Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Aqua(2,2′-bipyridine)trifluoridochromium(III) dihydrate

Hai-Xing Liu

Microscale Science Institute, Weifang University, Weifang 261061, People's Republic of China, and Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China
Correspondence e-mail: wj-crystal@163.com

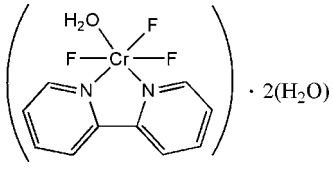
Received 6 August 2009; accepted 12 August 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.040; wR factor = 0.129; data-to-parameter ratio = 12.9.

The title compound, $[CrF_3(C_{10}H_8N_2)(H_2O)]\cdot 2H_2O$, was prepared by the reaction of CrF_3 and 2,2′-bipyridine under hydrous conditions. The metal centre is coordinated in a distorted octahedral mode by two N atoms from the organic ligand, three F atoms and one O atom of a water molecule. . The crystal packing is stabilized by O—H···O and O—H···F hydrogen-bonding contacts, which form a one-dimensional belt extending parallel to (100).

Related literature

For anion structures, see: Kumar *et al.* (2007); Krishnan *et al.* (2007); Wu *et al.* (2007); Dong *et al.* (2005). For related structures, see: Timco *et al.* (2005); Larsen *et al.* (2003); Ochsenbein *et al.* (2008).



· 2(H_2O)

Experimental

Crystal data

$[CrF_3(C_{10}H_8N_2)(H_2O)]\cdot 2H_2O$
M_r = 319.23
Monoclinic, $P2_1/c$
a = 9.0100 (18) Å
b = 7.4170 (15) Å
c = 20.759 (6) Å
β = 112.35 (3)°
V = 1283.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.93 mm^{-1}
T = 293 K
0.24 × 0.18 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
6478 measured reflections
2257 independent reflections
1916 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.129
S = 1.12
2257 reflections
175 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H2W1···F1	0.85	2.22	2.699 (3)	116
O1W—H2W1···F2^{i}	0.85	2.02	2.567 (3)	121
O1W—H2W1···F2^{i}	0.85	2.02	2.567 (3)	121
O1W—H1W1···F1^{ii}	0.85	1.97	2.550 (3)	125
O2W—H1W2···F3^{ii}	0.85	2.10	2.664 (4)	124
O2W—H2W2···O3W^{iii}	0.85	2.33	2.730 (5)	110
O3W—H2W3···F2^{iv}	0.80	1.98	2.767 (3)	171
O3W—H1W3···O3W^{v}	0.84	2.18	2.748 (7)	125

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2114).

References

Bruker (1997). *SMART* and *SAINT* . Bruker AXS Inc., Madison, Wisconsin, USA.
Dong, Y. B., Wang, H. Y., Ma, J. P. & Huang, R. Q. (2005). *Cryst. Growth Des.* **2**, 789–800.
Krishnan, S. M., Patel, N. M., Knapp, W. R. & Supkowski, R. M. (2007). *CrystEngComm*, **9**, 503–514.
Kumar, D. K., Das, A. & Dastidar, P. (2007). *CrystEngComm*, **9**, 548–555.
Larsen, F. K., Overgaard, J., Parsons, S., Rentschler, E., Smith, A. A., Timco, G. A. & Winpenny, R. E. P. (2003). *Angew. Chem. Int. Ed.* **42**, 5978–5981.
Ochsenbein, S. T., Tuna, F., Rancan, M., Davies, R. S. G., Muryn, C. A., Waldmann, O., Bircher, R., Sieber, A., Carver, G., Mutka, H., Fernandez-Alonso, F., Podlesnyak, A., Engelhardt, L. P., Timco, G. A., Güdel, H. U. & Winpenny, R. E. P. (2008). *Chem. Eur. J.* **14**, 5144–5158.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Timco, G. A., Batsanov, A. S., Larsen, F. K., Muryn, C. A., Overgaard, J., Teat, S. J. & Winpenny, R. E. P. (2005). *Chem. Commun.* pp. 3649–3651.
Wu, L. M., Teng, H. B., Feng, X. C. & Ke, X. B. (2007). *Cryst. Growth Des.* **7**, 1337–1342.

supporting information

Acta Cryst. (2009). E**65**, m1093 [doi:10.1107/S1600536809031808]

