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Syntheses and structures of 1-[2,2-dichloro-1-hydroxy-3-(4-methylphenyl)-3-oxopropyl]urea and 1-[2,2-dichloro-3-(4-fluorophenyl)-1-hydroxy-3-oxopropyl]urea

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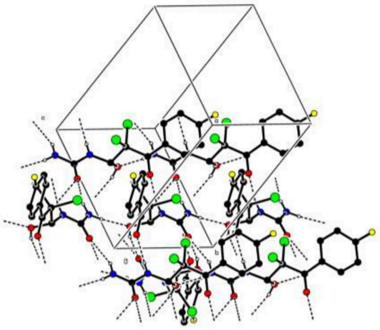
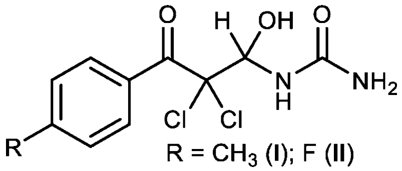
The title compounds, $C_{10}H_9Cl_2FN_2O_3$, (**I**), and $C_{11}H_{12}Cl_2N_2O_3$, (**II**), are α,α-dihalo-β-diketone urea derivatives, which contain 4-fluorophenyl and *p*-tolyl groups, respectively. The conformation about the $C_O—C_{Cl2}—C_O—N_u$ (O = keto, Cl2 = dichloro, u = urea) bond is *anti* in (**I**) and *gauche* in (**II**). In the crystals of both compounds, O—H⋯O hydrogen bonds generate inversion dimers and the dimers are linked into (100) layers by N—H⋯O hydrogen bonds. The Hirshfeld surface analyses of the crystal structures indicate that the most important contributions for the crystal packings are from H⋯O/O⋯H (22.3%), H⋯H (20.9%), H⋯Cl/Cl⋯H (15.6%) and H⋯C/C⋯H (10.3%) for (I) and H⋯H (31.7%), H⋯O/O⋯H (25.1%), H⋯Cl/Cl⋯H (21.1%) and H⋯C/C⋯H (9.5%) for (II).



1. Chemical context

The replacement of hydrogen atoms with halogen atoms at the active methylene group in β-diketones prevents keto–enol tautomerism and impacts on the reactivity of the corresponding α,α-dihalo-β- diketones (*e.g.*, Guseinov *et al.*, 2006). For instance, the reaction of α,α-dihalo-β-oxoaldehydes and their derivatives with N-nucleophilic reagents lead to hetero- or macrocyclic compounds (Guseinov *et al.*, 2024). This class of compounds can be used in the spectrophotometric determination of metal ions (Aliyev *et al.*, 2020), decoration of the secondary coordination sphere of metal complexes for catalysis (Aliyeva *et al.*, 2024), crystal growth and design (Naghiyev *et al.*, 2023) and heterogenous catalysis (Mahmudov *et al.*, 2022). In fact, the use of N-compounds has many synthetic advantages (Khalilov, 2021), such as easy modification and functionalization (Huseynov *et al.*, 2021), immobilization on solid materials through supramolecular interactions (Mamedov *et al.*, 2006), and crystal engineering (Hajiyeva *et al.*, 2024).



Herein, we describe the syntheses and crystal structures of the two title compounds, $C_{10}H_9Cl_2FN_2O_3$ (**I**) and

$C_{11}H_{12}Cl_2N_2O_3$ (**II**), which differ in the substituent at the *para* position of the phenyl group.

2. Structural commentary

In (**I**) (Fig. 1), the dihedral angle between the C7–C12 phenyl group and the C2/N1/N3/O2 urea moiety is 65.19 (8)°. The key torsion angles for the backbone of the molecule are C7—C6—C5—C4 = −179.65 (13), C6—C5—C4—N3 = −171.20 (12), C4—C5—C6—O6 = 2.95 (19) and C6—C5—C4—O4 = 66.65 (15)°. Atom C4 is a stereogenic centre: in the arbitrarily chosen asymmetric unit it has *R* configuration, but crystal symmetry generates a racemic mixture. Atoms F1, C6, C5 are displaced by −0.0244 (11), −0.0602 (15) and −0.0879 (15) Å, respectively, from the plane of the phenyl group. The N1—C2—O2, N3—C4—O4, N3—C4—C5, Cl1—C5—Cl2 and C4—C5—C6 bond angles in (**I**) are enlarged, while the O2—C2—N3 bond angle in (**I**) is narrowed compared to the corresponding values in (**II**): these small differences might arise due to steric reasons or 'packing effects'.

