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# Tris(3-chloropentane-2,4-dionato-$\kappa^2O,O'$)aluminium

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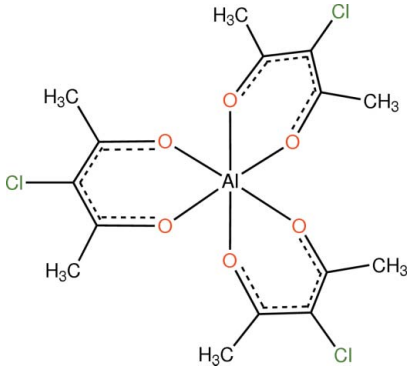
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.042; $wR$ factor = 0.125; data-to-parameter ratio = 18.3.

In the title compound, $[Al(C_5H_6ClO_2)_3]$, the $Al^{III}$ cation is situated on a twofold rotation axis and is coordinated by six O atoms from three 3-chloropentane-2,4-dionate ligands in an octahedral environment. Al—O bond lengths are in the range 1.8741 (14)–1.8772 (14) Å. In the crystal, molecules are linked *via* C—H···Cl contacts.

## Related literature

For applications of metal complexes with $\beta$-diketonate ligands, see: Bray *et al.* (2007); Garibay *et al.* (2009); Lichtenberger *et al.* (2010); Perdih (2011); Vreshch *et al.* (2004); Wu & Wang (2009). For related structures, see: Hon & Pfluger (1973); Perdih (2012).




## Experimental

### Crystal data

$[Al(C_5H_6ClO_2)_3]$
$M_r$ = 427.62
Monoclinic, $C2/c$
$a$ = 12.8790 (3) Å
$b$ = 9.9086 (2) Å
$c$ = 15.5311 (4) Å
$\beta$ = 106.368 (2)°
$V$ = 1901.64 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.56 mm$^{-1}$
$T$ = 293 K
0.33 × 0.25 × 0.08 mm

### Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.838, $T_{max}$ = 0.957
3916 measured reflections
2156 independent reflections
1759 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.014

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.125
$S$ = 1.05
2156 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C6—H6$A$···Cl1$^{i}$ | 0.96 | 2.94 | 3.796 (2) | 149 |

Symmetry code: (i) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z$.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2374).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, m806 [doi:10.1107/S1600536812023203]

## Tris(3-chloropentane-2,4-dionato-$\kappa^2O,O'$)aluminium

**Franc Perdih**

### Comment

$\beta$-Diketonates have been proven to be versatile ligands for various metal ions. They can be easily derivatized, thus modifying the electronic and steric nature of these ligands to design suitable structure/function relationships (Bray *et al.*, 2007; Garibay *et al.*, 2009; Perdih, 2011). $\beta$-Diketonate compounds of aluminium have received great attention due to the promise of the construction of cages (Vreshch *et al.*, 2004; Wu & Wang, 2009). Besides that, aluminium $\beta$-diketonates and malonates can be good precursors in metal-organic chemical vapour deposition (MOCVD) (Bray *et al.*, 2007; Garibay *et al.*, 2009; Lichtenberger *et al.*, 2010).

In the title molecule (Fig. 1), the aluminium(III) cation is situated on a twofold axis, and is surrounded by six O atoms from three 3-chloropentane-2,4-dionate ligands in a octahedral environment. The geometry around aluminium is close to the orthogonallity as can be seen from the angles. The Al—O bond lengths are in the range 1.8741 (14)–1.8772 (14) Å and are similar as for example in $Al(acac)_3$ (Hon & Pfluger, 1973). The displacement of the metal atom is best described by a bending of a chelate ligand about the "bite" atoms. The angles between the O—Al—O and the ligand chelate mean planes are 0.38° and 1.72°. For comparison these values are 0.78° and 12.68° in the isostructural iron(III) compound (Perdih, 2012). A 1-D framework is achieved due to intermolecular C6–H6A···Cl1 ($-x + 1/2, -y + 1/2, -z$) contacts (Fig. 2).

### Experimental

To a clear solution of $Al_2(SO_4)_3{\cdot}18H_2O$ (1 mmol, 0.67 g) in water (15 ml) a solution of 3-chloropentane-2,4-dione (6 mmol, 0.81 g) in methanol (5 ml) was added while stirring. Afterwards 1 *M* NaOH (6 ml) was slowly added and the resulting solution was stirred at 70°C for 15 minutes. After cooling to room temperature the light pink product was filtrated, washed with water (20 ml), and subsequently air-dried. Yield: 0.60 g, 70%. Crystals suitable for X-ray analysis were obtained by recrystallization from ethanol.