Aqua(2,2′-bipyridine)trifluoridochromium(III) dihydrate

Hai-Xing Liu

S1. Comment

In recent, the aspect of anion attracts much research interesting in coordination chemistry, like X^-, NO_3^-(Kumar *et al.*, 2007), BF_4^-, ClO_4^-(Krishnan *et al.*, 2007), SO_3^{2-}(Wu *et al.*, 2007). The anion components facilely either coordinate to metal atoms or fill the vacancy of Metal-organic frameworks, and intensively influence the supramolecular framework by hydrogen bonding and electrostatic interactions. But the study of F^- anion is still deficient. Because the HF strong acid easily attacks the glass surface and creates SiF_6^{2-} in the synthetical progress. Here we describe the synthesis and structure of the title Cr compound coordinating with F atom.

The title structure (Fig. 1) was build up of one Cr atom, one 2,2,-bipyridine ligand, three coordination F atoms, one coordination water molecule and two free water molecules. Cr atom is coordinated with two N atoms from 2,2′-bipyridine ligand, three F atoms, one water molecule, presenting a distorted octahedron geometry. The mean Cr—N, Cr—O and Cr—F bond lengths are similar to the reported (Timco *et al.*, 2005, Larsen *et al.*, 2003 & Ochsenbein *et al.*, 2008). The torsion angles of C1—N1—Cr1—O1w, C10—N2—Cr1—F3 are 4.13 (2) and -4.25 (2)°,respectively.

The free water molecules link each other by intermolecular O—H···O hydrogen bonds. And F atoms contact with water molecules *via* intermolecular O—H···F hydrogen bonds (Table 2). The hydrogen-bonding interactions display as the one-dimensional belt linking the the crystal packing as shown in Fig. 2.

S2. Experimental

All commercially obtained reagent-grade chemicals were used without further purification. The novelty $Cr(OH)_3$ was prepared by mixture $CrCl_3$ $6H_2O$ (5.33 g, 20 mmol) with NaOH (2.40 g, 60 mmol) in water solution. After filtered and washed with water, $Cr(OH)_3$ was added to hydrofluoric acid (1.20 g, 60 mmol). The stirring did not stop until the solid dissolved completely. The CrF_3 solution was obtained after increasing the pH value from 5 to 7. Ten drops of prepared CrF_3 solution were added in the solution of 2,2′-bipyridine (0.48 g, 3 mmol) in water and methanol (3:1 *v*/*v*, 40 ml). The resulting solution was refluxed for 2 h and filtered. The brown prism crystals were collected, after cooling and filtering (yield 1.10 g).

S3. Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C-H and O-H distances of 0.93–0.96 and 0.85 Å, respectively, and with $U_{iso}(H) = 1.2U_{eq}$ of the parent atoms.