In (**II**) (Fig. 1), the corresponding dihedral angle between the C7–C12 and C2/N1/N3/O2 planes is 62.70 (9)° and the equivalent backbone torsion angles are C7—C6—C5—C4 = 162.65 (14), C6—C5—C4—N3 = −62.41 (16), C4—C5—C6—O6 = −17.42 (19) and C6—C5—C4—O4 = 176.34 (12)°. Thus it may be seen that the conformation of the atoms about the C4—C5 bond is quite different in the two structures. The stereogenic atom C4 in (**II**) was arbitrarily assigned to have an *R* configuration, but crystal symmetry generates a racemic mixture.

3. Supramolecular features

In the crystals of both compounds, O—H⋯O hydrogen bonds (Tables 1 and 2) generate inversion dimers featuring $R_2^2(12)$ loops. In both structures, N—H⋯O hydrogen bonds link the dimers into (100) layers, although they are not isostuctural. The hydrogen-bond network encloses $R_3^3(14)$ loops in (**I**) (Fig. 2*a*) and $R_2^2(8)$ and $R_3^3(8)$ loops in (**II**) (Fig. 2*b*).

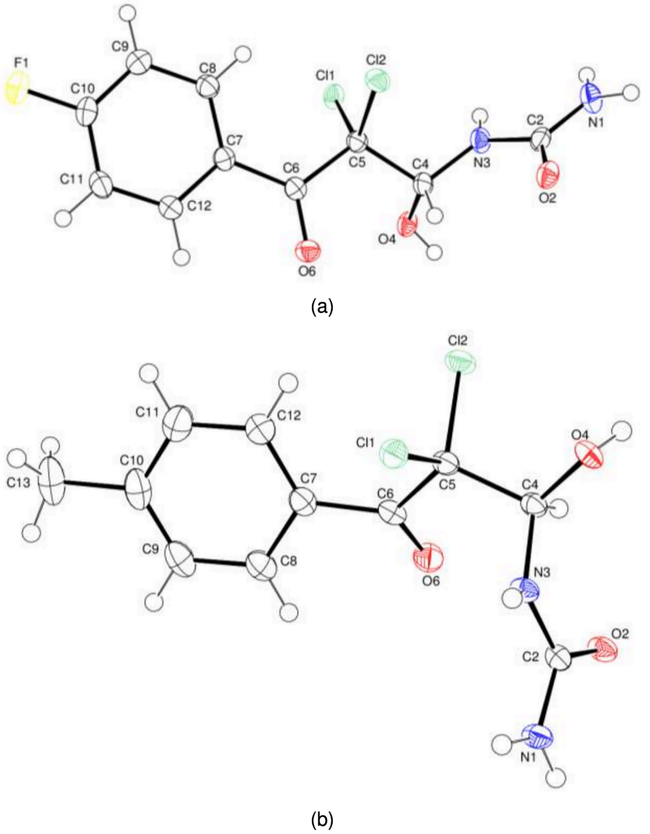


Figure 1
The asymmetric units of compounds (*a*) (**I**) and (*b*) (**II**) with 50% probability ellipsoids.