### Refinement

All H atoms were initially located in a difference Fourier maps and were subsequently treated as riding atoms in geometrically idealized positions, with C—H = 0.96 Å, and with $U_{iso}(H) = 1.5U_{eq}(C)$.

### Computing details

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *publCIF* (Westrip, 2010).

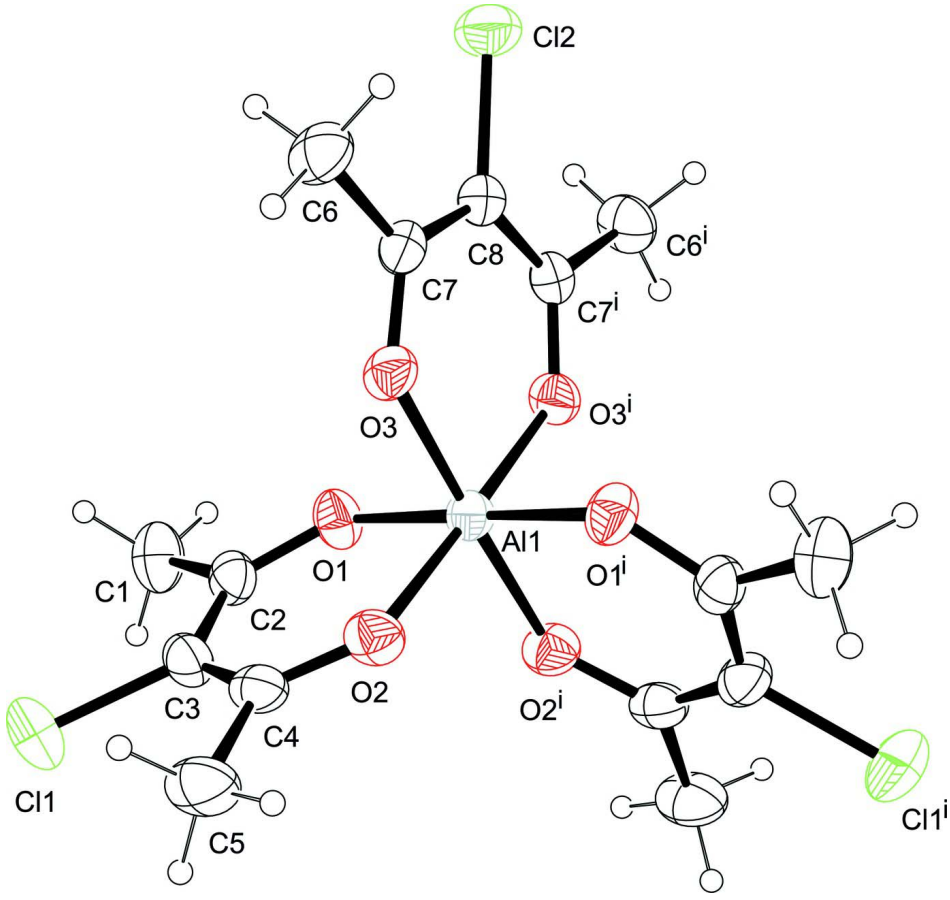


**Figure 1**

Molecular structure of the title complex showing displacement ellipsoids at the 30% probability level. Symmetry code: i = $-x + 1, y, -z + 1/2$.

