Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Synthesis, crystal structure and Hirshfeld surface analysis of 2,2-dichloro-3,3-diethoxy-1-(4-fluorophenyl)propan-1-ol

Saadet N. Guseynova,[a] Aida I. Samigullina,[b] Cemile Baydere Demir,[c] Necmi Dege,[d] Nayim Sepay,[e] Ennio Zangrando,[f] Khudayar I. Hasanov[g] and Alebel N. Belay[h]*

[a]Kosygin State University of Russia, 117997 Moscow, Russian Federation, [b]N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, 119991 Moscow, Russian Federation, [c]Physiotherapy Program, Vocational School of Health Services, Demiroglu Bilim University, 34570-Istanbul, Türkiye, [d]Department of Physics, Faculty of Sciences, Ondokuz Mayıs University, Samsun 55200, Türkiye, [e]Department of Chemistry, Lady Brabourne College, Kolkata 700017, India, [f]Department of Chemical and Pharmaceutical Sciences, University of Trieste, 34127 Trieste, Italy, [g]Azerbaijan Medical University, Scientific Research Centre (SRC), A. Kasumzade St. 14, AZ 1022, Baku, Azerbaijan, and [h]Department of Chemistry, Bahir Dar University, PO Box 79, Bahir Dar, Ethiopia. *Correspondence e-mail: alebel.nibret@bdu.edu.et

Received 20 January 2025
Accepted 6 March 2025

Edited by S.-L. Zheng, Harvard University, USA

Keywords: crystal structure; dichlorohydrin; ketoacetal; β-oxoaldehyde; Hirshfeld surface analysis.

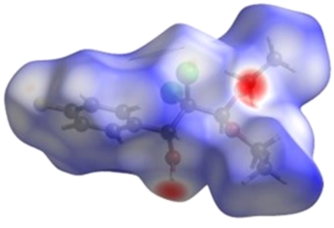
CCDC reference: 2429365

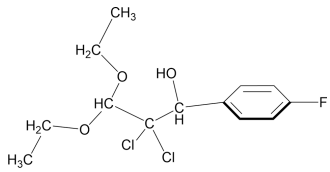
Supporting information: this article has supporting information at journals.iucr.org/e

The title molecule, $C_{13}H_{17}Cl_2FO_3$, crystallizes in the orthorhombic space group $P2_12_12_1$ with one molecule in the asymmetric unit. The skeleton of the molecule exhibits an *anti* conformation with a C—C—C—C(Ph) torsion angle of −174.97 (18)°. The species are weakly hydrogen bonded to form a polymeric chain elongated in the direction of the *b* axis. This interaction is realised by the hydroxyl group with an ether O atom of a symmetry-related species [O—H···O hydrogen-bond distance of 2.975 (2) Å]. No π-stacking interaction involving the fluorobenzyl moiety is detected in the crystal structure. Hirshfeld surface analysis, confirming the O—H···O donor–acceptor interactions, indicates that the most important contributions to the surface contacts are H···H (47.0%), Cl···H (19.5%), C···H (12.1%) and F···H (10.7%).

1. Chemical context

α-Haloketones and their derivatives are versatile synthetic precursors or building blocks for the synthesis of heterocyclic compounds, multidentate ligands, supramolecular synthons, *etc*. (Erian *et al.*, 2003; Guseinov *et al.*, 2017, 2022). We have recently isolated 20-membered macrocycles by the simple condensation of α,α-dihalo-β-oxoaldehydes with diaminofurazan in acetonitrile, where the interior and exterior sites of these macrocycles comprise hydrogen- and halogen-bond-donor sites, respectively (Guseinov *et al.*, 2024). On the other hand, the hydrogenation of ketones is an emerging area in synthetic organic chemistry and catalysis, of significant interest to the pharmaceutical industry, agrochemicals, *etc*. (Yang & Fang, 2023). Similar to other supramolecular systems (Gurbanov *et al.*, 2020, 2022), we believe that the halogen atoms in hydrogenation products of ketones can act as halogen-bond-donor centres in crystal engineering.