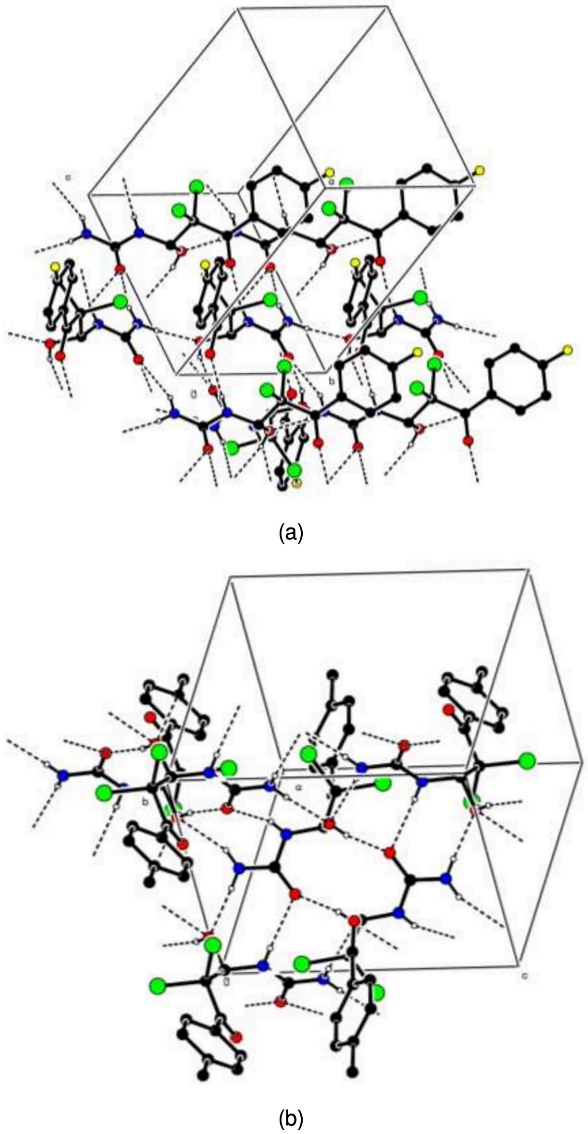


Figure 2
The partial packing diagrams of compounds (*a*) (**I**) and (*b*) (**II**). Intermolecular O—H⋯O and N—H⋯O hydrogen bonds are shown as dashed lines. H atoms not involved in these interactions have been omitted for clarity.

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯O4^{i}	0.90 (3)	2.06 (3)	2.9488 (19)	172 (2)
N1—H1B⋯O2^{ii}	0.86 (2)	2.48 (2)	3.297 (2)	158 (2)
N3—H3⋯O6^{iii}	0.87 (2)	2.06 (2)	2.9055 (18)	167 (2)
O4—H4⋯O2^{iv}	0.78 (3)	1.91 (3)	2.6596 (16)	161 (3)

Symmetry codes: (i) $x, y+1, z$; (ii) $x, -y+\frac{5}{2}, z-\frac{1}{2}$; (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $-x+2, -y+2, -z+1$.

Table 2
Hydrogen-bond geometry (Å, °) for (**II**).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯O4^{i}	0.82 (3)	2.27 (3)	3.0261 (19)	153 (2)
N1—H1B⋯O4^{ii}	0.84 (3)	2.14 (3)	2.9798 (18)	177 (2)
N3—H3⋯O2^{iii}	0.81 (3)	2.19 (3)	2.9445 (18)	156 (2)
O4—H4⋯O2^{iv}	0.78 (3)	1.83 (3)	2.5979 (16)	168 (3)

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

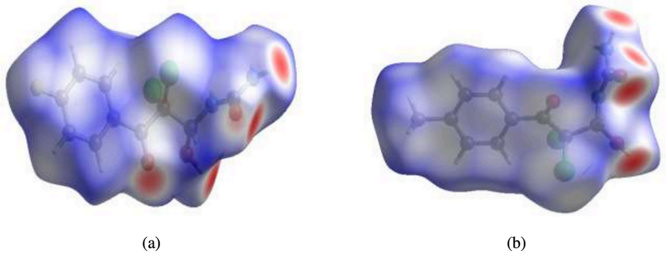

(a) (b)

Figure 3
Views of the three-dimensional Hirshfeld surfaces of compounds (*a*) (**I**) and (*b*) (**II**) plotted over $d_{\rm norm}$.

Table 3
Comparison of the fingerprint percentages for compounds (**I**) and (**II**).

Contacts	(**I**)	(**II**)
H⋯O/O⋯H	22.3	25.1
H⋯H	20.9	31.7
H⋯Cl/Cl⋯H	15.6	21.1
H⋯C/C⋯H	10.3	9.5
H⋯F/F⋯H	8.3	–
C⋯Cl/Cl⋯C	6.4	2.8
C⋯F/F⋯C	3.1	–
F⋯Cl/Cl⋯F	2.8	–
Cl⋯Cl	2.4	1.9
H⋯N/N⋯H	2.4	1.3
C⋯C	1.8	3.0
O⋯O	1.4	–
F⋯F	0.7	–
O⋯Cl/Cl⋯O	0.7	0.2
C⋯O/O⋯C	0.6	1.9
N⋯O/O⋯N	0.3	0.1
C⋯N/N⋯C	0.1	–
N⋯Cl/Cl⋯N	0.1	1.3

4. Hirshfeld surface analysis

For visualizing the intermolecular interactions in the crystals of (**I**) and (**II**), Hirshfeld surface (HS) analyses were carried out using *Crystal Explorer 17.5* (Spackman *et al.*, 2021). In the HSs plotted over $d_{\rm norm}$ (Fig. 3*a* and *b*), the contact distances equal, shorter and longer with respect to the sum of van der Waals radii are shown by the white, red and blue colours, respectively. According to the two-dimensional fingerprint plots, H⋯O/O⋯H, H⋯H and H⋯Cl/Cl⋯H contacts make the most important contributions to the HSs (Table 3, Figs. 4