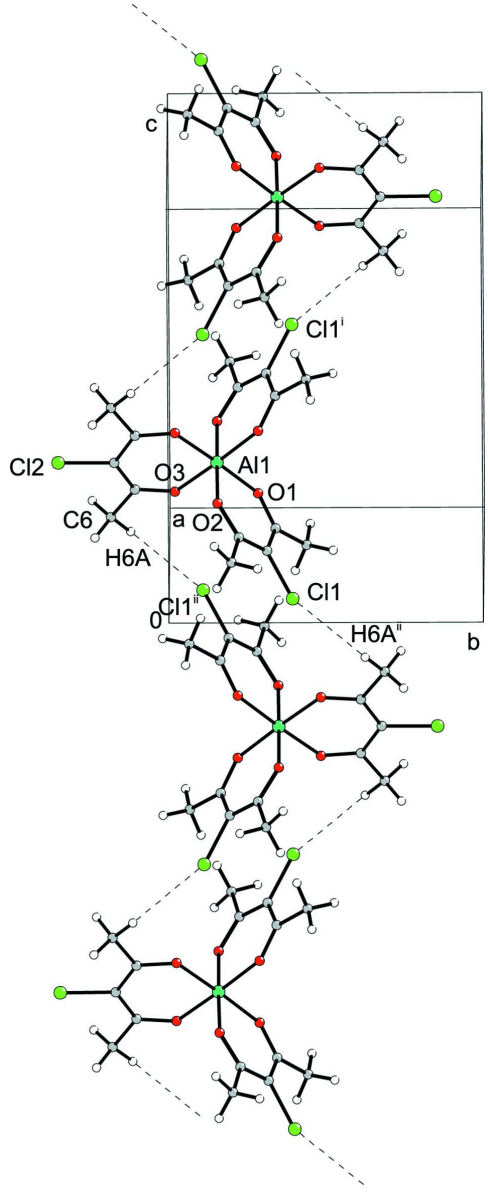


**Figure 2**

1D infinte chain with dashed lines indicating intermolecular C6—H6A···Cl1 hydrogen bonding. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Symmetry code: ii = –*x* + 1/2, –*y* + 1/2, –*z*.

### Tris(3-chloropentane-2,4-dionato-$\kappa^2O,O'$)aluminium

*Crystal data*

$[Al(C_5H_6ClO_2)_3]$
$M_r$ = 427.62
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
*a* = 12.8790 (3) Å
*b* = 9.9086 (2) Å
*c* = 15.5311 (4) Å
$\beta$ = 106.368 (2)°
*V* = 1901.64 (8) Å$^3$
*Z* = 4
*F*(000) = 880
$D_x$ = 1.494 Mg m$^{-3}$
Mo *K*$\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2269 reflections
$\theta$ = 2.6–27.5°
$\mu$ = 0.56 mm$^{-1}$

$T$ = 293 K
Plate, pink

0.33 × 0.25 × 0.08 mm

*Data collection*

Nonius KappaCCD area-detector diffractometer
Graphite monochromator
Detector resolution: 0.055 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.838, $T_{max}$ = 0.957

3916 measured reflections
2156 independent reflections
1759 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.014
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 5.6°
$h$ = −16→16
$k$ = −10→12
$l$ = −20→20

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.125
$S$ = 1.05
2156 reflections
118 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 1.1678P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

*Special details*

**Experimental**. 211 frames in 5 sets of $\omega$ scans. Rotation/frame = 2.0 °. Crystal-detector distance = 25.00 mm. Measuring time = 55 s/°.

**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Al1 | 0.5 | 0.15245 (8) | 0.25 | 0.0454 (2) |
| Cl1 | 0.36200 (7) | 0.39260 (8) | −0.04272 (5) | 0.0995 (3) |
| Cl2 | 0.5 | −0.35153 (8) | 0.25 | 0.0799 (3) |
| O1 | 0.39394 (11) | 0.28432 (14) | 0.20496 (10) | 0.0575 (4) |
| O2 | 0.53620 (11) | 0.15368 (15) | 0.14118 (9) | 0.0565 (4) |
| O3 | 0.39461 (10) | 0.01885 (13) | 0.20911 (9) | 0.0527 (3) |
| C1 | 0.2712 (2) | 0.4373 (3) | 0.1141 (2) | 0.0824 (8) |
| H1A | 0.2095 | 0.4025 | 0.0694 | 0.124* |
| H1B | 0.292 | 0.5223 | 0.0945 | 0.124* |
| H1C | 0.2532 | 0.4496 | 0.1695 | 0.124* |
| C2 | 0.36336 (15) | 0.33939 (18) | 0.12819 (15) | 0.0558 (5) |
| C3 | 0.41130 (17) | 0.3110 (2) | 0.06059 (14) | 0.0587 (5) |
| C4 | 0.49653 (15) | 0.2202 (2) | 0.06969 (12) | 0.0529 (4) |