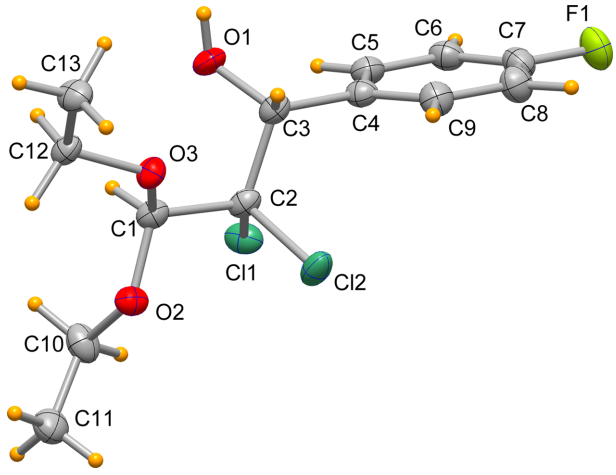


Figure 1
The molecular structure of the asymmetric unit of compound **1**. Displacement ellipsoids are drawn at the 50% probability level.

A series of crystal structures of compounds obained by reduction of 2,2,2-trichloro-1-arylethanones by *R*Mg*X*, having a Ph–CHOH–CCl_2– fragment, have also been reported (Essa *et al.*, 2013, 2105). Herein, we have used a simple method for the hydrogenation of 2,2-dichloro-3,3-diethoxy-1-(4-fluorophenyl)propan-1-one to prepare the title molecule, **1**.

2. Structural commentary

The molecule of the title compound (**1**) is shown in Fig. 1. The central chain with atoms C1, C2, C3 and C4 shows a staggered conformation, with a torsion angle about C2—C3 of 174.97 (18)°. The C2—Cl bond lengths are 1.792 (2) and 1.778 (2) Å, to be compared with the value of 1.798 Å reported by Negrier *et al.* (2002) for 2,2-dichloropropane and to the range of 1.791 (1)–1.800 (1) Å measured by Cornia *et al.* (2012) in a series of 2,2-dichlorobutan-1-one derivatives. The C1—O(ethoxy) bond lengths are 1.410 (3) and 1.392 (3) Å. The molecule shows intramolecular nonconventional hydrogen bonds C5—H5···O1 and C10—H10*B*···Cl1, with distances between the donor and acceptor atom of 2.820 (3) and 3.326 (3) Å, respectively.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82 (5)	2.17 (5)	2.975 (2)	165 (4)
O1—H1···O3^{i}	0.82 (5)	2.43 (5)	3.046 (2)	133 (4)

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

It is worth noting that the mentioned hydrogenation reaction for the preparation of the title molecule produced a racemic mixture of molecules, but the crystallization process separated the two chiral isomers. The present molecule crystallizes in the space group $P2_12_12_1$ and shows an absolute configuration of *R* at atom C3. The Flack parameter of the structural model determined by the single-crystal structure analysis is −0.001 (6).

3. Supramolecular features

The species are hydrogen bonded to form a linear polymeric chain elongated in the direction of crystallographic *b* axis (Fig. 2 and Table 1). The O1—H1···O2^{i} hydrogen bond has an O···O distance of 2.975 (2) Å, while a possible O1—H1···O3^{i} interaction having an O···O distance of 3.046 (2) Å is not to be excluded although weaker. In addition, the packing evidences C10—H10*A*···Cl1 interactions [C···Cl distance of 3.636 (3) Å], giving rise to chains developing along the *a* axis (Fig. 3).

4. Hirshfeld surface analysis

The Hirshfeld surface (HS) analysis (Spackman *et al.*, 2009) identifies and quantifies noncovalent interactions within a crystalline matrix (Biswas *et al.*, 2025; Das *et al.*, 2025; Sepay *et al.*, 2023). Four notable red spots are identified on the surface, specifically on the OH hydrogen, the OH oxygen and the

