	0.0679 (14)
O7	1.2888 (7)	0.2838 (4)	1.0339 (6)	0.0686 (15)
O8	1.2813 (7)	0.2989 (4)	1.2885 (6)	0.0578 (14)
H8O	1.366024	0.270333	1.290499	0.087*
N1	0.5641 (6)	0.4365 (3)	0.6532 (6)	0.0439 (11)
N2	0.9374 (6)	0.4869 (3)	0.9555 (6)	0.0422 (11)
C1	0.6020 (7)	0.5955 (4)	0.6425 (7)	0.0388 (12)
C2	0.5452 (7)	0.5140 (4)	0.5487 (6)	0.0395 (12)
H2	0.622057	0.505921	0.467737	0.047*
C3	0.3768 (8)	0.5244 (5)	0.4683 (7)	0.0469 (13)
H3A	0.337458	0.466321	0.437155	0.056*
H3B	0.303802	0.547195	0.542577	0.056*
C4	0.3661 (8)	0.5834 (4)	0.3295 (7)	0.0458 (13)
C5	0.4376 (8)	0.4347 (6)	0.7677 (9)	0.062 (2)
H8A	0.470785	0.395257	0.850250	0.093*
H8B	0.336623	0.414570	0.718912	0.093*
H8C	0.423522	0.492985	0.808217	0.093*
C6	0.5626 (11)	0.3524 (5)	0.5675 (11)	0.067 (2)
H6A	0.635292	0.356419	0.485227	0.100*
H6B	0.454227	0.340600	0.525437	0.100*
H6C	0.597207	0.305432	0.636040	0.100*
C7	0.8884 (8)	0.3294 (4)	0.9699 (7)	0.0438 (12)
C8	1.0189 (7)	0.4011 (4)	0.9867 (7)	0.0395 (12)
H8	1.093246	0.391531	0.904066	0.047*
C9	1.1207 (9)	0.3964 (4)	1.1380 (8)	0.0499 (14)