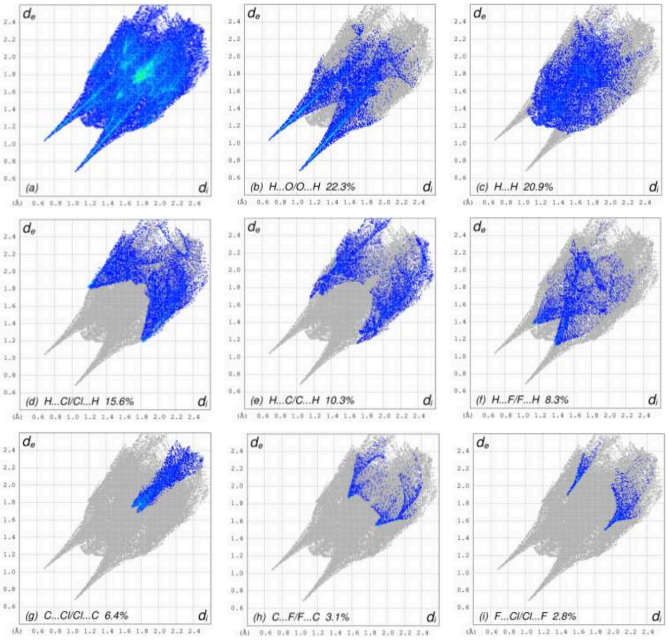


Figure 4
The two-dimensional fingerprint plots for compound (**I**), showing (*a*) all interactions, and delineated into different contact types (*b*)–(i). The $d_{\rm i}$ and $d_{\rm e}$ values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

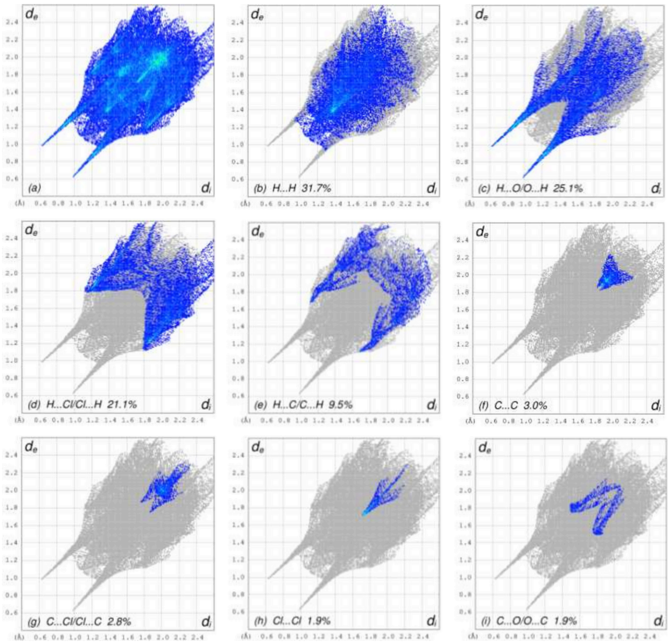


Figure 5
The two-dimensional fingerprint plots for compound (**II**), showing (*a*) all interactions, and delineated into different contact types (*b*)–(i). The $\rm d_i$ and $\rm d_e$ values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

Table 4
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{10}H_9Cl_2FN_2O_3$	$C_{11}H_{12}Cl_2N_2O_3$
M_r	295.09	291.13
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Temperature (K)	100	100
a, b, c (Å)	15.6136 (1), 7.2214 (1), 10.7929 (1)	12.62183 (13), 8.77585 (9), 11.59930 (11)
β (°)	104.622 (1)	94.8294 (9)
V (Å^3)	1177.51 (2)	1280.26 (2)
Z	4	4
Radiation type	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	5.14	4.60
Crystal size (mm)	0.32 × 0.15 × 0.05	0.45 × 0.35 × 0.16
Data collection		
Diffractometer	XtaLAB Synergy, Dualflex, HyPix	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2024)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2024)
T_{min}, T_{max}	0.408, 1.000	0.277, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16149, 2562, 2494	17344, 2808, 2738
R_{int}	0.035	0.050
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.640	0.640
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.082, 1.06	0.037, 0.104, 1.06
No. of reflections	2562	2808
No. of parameters	179	180
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.31	0.51, −0.43

Computer programs: *CrysAlis PRO* (Rigaku OD, 2024), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

and 5), and they have significant differences due to the different numbers and values of the close contacts in (**I**) and (**II**).