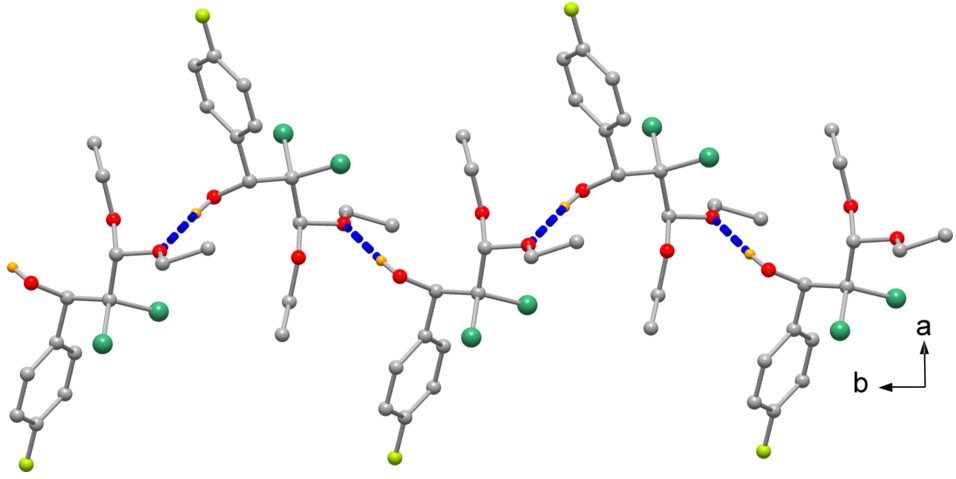


Figure 2
Mono-periodic array built by hydrogen bonds developed in the direction of the *b* axis.

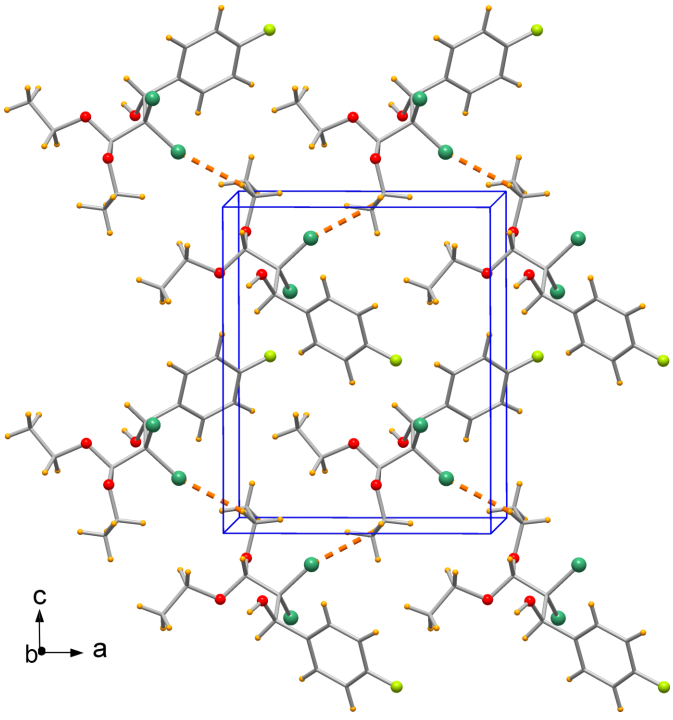


Figure 3
The packing of molecules connected by nonconventional C10—H10*A*···Cl1 hydrogen bonds.

acetal O atoms, indicating substantial donor–acceptor interactions, particularly associated with O—H···O(π) and C—H···O interactions [Fig. 4(*a*)]. Additionally, faint red patches around the F and Cl atoms suggest their minor contributions to the crystal interactions. The HS study shows that there are no π–π interactions in the solid state.

The HS analysis reveals that the intermolecular interactions in the crystal structure of compound **1** are primarily driven by H···H interactions [Figs. 4(*b*)–4(*f*)]. A notable spike in the d_e *versus* d_i plots was observed at approximately $d_e + d_i \simeq 2.38$ Å, accounting for 47.0% of the total interactions [Fig. 4(*b*)]. The Cl···H interactions followed, at around 3.32 Å, contributing 19.5% [Fig. 4(*c*)]. Other weak interactions included C···H ($d_e + d_i \simeq 3.26$ Å, 12.1%), F···H ($d_e + d_i \simeq 2.78$ Å, 10.7%) and Cl···F ($d_e + d_i \simeq 3.6$ Å, 1.9%). O···H interactions, similar to H···H interactions, showed a spike at 2.38 Å but represented only 8.1% of the total interactions [Fig. 4(*f*)]. The analysis identifies three weak interactions, *i.e.* C···H, F···H and Cl···F, affecting the crystallization process. The interactions are ranked in importance as H···H > Cl···H > C···H > F···H > O···H > Cl···F, with 12% of interactions from O—H···C(OEt)$_2$ contacts. Total polar interactions account for 40.2%, while nonpolar and van der Waals interactions make up 59.1%, indicating the amphiphilic nature of the compound.