H9A	1.181580	0.450948	1.151865	0.060*
H9B	1.048333	0.391704	1.221919	0.060*
C10	1.2384 (7)	0.3197 (4)	1.1473 (7)	0.0436 (12)
C11	0.8385 (11)	0.5139 (6)	1.0840 (9)	0.067 (2)
H11A	0.790995	0.570782	1.061941	0.100*
H11B	0.906629	0.517222	1.177456	0.100*
H19C	0.753661	0.471417	1.096277	0.100*
C12	1.0545 (10)	0.5576 (5)	0.9208 (11)	0.067 (2)
H12A	1.108472	0.542490	0.829352	0.100*
H12B	1.133630	0.563742	1.005790	0.100*
H12C	0.997048	0.612331	0.904808	0.100*
N3	1.0276 (9)	0.7370 (4)	0.4178 (8)	0.0607 (15)
H3C	1.036403	0.795574	0.417595	0.073*
H3D	1.101606	0.715613	0.356926	0.073*
C13	1.0657 (14)	0.7039 (7)	0.5796 (11)	0.079 (3)
H13A	1.055159	0.640779	0.581505	0.119*
H13B	0.991100	0.729742	0.647664	0.119*
H13C	1.175078	0.720115	0.612551	0.119*
C14	0.8635 (12)	0.7125 (7)	0.3526 (12)	0.078 (2)
H14A	0.854076	0.727475	0.245084	0.118*
H14B	0.782581	0.743983	0.406017	0.118*
H14C	0.847500	0.650115	0.364519	0.118*
O1W	0.4309 (7)	0.6707 (5)	−0.0045 (7)	0.0703 (15)
H1WA	0.492 (14)	0.704 (8)	−0.056 (13)	0.105*
H1WB	0.341 (10)	0.699 (8)	0.004 (8)	0.105*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.0310 (4)	0.0267 (4)	0.0401 (4)	−0.0011 (3)	−0.0118 (3)	0.0011 (3)
Cl1	0.0540 (8)	0.0532 (10)	0.0636 (8)	−0.0009 (6)	0.0104 (6)	−0.0049 (6)
O1	0.049 (2)	0.033 (2)	0.056 (2)	−0.0003 (16)	−0.016 (2)	−0.0034 (17)
O2	0.053 (2)	0.033 (2)	0.064 (3)	0.0047 (18)	−0.017 (2)	0.0016 (19)
O3	0.057 (3)	0.079 (4)	0.057 (3)	0.025 (3)	−0.001 (2)	0.010 (3)
O4	0.058 (3)	0.094 (5)	0.067 (3)	0.023 (3)	0.008 (2)	0.022 (3)
O5	0.060 (3)	0.040 (2)	0.067 (3)	−0.009 (2)	−0.025 (2)	0.009 (2)
O6	0.082 (4)	0.047 (3)	0.072 (3)	−0.004 (2)	−0.015 (3)	0.019 (2)
O7	0.073 (3)	0.077 (4)	0.056 (3)	0.029 (3)	0.003 (2)	0.005 (3)
O8	0.071 (4)	0.048 (3)	0.052 (3)	0.016 (2)	−0.016 (2)	0.000 (2)
N1	0.040 (2)	0.035 (3)	0.055 (3)	0.0016 (18)	−0.011 (2)	0.002 (2)
N2	0.046 (3)	0.034 (3)	0.045 (2)	−0.0006 (19)	−0.009 (2)	0.0015 (18)
C1	0.038 (3)	0.033 (3)	0.045 (3)	−0.003 (2)	−0.005 (2)	−0.002 (2)
C2	0.037 (3)	0.038 (3)	0.043 (3)	−0.003 (2)	−0.006 (2)	0.000 (2)
C3	0.040 (3)	0.046 (3)	0.054 (3)	−0.007 (2)	−0.011 (2)	0.002 (3)
C4	0.046 (3)	0.041 (3)	0.049 (3)	−0.001 (3)	−0.010 (2)	−0.001 (3)
C5	0.044 (3)	0.071 (5)	0.071 (4)	−0.008 (3)	−0.003 (3)	0.026 (4)
C6	0.071 (5)	0.035 (4)	0.090 (6)	−0.001 (3)	−0.035 (4)	−0.008 (3)
C7	0.051 (3)	0.029 (3)	0.051 (3)	0.002 (2)	−0.005 (3)	−0.001 (2)

C8	0.044 (3)	0.030 (3)	0.044 (3)	0.004 (2)	−0.004 (2)	−0.002 (2)
C9	0.061 (4)	0.035 (3)	0.051 (3)	0.008 (3)	−0.016 (3)	−0.004 (2)
C10	0.042 (3)	0.039 (3)	0.048 (3)	0.001 (2)	−0.010 (2)	0.002 (2)
C11	0.080 (5)	0.061 (5)	0.059 (4)	0.027 (4)	0.001 (4)	−0.011 (3)
C12	0.063 (4)	0.046 (4)	0.086 (5)	−0.013 (3)	−0.039 (4)	0.013 (4)
N3	0.069 (4)	0.047 (3)	0.066 (3)	0.001 (3)	0.008 (3)	0.000 (3)
C13	0.101 (7)	0.066 (6)	0.072 (5)	0.011 (5)	0.010 (5)	0.003 (4)
C14	0.072 (5)	0.071 (6)	0.093 (6)	0.000 (4)	0.008 (5)	−0.014 (5)
O1W	0.063 (3)	0.070 (4)	0.080 (4)	0.001 (3)	0.013 (3)	0.019 (3)

Geometric parameters (Å, º)