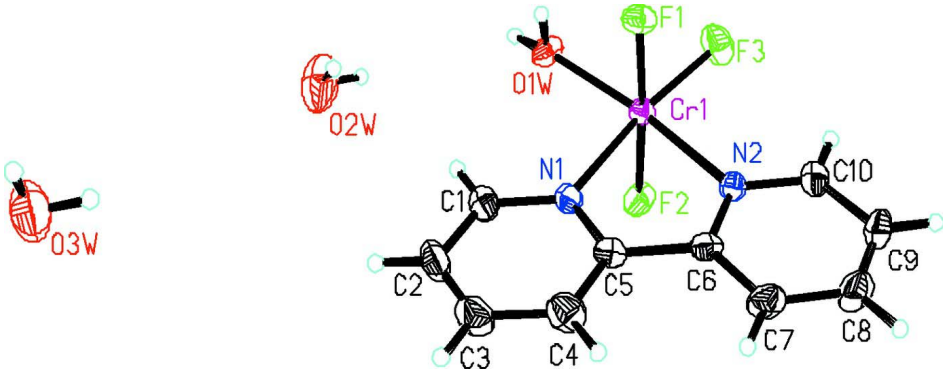


Figure 1

The molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

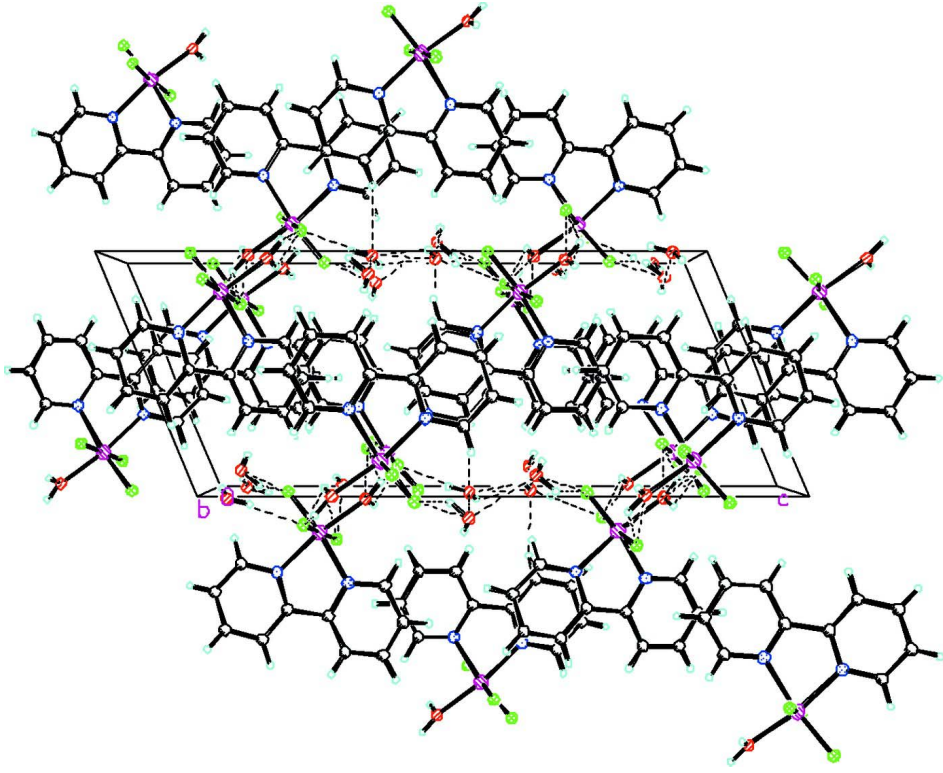


Figure 2

The packing view of the molecules of (I) along the crystallographic b direction.

Aqua(2,2′-bipyridine)trifluoridochromium(III) dihydrate

Crystal data

$[CrF_3(C_{10}H_8N_2)(H_2O)]\cdot 2H_2O$
$M_r = 319.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.0100\ (18)$ Å
$b = 7.4170\ (15)$ Å
$c = 20.759\ (6)$ Å
$\beta = 112.35\ (3)°$
$V = 1283.1\ (5)$ Å^3
$Z = 4$
$F(000) = 652$
$D_x = 1.653$ Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1024 reflections
θ = 2.4–25.0°
μ = 0.93 mm^{-1}
T = 293 K
Prism, brown
0.24 × 0.18 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
6478 measured reflections
2257 independent reflections
1916 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.0°, θ_{min} = 2.4°
h = −10→10
k = −8→8
l = −21→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.129
S = 1.12
2257 reflections
175 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0755P)^2 + 0.6391P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
Cr1	0.15638 (5)	0.28271 (7)	0.32953 (2)	0.0283 (2)
F1	0.2014 (2)	0.0442 (2)	0.31271 (9)	0.0393 (5)
F2	0.1128 (2)	0.5267 (2)	0.34286 (9)	0.0436 (5)
F3	−0.0083 (2)	0.2083 (3)	0.35635 (12)	0.0521 (6)
N1	0.3615 (3)	0.3591 (4)	0.31451 (13)	0.0331 (6)
N2	0.3252 (3)	0.2663 (3)	0.42877 (13)	0.0318 (6)
C1	0.3689 (4)	0.4133 (5)	0.25417 (18)	0.0453 (8)
H1A	0.2741	0.4299	0.2157	0.054*
C2	0.5145 (5)	0.4453 (5)	0.2477 (2)	0.0540 (10)
H2A	0.5168	0.4829	0.2054	0.065*
C3	0.6529 (5)	0.4212 (6)	0.3035 (2)	0.0579 (10)
H3A	0.7513	0.4406	0.2997	0.069*
C4	0.6469 (4)	0.3678 (6)	0.3661 (2)	0.0533 (10)