5. Database survey

The Cambridge Structural Database (CSD, Version 5.45, update of March 2024; Groom *et al.*, 2016) was searched for molecules with comparable groups. The CCl_2 fragment can be compared with that of the molecular structure reports by Negrier *et al.* (2002; refcode QQQCZS01) describing a study on the polymorphism of 2,2-dichloropropane. On the other hand, a study with a series of ketene acetals comprising a 2,2-dichlorobutan-1-one fragment was published by Cornia *et al.* (2012; NEHQIC, NEHQOI, NEHQUO, NEHRAV, NEHREZ, NEHREZ01, NEHROJ and NEHRUP). Of interest are structures comprising a Ph–CHOH–CCl_2– frag-

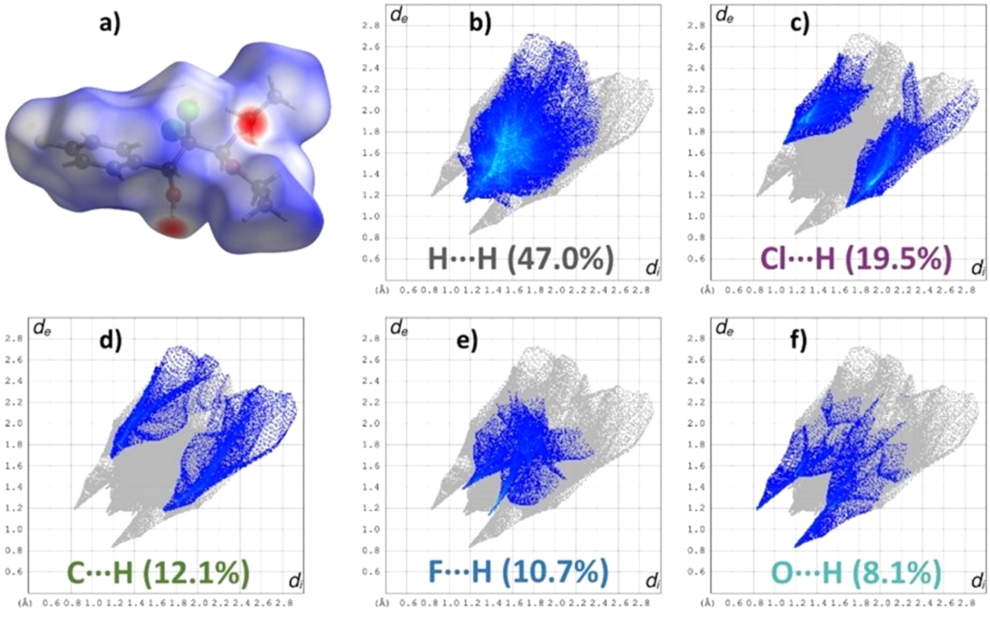


Figure 4
The HS of compound **1** over (*a*) d_{norm}, the d_e *versus* d_i plot of the (*b*) H···H, (*c*) Cl···H, (*d*) C···H, (*e*) F···H and (*f*) O···H interactions from the Hirshfeld surface analysis.

ment reported by Essa *et al.* (2013, 2015; BETPAT, BETPEX, BETPIB, UHIQOT, UHIQUZ, UHIRAG and UHISOV).

6. Synthesis and crystallization

A solution of 2,2-dichloro-3,3-diethoxy-1-(4-fluorophenyl)-propan-1-one (3.09 g, 0.01 mol) in methanol (30 ml) was cooled to 263 K. After addition of sodium borohydride (0.4 g, 0.011 mol), the mixture was stirred for 2 h at room temperature. Distilled water (20 ml) and concentrated hydrochloric acid (1 ml) were then added to the resulting solution and the organic solvent was distilled off on a rotary evaporator. The resulting suspension was extracted with diethyl ether twice and the organic fractions were combined and dried over anhydrous magnesium sulfate. After reaction, the solvent was distilled off in a vacuum of a water-jet pump, and the precipitated powder was recrystallized from chloroform to give white crystals of 2,2-dichloro-3,3-diethoxy-1-(4-fluorophenyl)-propan-1-ol (yield: 2.72 g, 88%; m.p. 485–487 K). ^{1}H NMR (300 MHz, DMSO-d_6): δ 1.22 ($2CH_3$, 6H), 3.63–3.94 ($2CH_2$, 4H), 4.77 (*s*, 1H), 5.03 (*d*, J = 5.0 Hz, 1H), 6.42 (*d*, J = 5.0 Hz, 1H), 7.19 (*m*, 2H), 7.55 (*m*, 2H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 15.51, 62.33, 79.35, 95.15, 113.45, 115.64, 127.98, 134.18, 160.28.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The positions and displacement parameters of the H atoms have been refined.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{17}Cl_2FO_3$
M_r	311.16
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, b, c (Å)	10.5608 (3), 10.8048 (3), 12.8872 (4)
V (Å^3)	1470.52 (7)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	4.10
Crystal size (mm)	0.15 × 0.12 × 0.08
Data collection	
Diffractometer	Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2022)
T_{min}, T_{max}	0.611, 0.810
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15942, 3123, 3111
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.634
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.025, 0.065, 1.05
No. of reflections	3123
No. of parameters	241
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.17, −0.18
Absolute structure	Flack x determined using 1296 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.001 (6)

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2019* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg, 1999) and *WinGX* (Farrugia, 2012).