Cr1—O5	1.954 (5)	C5—H8C	0.9600
Cr1—O1	1.960 (5)	C6—H6A	0.9600
Cr1—N1	2.025 (5)	C6—H6B	0.9600
Cr1—N2	2.030 (5)	C6—H6C	0.9600
Cr1—Cl1	2.5301 (16)	C7—C8	1.527 (8)
O1—C1	1.259 (7)	C8—C9	1.518 (9)
O2—C1	1.250 (8)	C8—H8	0.9800
O3—C4	1.191 (8)	C9—C10	1.511 (9)
O4—C4	1.300 (9)	C9—H9A	0.9700
O4—H4O	0.8200	C9—H9B	0.9700
O5—C7	1.266 (8)	C11—H11A	0.9600
O6—C7	1.230 (8)	C11—H11B	0.9600
O7—C10	1.220 (9)	C11—H19C	0.9600
O8—C10	1.294 (8)	C12—H12A	0.9600
O8—H8O	0.8200	C12—H12B	0.9600
N1—C6	1.475 (9)	C12—H12C	0.9600
N1—C5	1.481 (9)	N3—C14	1.480 (12)
N1—C2	1.485 (8)	N3—C13	1.507 (11)
N2—C11	1.477 (9)	N3—H3C	0.8900
N2—C8	1.480 (7)	N3—H3D	0.8900
N2—C12	1.483 (9)	C13—H13A	0.9600
C1—C2	1.536 (8)	C13—H13B	0.9600
C2—C3	1.523 (8)	C13—H13C	0.9600
C2—H2	0.9800	C14—H14A	0.9600
C3—C4	1.499 (9)	C14—H14B	0.9600
C3—H3A	0.9700	C14—H14C	0.9600
C3—H3B	0.9700	O1W—H1WA	0.86 (3)
C5—H8A	0.9600	O1W—H1WB	0.86 (3)
C5—H8B	0.9600		

O5—Cr1—O1	159.6 (2)	N1—C6—H6C	109.5
O5—Cr1—N1	91.9 (2)	H6A—C6—H6C	109.5
O1—Cr1—N1	83.62 (19)	H6B—C6—H6C	109.5
O5—Cr1—N2	83.8 (2)	O6—C7—O5	123.1 (6)
O1—Cr1—N2	93.71 (19)	O6—C7—C8	121.5 (6)
N1—Cr1—N2	160.3 (2)	O5—C7—C8	115.3 (5)

O5—Cr1—Cl1	100.87 (19)	N2—C8—C9	114.9 (5)
O1—Cr1—Cl1	99.51 (15)	N2—C8—C7	107.3 (5)
N1—Cr1—Cl1	98.83 (16)	C9—C8—C7	113.5 (5)
N2—Cr1—Cl1	100.88 (16)	N2—C8—H8	106.9
C1—O1—Cr1	113.7 (4)	C9—C8—H8	106.9
C4—O4—H4O	109.5	C7—C8—H8	106.9
C7—O5—Cr1	114.1 (4)	C10—C9—C8	113.7 (5)
C10—O8—H8O	109.5	C10—C9—H9A	108.8
C6—N1—C5	109.7 (6)	C8—C9—H9A	108.8
C6—N1—C2	112.1 (6)	C10—C9—H9B	108.8
C5—N1—C2	111.9 (5)	C8—C9—H9B	108.8
C6—N1—Cr1	113.4 (4)	H9A—C9—H9B	107.7
C5—N1—Cr1	105.9 (4)	O7—C10—O8	124.7 (6)
C2—N1—Cr1	103.7 (3)	O7—C10—C9	123.1 (6)
C11—N2—C8	111.6 (5)	O8—C10—C9	112.1 (6)
C11—N2—C12	110.2 (7)	N2—C11—H11A	109.5
C8—N2—C12	112.3 (5)	N2—C11—H11B	109.5
C11—N2—Cr1	106.2 (5)	H11A—C11—H11B	109.5
C8—N2—Cr1	103.4 (4)	N2—C11—H19C	109.5
C12—N2—Cr1	112.8 (4)	H11A—C11—H19C	109.5
O2—C1—O1	124.0 (6)	H11B—C11—H19C	109.5
O2—C1—C2	119.0 (5)	N2—C12—H12A	109.5
O1—C1—C2	116.9 (5)	N2—C12—H12B	109.5
N1—C2—C3	115.0 (5)	H12A—C12—H12B	109.5
N1—C2—C1	107.0 (4)	N2—C12—H12C	109.5
C3—C2—C1	113.6 (5)	H12A—C12—H12C	109.5
N1—C2—H2	106.9	H12B—C12—H12C	109.5
C3—C2—H2	106.9	C14—N3—C13	114.1 (8)
C1—C2—H2	106.9	C14—N3—H3C	108.7
C4—C3—C2	116.1 (5)	C13—N3—H3C	108.7
C4—C3—H3A	108.3	C14—N3—H3D	108.7
C2—C3—H3A	108.3	C13—N3—H3D	108.7
C4—C3—H3B	108.3	H3C—N3—H3D	107.6
C2—C3—H3B	108.3	N3—C13—H13A	109.5
H3A—C3—H3B	107.4	N3—C13—H13B	109.5
O3—C4—O4	121.7 (7)	H13A—C13—H13B	109.5
O3—C4—C3	123.2 (6)	N3—C13—H13C	109.5
O4—C4—C3	114.9 (6)	H13A—C13—H13C	109.5
N1—C5—H8A	109.5	H13B—C13—H13C	109.5
N1—C5—H8B	109.5	N3—C14—H14A	109.5
H8A—C5—H8B	109.5	N3—C14—H14B	109.5
N1—C5—H8C	109.5	H14A—C14—H14B	109.5
H8A—C5—H8C	109.5	N3—C14—H14C	109.5
H8B—C5—H8C	109.5	H14A—C14—H14C	109.5
N1—C6—H6A	109.5	H14B—C14—H14C	109.5
N1—C6—H6B	109.5	H1WA—O1W—H1WB	107 (10)
H6A—C6—H6B	109.5		