H4A	0.7410	0.3513	0.4050	0.064*
C5	0.4997 (4)	0.3393 (4)	0.37029 (17)	0.0358 (7)
C6	0.4791 (4)	0.2890 (4)	0.43569 (17)	0.0353 (7)
C7	0.6048 (5)	0.2693 (5)	0.4996 (2)	0.0506 (10)
H7A	0.7105	0.2842	0.5037	0.061*
C8	0.5698 (6)	0.2272 (6)	0.5569 (2)	0.0613 (12)
H8A	0.6521	0.2127	0.6003	0.074*
C9	0.4128 (5)	0.2068 (5)	0.54970 (18)	0.0559 (11)
H9A	0.3875	0.1802	0.5881	0.067*
C10	0.2933 (5)	0.2264 (5)	0.48461 (18)	0.0432 (8)
H10A	0.1870	0.2113	0.4796	0.052*
O1W	0.0184 (3)	0.3056 (3)	0.22962 (11)	0.0363 (5)
H1W1	−0.0536	0.3443	0.1923	0.044*
H2W1	0.0476	0.2023	0.2211	0.044*
O2W	0.0891 (5)	0.4622 (5)	0.06932 (16)	0.0883 (11)
H1W2	0.0245	0.4742	0.0901	0.106*
H2W2	0.1487	0.3719	0.0871	0.106*
O3W	0.9992 (4)	0.1082 (5)	0.05359 (17)	0.0790 (10)
H2W3	0.9750	0.0764	0.0850	0.095*
H1W3	1.0333	0.0066	0.0477	0.095*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.0252 (3)	0.0273 (3)	0.0288 (3)	0.00016 (18)	0.0059 (2)	0.00170 (18)
F1	0.0344 (10)	0.0296 (10)	0.0462 (10)	0.0000 (8)	0.0068 (8)	−0.0007 (8)
F2	0.0530 (12)	0.0329 (10)	0.0371 (10)	0.0091 (9)	0.0084 (9)	−0.0017 (8)
F3	0.0343 (11)	0.0647 (15)	0.0608 (13)	−0.0003 (10)	0.0221 (10)	0.0137 (10)
N1	0.0326 (14)	0.0322 (14)	0.0334 (14)	−0.0042 (11)	0.0113 (11)	0.0016 (11)
N2	0.0322 (14)	0.0298 (14)	0.0288 (14)	−0.0015 (11)	0.0063 (11)	0.0028 (10)
C1	0.048 (2)	0.046 (2)	0.0418 (19)	−0.0066 (17)	0.0172 (16)	0.0051 (15)
C2	0.068 (3)	0.050 (2)	0.059 (2)	−0.0085 (19)	0.042 (2)	0.0035 (18)
C3	0.043 (2)	0.066 (3)	0.072 (3)	−0.0106 (19)	0.030 (2)	−0.001 (2)
C4	0.0329 (18)	0.063 (2)	0.062 (2)	−0.0048 (18)	0.0159 (17)	0.000 (2)
C5	0.0315 (16)	0.0313 (16)	0.0425 (18)	−0.0038 (13)	0.0116 (14)	−0.0021 (14)
C6	0.0303 (17)	0.0296 (17)	0.0384 (18)	−0.0028 (13)	0.0046 (14)	−0.0014 (13)
C7	0.0354 (19)	0.052 (2)	0.046 (2)	−0.0042 (16)	−0.0048 (17)	0.0025 (16)
C8	0.062 (3)	0.062 (3)	0.037 (2)	−0.003 (2)	−0.0079 (19)	0.0087 (17)
C9	0.072 (3)	0.057 (3)	0.0294 (19)	−0.006 (2)	0.0090 (18)	0.0086 (16)
C10	0.045 (2)	0.045 (2)	0.0367 (19)	−0.0040 (16)	0.0128 (16)	0.0048 (15)
O1W	0.0351 (12)	0.0310 (11)	0.0305 (11)	0.0072 (9)	−0.0013 (9)	−0.0002 (9)
O2W	0.124 (3)	0.079 (2)	0.081 (2)	0.037 (2)	0.060 (2)	0.0043 (18)
O3W	0.096 (2)	0.083 (2)	0.079 (2)	−0.002 (2)	0.0559 (19)	0.0067 (19)

Geometric parameters (Å, º)

Cr1—F3	1.856 (2)	C4—H4A	0.9300
Cr1—F1	1.8769 (18)	C5—C6	1.486 (5)