Acknowledgements

This work has been supported by the Kosygin State University of Russia and Azerbaijan Medical University. The authors contributions are as follows: conceptualization, EZ and ANB; synthesis, SNG; X-ray analysis, AIS; writing (review and editing of the manuscript), EZ, CBD and ND; funding, KIH; Hirshfeld surface analysis, NS; supervision, EZ and ANB.

References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies Ltd, Yarnton, Oxfordshire, England.
Biswas, G., Afzal, M., Halder, U. C. & Sepay, N. A. (2025). *J. Mol. Struct.* **1322**, 140358.
Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Cornia, A., Felluga, F., Frenna, V., Ghelfi, F., Parsons, A. F., Pattarozzi, M., Roncaglia, F. & Spinelli, D. (2012). *Tetrahedron*, **68**, 5863–5881.
Das, S., Samanta, S., Mandal, A. & Sepay, N. (2023). *J. Mol. Struct.* **1292**, 136136.
Erian, A. W., Sherif, S. M. & Gaber, H. M. (2003). *Molecules*, **8**, 793–865.
Essa, A. H., Lerrick, R. I., Çiftçi, E., Harrington, R. W., Waddell, P. G., Clegg, W. & Hall, M. J. (2015). *Org. Biomol. Chem.* **13**, 5793–5803.
Essa, A. H., Lerrick, R. I., Tuna, F., Harrington, R. W., Clegg, W. & Hall, M. J. (2013). *Chem. Commun.* **49**, 2756–2758.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Gurbanov, A. V., Kuznetsov, M. L., Karmakar, A., Aliyeva, V. A., Mahmudov, K. T. & Pombeiro, A. J. L. (2022). *Dalton Trans.* **51**, 1019–1031.
Gurbanov, A. V., Kuznetsov, M. L., Mahmudov, K. T., Pombeiro, A. J. L. & Resnati, G. (2020). *Chem. A Eur. J.* **26**, 14833–14837.
Guseinov, F. I., Malinnikov, V. M., Lialin, K. N., Kobrakov, K. I., Shuvalova, E. V., Nelyubina, Y. V., Ugrak, B. I., Kustov, L. M. & Mahmudov, K. T. (2022). *Dyes Pigments*, **197**, 109898.
Guseinov, F. I., Ovsyannikov, V. O., Shuvalova, E. V., Kustov, L. M., Kobrakov, K. I., Samigullina, A. I. & Mahmudov, K. T. (2024). *New J. Chem.* **48**, 12869–12872.
Guseinov, F. I., Pistsov, M. F., Movsumzade, E. M., Kustov, L. M., Tafeenko, V. A., Chernyshev, V. V., Gurbanov, A. V., Mahmudov, K. T. & Pombeiro, A. J. L. (2017). *Crystals*, **7**, 327.
Negrier, P., Pardo, L. C., Salud, J., Tamarit, J. Ll., Barrio, M. O., López, D., Würflinger, A. & Mondieig, D. (2002). *Chem. Mater.* **14**, 1921–1929.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Rigaku OD (2022). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, Oxfordshire, England.
Sepay, N., Mandal, A. & Chakraborty, A. (2023). *J. Organomet. Chem.* **983**, 122556.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spackman, M. A. & Jayatilaka, D. (2009). *CrystEngComm*, **11**, 19–32.
Yang, S. & Fang, X. (2023). *Tetrahedron*, **145**, 133609.

supporting information

Acta Cryst. (2025). E**81**, 444-447 [https://doi.org/10.1107/S2056989025002154]

Synthesis, crystal structure and Hirshfeld surface analysis of 2,2-dichloro-3,3-diethoxy-1-(4-fluorophenyl)propan-1-ol

Saadet N. Guseynova, Aida I. Samigullina, Cemile Baydere Demir, Necmi Dege, Nayim Sepay, Ennio Zangrando, Khudayar I. Hasanov and Alebel N. Belay