5. Synthesis and crystallization

A solution of 2,2-dichloro-3-oxo-3-(p-tolyl)propanal (231 mg) for (**I**) or 2,2-dichloro-3-(4-fluorophenyl)-3-oxopropanal (235 mg) for (**II**) and urea (60 mg) in 15 ml of dry acetonitrile was stirred at room temperature for 6 h. The solvent was removed *in vacuo*, and the remaining white powder was recrystallized from acetone solution and the title compounds were isolated. The reaction scheme is shown in Fig. 6.

(**I**): yield 82%; m.p. 378–380 K. Analysis calculated (%) for $C_{11}H_{12}Cl_2N_2O_3$: C 45.38, H 4.15, N 9.62; found C 45.36, H 4.11, N 9.60. ^{1}H NMR (300MHz, DMSO-d_6): 2.41 (*s*, CH_3), 5.94 (*s*, 2H, NH_2), 6.01–6.08 (*d.d*, CH), 6.72 (*d*, OH), 6.85 (*d*. NH), 7.38 (*d*. 2H, Ar), 7.95 (*d*, 2H, Ar). ^{13}C NMR (75 MHz, DMSO-d_6): 21.32, 88.03, 104.05, 128.74, 128.91, 133.77, 142.81, 162.72, 186.95.

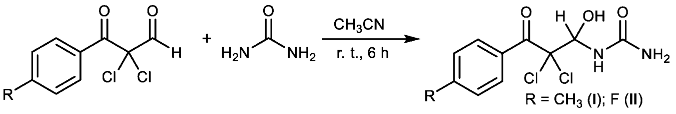


Figure 6
The synthesis of the title compounds.

(**II**): yield 78%; m.p. 397–398 K. Analysis calculated (%) for $C_{10}H_9Cl_2FN_2O_3$: C 40.70, H 3.07, N 9.49; found C 40.65, H 3.02, N 9.45. ^{1}H NMR (300MHz, DMSO-d_6): 5.96 (*s*, 2H, NH_2), 6.03–6.08 (*d.d*, CH), 6.78 (d, OH), 6.93 (*d*, NH), 7.46 (*t*, 2H, Ar), 8.12 (*d.d*, 2H, Ar). ^{13}C NMR (75 MHz, DMSO-d_6): 88.10, 104.12, 115.46, 130.49, 132.37, 162.74, 167.35, 186.92.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. The OH and NH_2 hydrogen atoms were located in difference-Fourier maps, and refined isotropically. The C-bond hydrogen-atom positions were placed geometrically (C—H = 0.95–1.00 Å) and refined using a riding model by applying the constraint $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

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supporting information

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Syntheses and structures of 1-[2,2-dichloro-1-hydroxy-3-(4-methylphenyl)-3-oxopropyl]urea and 1-[2,2-dichloro-3-(4-fluorophenyl)-1-hydroxy-3-oxopropyl]-urea

Firudin I. Guseinov, Tuncer Hökelek, Ksenia A. Afanaseva, Elena V. Shuvalova, Lev M. Glukhov, Aida I. Samigullina and Alebel N. Belay

Computing details

1-[2,2-Dichloro-1-hydroxy-3-(4-methylphenyl)-3-oxopropyl]urea (I)

Crystal data

$C_{10}H_9Cl_2FN_2O_3$
$M_r = 295.09$
Monoclinic, $P2_1/c$
$a = 15.6136$ (1) Å
$b = 7.2214$ (1) Å
$c = 10.7929$ (1) Å
$\beta = 104.622$ (1)°
$V = 1177.51$ (2) Å^3
$Z = 4$

$F(000) = 600$
$D_x = 1.665$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 10775 reflections
$\theta = 2.9$–80.5°
$\mu = 5.14$ mm^{-1}
$T = 100$ K
Prism, colorless
0.32 × 0.15 × 0.05 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2024)

$T_{\min} = 0.408$, $T_{\max} = 1.000$
16149 measured reflections
2562 independent reflections
2494 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.035$
$\theta_{\max} = 80.7°$, $\theta_{\min} = 2.9°$
$h = -19 \rightarrow 19$
$k = -9 \rightarrow 9$
$l = -10 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.082$
$S = 1.06$
2562 reflections
179 parameters
0 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0418P)^2 + 0.7639P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}