Cr1—F2	1.8942 (19)	C6—C7	1.386 (5)
Cr1—O1W	1.979 (2)	C7—C8	1.379 (6)
Cr1—N2	2.047 (3)	C7—H7A	0.9300
Cr1—N1	2.067 (3)	C8—C9	1.373 (6)
N1—C1	1.341 (4)	C8—H8A	0.9300
N1—C5	1.348 (4)	C9—C10	1.378 (5)
N2—C10	1.329 (4)	C9—H9A	0.9300
N2—C6	1.350 (4)	C10—H10A	0.9300
C1—C2	1.388 (5)	O1W—H1W1	0.8498
C1—H1A	0.9300	O1W—H2W1	0.8500
C2—C3	1.353 (6)	O2W—H1W2	0.8500
C2—H2A	0.9300	O2W—H2W2	0.8500
C3—C4	1.378 (6)	O3W—H2W3	0.7978
C3—H3A	0.9300	O3W—H1W3	0.840 (10)
C4—C5	1.378 (5)		

F3—Cr1—F1	91.69 (9)	C2—C3—H3A	120.3
F3—Cr1—F2	90.37 (10)	C4—C3—H3A	120.3
F1—Cr1—F2	177.37 (8)	C3—C4—C5	119.1 (4)
F3—Cr1—O1W	94.86 (10)	C3—C4—H4A	120.4
F1—Cr1—O1W	88.84 (8)	C5—C4—H4A	120.4
F2—Cr1—O1W	89.35 (8)	N1—C5—C4	121.7 (3)
F3—Cr1—N2	93.05 (10)	N1—C5—C6	114.7 (3)
F1—Cr1—N2	90.05 (9)	C4—C5—C6	123.6 (3)
F2—Cr1—N2	91.48 (9)	N2—C6—C7	121.4 (3)
O1W—Cr1—N2	172.04 (10)	N2—C6—C5	114.5 (3)
F3—Cr1—N1	171.69 (10)	C7—C6—C5	124.1 (3)
F1—Cr1—N1	87.80 (9)	C8—C7—C6	118.6 (4)
F2—Cr1—N1	90.39 (10)	C8—C7—H7A	120.7
O1W—Cr1—N1	93.42 (10)	C6—C7—H7A	120.7
N2—Cr1—N1	78.66 (11)	C9—C8—C7	119.7 (4)
C1—N1—C5	118.5 (3)	C9—C8—H8A	120.2
C1—N1—Cr1	126.0 (2)	C7—C8—H8A	120.2
C5—N1—Cr1	115.3 (2)	C8—C9—C10	118.9 (4)
C10—N2—C6	119.3 (3)	C8—C9—H9A	120.5
C10—N2—Cr1	124.5 (2)	C10—C9—H9A	120.5
C6—N2—Cr1	116.1 (2)	N2—C10—C9	122.1 (4)
N1—C1—C2	121.7 (3)	N2—C10—H10A	119.0
N1—C1—H1A	119.1	C9—C10—H10A	119.0
C2—C1—H1A	119.1	Cr1—O1W—H1W1	160.5
C3—C2—C1	119.5 (3)	Cr1—O1W—H2W1	91.2
C3—C2—H2A	120.3	H1W1—O1W—H2W1	107.7
C1—C2—H2A	120.3	H1W2—O2W—H2W2	107.7
C2—C3—C4	119.4 (4)	H2W3—O3W—H1W3	94.7

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H2*W*1···F1	0.85	2.22	2.699 (3)	116
O1*W*—H2*W*1···F2^{i}	0.85	2.02	2.567 (3)	121
O1*W*—H2*W*1···F2^{i}	0.85	2.02	2.567 (3)	121
O1*W*—H1*W*1···F1^{ii}	0.85	1.97	2.550 (3)	125
O2*W*—H1*W*2···F3^{ii}	0.85	2.10	2.664 (4)	124
O2*W*—H2*W*2···O3*W*^{iii}	0.85	2.33	2.730 (5)	110
O3*W*—H2*W*3···F2^{iv}	0.80	1.98	2.767 (3)	171
O3*W*—H2*W*3···F3^{iv}	0.80	2.96	3.490 (4)	126
O3*W*—H1*W*3···O3*W*^{v}	0.84	2.18	2.748	125

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