Computing details

2,2-Dichloro-3,3-diethoxy-1-(4-fluorophenyl)propan-1-ol

Crystal data

$C_{13}H_{17}Cl_2FO_3$
M_r = 311.16
Orthorhombic, $P2_12_12_1$
a = 10.5608 (3) Å
b = 10.8048 (3) Å
c = 12.8872 (4) Å
V = 1470.52 (7) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.405 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 13434 reflections
θ = 3.4–77.6°
μ = 4.10 mm^{-1}
T = 100 K
Block, colourless
0.15 × 0.12 × 0.08 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Radiation source: micro-focus sealed X-ray tube
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2022)
T_{min} = 0.611, T_{max} = 0.810
15942 measured reflections
3123 independent reflections
3111 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 77.8°, θ_{min} = 5.3°
h = −12→13
k = −13→11
l = −15→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.065
S = 1.05
3123 reflections
241 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0332P)^2 + 0.5017P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: SHELXL2019 (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0022 (4)
Absolute structure: Flack *x* determined using 1296 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.001 (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}
Cl1	0.21324 (5)	0.29037 (5)	0.37004 (4)	0.02959 (15)
Cl2	0.30012 (5)	0.13688 (5)	0.19928 (5)	0.03094 (15)
F1	−0.11669 (14)	0.50329 (16)	0.00613 (12)	0.0394 (4)
O1	0.38547 (17)	0.48111 (15)	0.26820 (14)	0.0308 (4)
H1	0.430 (4)	0.531 (4)	0.236 (3)	0.067 (13)*
O2	0.46588 (15)	0.13111 (14)	0.38223 (12)	0.0255 (3)
O3	0.55686 (14)	0.25502 (14)	0.26145 (12)	0.0240 (3)
C1	0.4609 (2)	0.2489 (2)	0.33530 (17)	0.0236 (4)
H1A	0.469 (3)	0.315 (3)	0.388 (2)	0.032 (7)*
C2	0.3356 (2)	0.2697 (2)	0.27532 (18)	0.0239 (4)
C3	0.3432 (2)	0.3840 (2)	0.20378 (18)	0.0249 (4)
H3	0.410 (3)	0.363 (3)	0.149 (2)	0.033 (8)*
C4	0.2194 (2)	0.41543 (19)	0.15014 (17)	0.0245 (4)
C5	0.1336 (2)	0.4959 (2)	0.19664 (19)	0.0266 (4)
H5	0.153 (3)	0.530 (3)	0.261 (2)	0.024 (7)*
C6	0.0200 (2)	0.5259 (2)	0.14852 (19)	0.0279 (5)
H6	−0.044 (3)	0.580 (3)	0.177 (2)	0.036 (8)*
C7	−0.0049 (2)	0.4741 (2)	0.05275 (19)	0.0301 (5)
C8	0.0770 (2)	0.3949 (2)	0.00310 (18)	0.0319 (5)
H8	0.055 (3)	0.361 (3)	−0.068 (3)	0.043 (8)*
C9	0.1903 (2)	0.3664 (2)	0.05271 (18)	0.0292 (5)
H9	0.248 (3)	0.310 (3)	0.021 (2)	0.031 (7)*
C10	0.4336 (2)	0.1240 (3)	0.49060 (18)	0.0328 (5)
H10A	0.477 (3)	0.194 (3)	0.528 (2)	0.033 (7)*
H10B	0.339 (4)	0.135 (4)	0.500 (3)	0.055 (10)*
C11	0.4728 (3)	−0.0001 (3)	0.5306 (2)	0.0356 (6)
H11A	0.436 (3)	−0.064 (3)	0.492 (3)	0.038 (8)*
H11B	0.452 (3)	−0.004 (3)	0.604 (3)	0.050 (9)*
H11C	0.565 (3)	−0.008 (3)	0.520 (3)	0.046 (9)*
C12	0.67911 (19)	0.2820 (2)	0.30512 (18)	0.0253 (4)
H12A	0.709 (3)	0.210 (3)	0.349 (2)	0.029 (7)*
H12B	0.671 (3)	0.361 (3)	0.350 (3)	0.041 (8)*
C13	0.7697 (2)	0.3031 (2)	0.2167 (2)	0.0314 (5)
H13A	0.854 (3)	0.315 (3)	0.243 (2)	0.035 (8)*
H13B	0.741 (3)	0.376 (3)	0.180 (3)	0.048 (9)*
H13C	0.772 (3)	0.230 (3)	0.172 (2)	0.040 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0233 (2)	0.0294 (3)	0.0361 (3)	0.0038 (2)	0.0084 (2)	0.0058 (2)
Cl2	0.0267 (3)	0.0218 (2)	0.0443 (3)	−0.0002 (2)	−0.0073 (2)	−0.0079 (2)
F1	0.0330 (8)	0.0470 (9)	0.0381 (8)	0.0109 (7)	−0.0091 (6)	0.0037 (7)
O1	0.0322 (9)	0.0209 (7)	0.0392 (9)	−0.0089 (7)	−0.0052 (7)	0.0006 (7)
O2	0.0293 (7)	0.0232 (7)	0.0242 (7)	−0.0001 (6)	0.0035 (6)	−0.0002 (6)
O3	0.0170 (7)	0.0280 (8)	0.0270 (7)	−0.0020 (6)	0.0015 (6)	−0.0020 (6)
C1	0.0221 (10)	0.0223 (10)	0.0265 (10)	−0.0008 (8)	0.0042 (8)	−0.0024 (8)
C2	0.0201 (9)	0.0209 (10)	0.0305 (10)	−0.0015 (8)	0.0032 (8)	−0.0032 (8)
C3	0.0219 (9)	0.0246 (10)	0.0281 (10)	−0.0031 (8)	0.0013 (8)	−0.0019 (9)
C4	0.0226 (10)	0.0231 (10)	0.0278 (10)	−0.0020 (8)	0.0026 (8)	0.0022 (8)
C5	0.0291 (11)	0.0217 (10)	0.0290 (11)	−0.0016 (9)	0.0012 (9)	−0.0002 (9)
C6	0.0274 (11)	0.0242 (10)	0.0323 (11)	0.0034 (9)	0.0029 (9)	0.0036 (9)
C7	0.0260 (11)	0.0321 (12)	0.0320 (11)	0.0014 (9)	−0.0015 (9)	0.0090 (10)
C8	0.0330 (12)	0.0359 (13)	0.0270 (12)	0.0035 (10)	−0.0006 (9)	−0.0007 (10)
C9	0.0275 (11)	0.0325 (11)	0.0275 (10)	0.0020 (10)	0.0027 (9)	−0.0008 (9)
C10	0.0310 (12)	0.0444 (14)	0.0229 (11)	0.0053 (11)	0.0023 (9)	0.0021 (10)
C11	0.0378 (14)	0.0410 (14)	0.0281 (12)	−0.0037 (11)	0.0002 (10)	0.0050 (10)
C12	0.0194 (10)	0.0248 (10)	0.0316 (10)	−0.0036 (8)	−0.0001 (8)	−0.0024 (9)
C13	0.0221 (11)	0.0347 (12)	0.0374 (12)	−0.0030 (9)	0.0039 (9)	0.0053 (10)