| | | | | |
|---|---|---|---|---|
| C5 | 0.5460 (2) | 0.1956 (3) | −0.00518 (15) | 0.0756 (7) |
| H5A | 0.6094 | 0.1406 | 0.016 | 0.113* |
| H5B | 0.5656 | 0.2803 | −0.0262 | 0.113* |
| H5C | 0.4947 | 0.1502 | −0.0534 | 0.113* |
| C6 | 0.29806 (17) | −0.1843 (2) | 0.17442 (17) | 0.0671 (6) |
| H6A | 0.2393 | −0.1214 | 0.156 | 0.101* |
| H6B | 0.2852 | −0.2439 | 0.2191 | 0.101* |
| H6C | 0.3034 | −0.2359 | 0.1235 | 0.101* |
| C7 | 0.40167 (15) | −0.10871 (18) | 0.21301 (11) | 0.0468 (4) |
| C8 | 0.5 | −0.1744 (3) | 0.25 | 0.0493 (6) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Al1 | 0.0412 (4) | 0.0466 (4) | 0.0457 (4) | 0 | 0.0078 (3) | 0 |
| Cl1 | 0.1147 (6) | 0.0930 (5) | 0.0719 (4) | 0.0188 (4) | −0.0043 (4) | 0.0304 (3) |
| Cl2 | 0.0779 (6) | 0.0479 (4) | 0.0966 (6) | 0 | −0.0039 (4) | 0 |
| O1 | 0.0517 (7) | 0.0531 (8) | 0.0671 (8) | 0.0097 (6) | 0.0155 (6) | 0.0005 (6) |
| O2 | 0.0512 (7) | 0.0686 (8) | 0.0482 (7) | 0.0139 (6) | 0.0116 (5) | 0.0076 (6) |
| O3 | 0.0422 (6) | 0.0522 (7) | 0.0565 (7) | 0.0005 (5) | 0.0021 (5) | −0.0059 (6) |
| C1 | 0.0621 (13) | 0.0577 (12) | 0.114 (2) | 0.0167 (11) | 0.0029 (13) | −0.0025 (13) |
| C2 | 0.0430 (9) | 0.0406 (9) | 0.0739 (13) | −0.0008 (7) | 0.0002 (8) | −0.0001 (8) |
| C3 | 0.0546 (11) | 0.0536 (10) | 0.0573 (11) | −0.0005 (9) | −0.0015 (8) | 0.0105 (9) |
| C4 | 0.0461 (9) | 0.0589 (10) | 0.0483 (9) | −0.0071 (8) | 0.0043 (7) | 0.0034 (8) |
| C5 | 0.0700 (14) | 0.1046 (19) | 0.0528 (11) | 0.0003 (14) | 0.0182 (10) | 0.0051 (12) |
| C6 | 0.0496 (11) | 0.0674 (13) | 0.0775 (14) | −0.0105 (10) | 0.0066 (10) | −0.0102 (11) |
| C7 | 0.0457 (9) | 0.0526 (10) | 0.0400 (8) | −0.0046 (7) | 0.0088 (7) | −0.0045 (7) |
| C8 | 0.0512 (14) | 0.0484 (13) | 0.0443 (12) | 0 | 0.0072 (10) | 0 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Al1—O3 | 1.8741 (14) | C1—H1C | 0.96 |
| Al1—O3$^{i}$ | 1.8741 (14) | C2—C3 | 1.389 (3) |
| Al1—O2$^{i}$ | 1.8756 (13) | C3—C4 | 1.395 (3) |
| Al1—O2 | 1.8756 (13) | C4—C5 | 1.495 (3) |
| Al1—O1$^{i}$ | 1.8772 (14) | C5—H5A | 0.96 |
| Al1—O1 | 1.8772 (14) | C5—H5B | 0.96 |
| Cl1—C3 | 1.748 (2) | C5—H5C | 0.96 |
| Cl2—C8 | 1.755 (3) | C6—C7 | 1.500 (3) |
| O1—C2 | 1.269 (3) | C6—H6A | 0.96 |
| O2—C4 | 1.268 (2) | C6—H6B | 0.96 |
| O3—C7 | 1.267 (2) | C6—H6C | 0.96 |
| C1—C2 | 1.500 (3) | C7—C8 | 1.396 (2) |
| C1—H1A | 0.96 | C8—C7$^{i}$ | 1.396 (2) |
| C1—H1B | 0.96 | | |
| | | | |
| O3—Al1—O3$^{i}$ | 90.12 (8) | C3—C2—C1 | 121.5 (2) |
| O3—Al1—O2$^{i}$ | 88.26 (6) | C2—C3—C4 | 123.97 (18) |
| O3$^{i}$—Al1—O2$^{i}$ | 92.26 (6) | C2—C3—Cl1 | 118.44 (16) |
| O3—Al1—O2 | 92.26 (6) | C4—C3—Cl1 | 117.59 (17) |