Cr1—O1—C1—O2	176.8 (5)	Cr1—O5—C7—O6	164.7 (6)
Cr1—O1—C1—C2	−6.8 (7)	Cr1—O5—C7—C8	−14.7 (7)
C6—N1—C2—C3	70.4 (7)	C11—N2—C8—C9	−53.9 (8)
C5—N1—C2—C3	−53.3 (7)	C12—N2—C8—C9	70.4 (7)
Cr1—N1—C2—C3	−166.9 (4)	Cr1—N2—C8—C9	−167.7 (5)
C6—N1—C2—C1	−162.4 (6)	C11—N2—C8—C7	73.3 (7)
C5—N1—C2—C1	73.9 (6)	C12—N2—C8—C7	−162.4 (6)
Cr1—N1—C2—C1	−39.8 (5)	Cr1—N2—C8—C7	−40.5 (5)
O2—C1—C2—N1	−150.3 (5)	O6—C7—C8—N2	−140.5 (6)
O1—C1—C2—N1	33.1 (7)	O5—C7—C8—N2	38.9 (7)
O2—C1—C2—C3	−22.4 (8)	O6—C7—C8—C9	−12.5 (9)
O1—C1—C2—C3	161.1 (5)	O5—C7—C8—C9	166.9 (6)
N1—C2—C3—C4	−162.3 (5)	N2—C8—C9—C10	−163.7 (5)
C1—C2—C3—C4	74.0 (7)	C7—C8—C9—C10	72.3 (7)
C2—C3—C4—O3	−150.8 (7)	C8—C9—C10—O7	23.7 (10)
C2—C3—C4—O4	34.8 (9)	C8—C9—C10—O8	−157.9 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*O*···O1*W*	0.82	1.77	2.591 (8)	180
O8—H8*O*···O2^{i}	0.82	1.91	2.585 (7)	139
N3—H3*C*···Cl1^{ii}	0.89	2.24	3.121 (7)	172
N3—H3*D*···O3^{iii}	0.89	1.94	2.763 (9)	153
O1*W*—H1*WA*···O7^{ii}	0.86 (3)	2.17 (8)	2.895 (8)	142 (12)
O1*W*—H1*WB*···O6^{iv}	0.86 (3)	2.18 (3)	3.006 (9)	160 (7)
C6—H6*B*···O8^{v}	0.96	2.51	3.351 (9)	146
C6—H6*C*···O5	0.96	2.46	3.038 (9)	119
C12—H12*C*···O6^{vi}	0.96	2.52	3.193 (10)	127
C13—H13*B*···O8^{vi}	0.96	2.56	3.451 (12)	154

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