Geometric parameters (Å, º)

Cl1—C2	1.792 (2)	C6—C7	1.380 (4)
Cl2—C2	1.778 (2)	C6—H6	0.97 (3)
F1—C7	1.362 (3)	C7—C8	1.374 (4)
O1—C3	1.410 (3)	C8—C9	1.392 (3)
O1—H1	0.82 (5)	C8—H8	1.01 (4)
O2—C1	1.410 (3)	C9—H9	0.96 (3)
O2—C10	1.440 (3)	C10—C11	1.495 (4)
O3—C1	1.392 (3)	C10—H10A	1.01 (3)
O3—C12	1.438 (3)	C10—H10B	1.01 (4)
C1—C2	1.549 (3)	C11—H11A	0.94 (3)
C1—H1A	0.99 (3)	C11—H11B	0.97 (4)
C2—C3	1.544 (3)	C11—H11C	0.99 (4)
C3—C4	1.518 (3)	C12—C13	1.505 (3)
C3—H3	1.02 (3)	C12—H12A	1.01 (3)
C4—C5	1.391 (3)	C12—H12B	1.03 (3)
C4—C9	1.397 (3)	C13—H13A	0.97 (3)
C5—C6	1.389 (3)	C13—H13B	0.97 (4)
C5—H5	0.93 (3)	C13—H13C	0.98 (3)
C3—O1—H1	112 (3)	C8—C7—C6	123.3 (2)
C1—O2—C10	117.08 (18)	C7—C8—C9	117.7 (2)
C1—O3—C12	113.31 (17)	C7—C8—H8	120 (2)
O3—C1—O2	108.00 (16)	C9—C8—H8	122 (2)