| | | | |
|---|---|---|---|
| O3$^{i}$—Al1—O2 | 88.26 (6) | O2—C4—C3 | 122.35 (18) |
| O2$^{i}$—Al1—O2 | 179.26 (10) | O2—C4—C5 | 116.20 (19) |
| O3—Al1—O1$^{i}$ | 178.02 (6) | C3—C4—C5 | 121.44 (19) |
| O3$^{i}$—Al1—O1$^{i}$ | 89.08 (6) | C4—C5—H5A | 109.5 |
| O2$^{i}$—Al1—O1$^{i}$ | 89.96 (6) | C4—C5—H5B | 109.5 |
| O2—Al1—O1$^{i}$ | 89.53 (7) | H5A—C5—H5B | 109.5 |
| O3—Al1—O1 | 89.08 (6) | C4—C5—H5C | 109.5 |
| O3$^{i}$—Al1—O1 | 178.02 (6) | H5A—C5—H5C | 109.5 |
| O2$^{i}$—Al1—O1 | 89.53 (7) | H5B—C5—H5C | 109.5 |
| O2—Al1—O1 | 89.96 (6) | C7—C6—H6A | 109.5 |
| O1$^{i}$—Al1—O1 | 91.78 (9) | C7—C6—H6B | 109.5 |
| C2—O1—Al1 | 130.55 (13) | H6A—C6—H6B | 109.5 |
| C4—O2—Al1 | 130.63 (13) | C7—C6—H6C | 109.5 |
| C7—O3—Al1 | 130.76 (12) | H6A—C6—H6C | 109.5 |
| C2—C1—H1A | 109.5 | H6B—C6—H6C | 109.5 |
| C2—C1—H1B | 109.5 | O3—C7—C8 | 121.98 (17) |
| H1A—C1—H1B | 109.5 | O3—C7—C6 | 115.78 (17) |
| C2—C1—H1C | 109.5 | C8—C7—C6 | 122.24 (19) |
| H1A—C1—H1C | 109.5 | C7$^{i}$—C8—C7 | 124.4 (2) |
| H1B—C1—H1C | 109.5 | C7$^{i}$—C8—Cl2 | 117.82 (12) |
| O1—C2—C3 | 122.47 (17) | C7—C8—Cl2 | 117.82 (12) |
| O1—C2—C1 | 116.0 (2) | | |

| | | | |
|---|---|---|---|
| O3—Al1—O1—C2 | −89.43 (17) | C1—C2—C3—C4 | −179.5 (2) |
| O2$^{i}$—Al1—O1—C2 | −177.70 (17) | O1—C2—C3—Cl1 | 179.76 (15) |
| O2—Al1—O1—C2 | 2.83 (18) | C1—C2—C3—Cl1 | 0.1 (3) |
| O1$^{i}$—Al1—O1—C2 | 92.36 (17) | Al1—O2—C4—C3 | −0.2 (3) |
| O3—Al1—O2—C4 | 87.74 (18) | Al1—O2—C4—C5 | 179.99 (15) |
| O3$^{i}$—Al1—O2—C4 | 177.79 (18) | C2—C3—C4—O2 | 1.3 (3) |
| O1$^{i}$—Al1—O2—C4 | −93.12 (18) | Cl1—C3—C4—O2 | −178.31 (15) |
| O1—Al1—O2—C4 | −1.34 (18) | C2—C3—C4—C5 | −179.0 (2) |
| O3$^{i}$—Al1—O3—C7 | 1.05 (13) | Cl1—C3—C4—C5 | 1.4 (3) |
| O2$^{i}$—Al1—O3—C7 | −91.21 (17) | Al1—O3—C7—C8 | −2.0 (3) |
| O2—Al1—O3—C7 | 89.32 (16) | Al1—O3—C7—C6 | 178.65 (13) |
| O1—Al1—O3—C7 | 179.24 (16) | O3—C7—C8—C7$^{i}$ | 1.01 (12) |
| Al1—O1—C2—C3 | −2.7 (3) | C6—C7—C8—C7$^{i}$ | −179.72 (19) |
| Al1—O1—C2—C1 | 176.98 (15) | O3—C7—C8—Cl2 | −178.99 (12) |
| O1—C2—C3—C4 | 0.2 (3) | C6—C7—C8—Cl2 | 0.28 (19) |

Symmetry code: (i) −*x*+1, *y*, −*z*+1/2.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C6—H6*A*···Cl1$^{ii}$ | 0.96 | 2.94 | 3.796 (2) | 149 |

Symmetry code: (ii) −*x*+1/2, −*y*+1/2, −*z*.