O3—C1—C2	105.90 (17)	C8—C9—C4	121.2 (2)
O2—C1—C2	112.15 (17)	C8—C9—H9	119.4 (18)
O3—C1—H1A	111.9 (18)	C4—C9—H9	119.4 (18)
O2—C1—H1A	110.7 (18)	O2—C10—C11	108.5 (2)
C2—C1—H1A	108.2 (18)	O2—C10—H10A	108.3 (18)
C3—C2—C1	111.72 (17)	C11—C10—H10A	112.6 (17)
C3—C2—Cl2	109.13 (16)	O2—C10—H10B	110 (2)
C1—C2—Cl2	109.77 (14)	C11—C10—H10B	110 (2)
C3—C2—Cl1	110.15 (15)	H10A—C10—H10B	108 (3)
C1—C2—Cl1	107.12 (15)	C10—C11—H11A	111 (2)
Cl2—C2—Cl1	108.90 (11)	C10—C11—H11B	108 (2)
O1—C3—C4	112.01 (18)	H11A—C11—H11B	113 (3)
O1—C3—C2	105.07 (18)	C10—C11—H11C	108 (2)
C4—C3—C2	113.98 (17)	H11A—C11—H11C	105 (3)
O1—C3—H3	110.7 (17)	H11B—C11—H11C	111 (3)
C4—C3—H3	109.4 (17)	O3—C12—C13	107.75 (19)
C2—C3—H3	105.5 (18)	O3—C12—H12A	109.9 (16)
C5—C4—C9	118.8 (2)	C13—C12—H12A	110.4 (16)
C5—C4—C3	120.3 (2)	O3—C12—H12B	108.3 (18)
C9—C4—C3	120.9 (2)	C13—C12—H12B	110.6 (18)
C6—C5—C4	121.1 (2)	H12A—C12—H12B	110 (2)
C6—C5—H5	119.4 (17)	C12—C13—H13A	110.0 (19)
C4—C5—H5	119.6 (17)	C12—C13—H13B	107 (2)
C7—C6—C5	118.0 (2)	H13A—C13—H13B	111 (3)
C7—C6—H6	117.2 (19)	C12—C13—H13C	109.7 (18)
C5—C6—H6	124.9 (19)	H13A—C13—H13C	107 (3)
F1—C7—C8	119.0 (2)	H13B—C13—H13C	112 (3)
F1—C7—C6	117.8 (2)		

C12—O3—C1—O2	−80.7 (2)	O1—C3—C4—C5	−28.8 (3)
C12—O3—C1—C2	158.94 (17)	C2—C3—C4—C5	90.3 (2)
C10—O2—C1—O3	145.47 (19)	O1—C3—C4—C9	149.9 (2)
C10—O2—C1—C2	−98.2 (2)	C2—C3—C4—C9	−91.0 (2)
O3—C1—C2—C3	−49.1 (2)	C9—C4—C5—C6	1.1 (3)
O2—C1—C2—C3	−166.62 (17)	C3—C4—C5—C6	179.8 (2)
O3—C1—C2—Cl2	72.14 (18)	C4—C5—C6—C7	−0.5 (3)
O2—C1—C2—Cl2	−45.4 (2)	C5—C6—C7—F1	179.7 (2)
O3—C1—C2—Cl1	−169.77 (14)	C5—C6—C7—C8	−0.1 (4)
O2—C1—C2—Cl1	72.67 (18)	F1—C7—C8—C9	−179.8 (2)
C1—C2—C3—O1	−52.0 (2)	C6—C7—C8—C9	−0.1 (4)
Cl2—C2—C3—O1	−173.54 (14)	C7—C8—C9—C4	0.7 (4)
Cl1—C2—C3—O1	66.95 (19)	C5—C4—C9—C8	−1.2 (3)
C1—C2—C3—C4	−174.97 (18)	C3—C4—C9—C8	−179.9 (2)
Cl2—C2—C3—C4	63.5 (2)	C1—O2—C10—C11	−167.2 (2)
Cl1—C2—C3—C4	−56.0 (2)	C1—O3—C12—C13	−172.42 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82 (5)	2.17 (5)	2.975 (2)	165 (4)
O1—H1···O3^{i}	0.82 (5)	2.43 (5)	3.046 (2)	133 (4)

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