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	0.75812 (2)	0.64065 (5)	0.20981 (3)	0.02166 (11)
Cl2	0.71108 (2)	0.90786 (5)	0.38664 (3)	0.02237 (11)
F1	0.45932 (7)	0.18867 (16)	0.44559 (10)	0.0321 (2)
O2	0.91429 (7)	1.16468 (16)	0.49959 (11)	0.0231 (2)
O4	0.93636 (8)	0.66517 (16)	0.38219 (11)	0.0216 (2)
H4	0.9848 (18)	0.692 (4)	0.417 (2)	0.038 (7)*
O6	0.83823 (7)	0.53628 (16)	0.55147 (10)	0.0220 (2)
N1	0.91321 (10)	1.2742 (2)	0.30188 (16)	0.0279 (3)
H1A	0.9206 (17)	1.389 (4)	0.334 (2)	0.040 (7)*
H1B	0.9071 (15)	1.259 (4)	0.221 (2)	0.032 (6)*
N3	0.88523 (9)	0.96745 (19)	0.32966 (13)	0.0222 (3)
H3	0.8796 (15)	0.958 (4)	0.248 (2)	0.035 (6)*
C2	0.90498 (10)	1.1385 (2)	0.38332 (16)	0.0204 (3)
C4	0.87990 (10)	0.8058 (2)	0.40405 (14)	0.0195 (3)
H4A	0.896975	0.839528	0.496915	0.023*
C5	0.78496 (10)	0.7248 (2)	0.37028 (13)	0.0185 (3)
C6	0.77450 (10)	0.5688 (2)	0.46381 (14)	0.0178 (3)
C7	0.68898 (10)	0.4691 (2)	0.45000 (14)	0.0187 (3)
C8	0.61114 (10)	0.5077 (2)	0.35608 (15)	0.0220 (3)
H8	0.611405	0.600113	0.293538	0.026*
C9	0.53355 (11)	0.4121 (2)	0.35358 (16)	0.0239 (3)
H9	0.480694	0.437166	0.289650	0.029*
C10	0.53522 (11)	0.2797 (2)	0.44634 (16)	0.0238 (3)
C11	0.61052 (11)	0.2368 (2)	0.54016 (16)	0.0261 (3)
H11	0.609462	0.144287	0.602320	0.031*
C12	0.68768 (11)	0.3321 (2)	0.54142 (15)	0.0228 (3)
H12	0.740343	0.304306	0.605008	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02614 (19)	0.0226 (2)	0.01572 (18)	−0.00771 (14)	0.00426 (13)	−0.00293 (12)
Cl2	0.02389 (19)	0.01773 (19)	0.0245 (2)	0.00268 (13)	0.00429 (14)	0.00032 (13)
F1	0.0255 (5)	0.0325 (6)	0.0395 (6)	−0.0097 (4)	0.0100 (4)	0.0007 (5)
O2	0.0221 (5)	0.0199 (6)	0.0272 (6)	−0.0036 (4)	0.0059 (4)	−0.0061 (4)
O4	0.0204 (5)	0.0172 (5)	0.0271 (6)	−0.0031 (4)	0.0057 (4)	−0.0057 (4)
O6	0.0226 (5)	0.0229 (6)	0.0188 (5)	0.0006 (4)	0.0023 (4)	0.0011 (4)
N1	0.0359 (8)	0.0167 (7)	0.0321 (8)	−0.0040 (6)	0.0103 (6)	−0.0020 (6)
N3	0.0285 (7)	0.0174 (7)	0.0200 (6)	−0.0056 (5)	0.0046 (5)	−0.0023 (5)

C2	0.0147 (6)	0.0177 (7)	0.0281 (8)	−0.0015 (5)	0.0041 (6)	−0.0038 (6)
C4	0.0219 (7)	0.0151 (7)	0.0210 (7)	−0.0024 (6)	0.0043 (5)	−0.0024 (6)
C5	0.0214 (7)	0.0158 (7)	0.0171 (6)	−0.0011 (6)	0.0029 (5)	−0.0017 (5)
C6	0.0215 (7)	0.0160 (7)	0.0164 (7)	0.0001 (5)	0.0057 (5)	−0.0024 (5)
C7	0.0218 (7)	0.0165 (7)	0.0186 (7)	−0.0008 (6)	0.0063 (5)	−0.0019 (5)
C8	0.0239 (7)	0.0211 (7)	0.0204 (7)	−0.0011 (6)	0.0045 (6)	0.0014 (6)
C9	0.0219 (7)	0.0254 (8)	0.0237 (7)	−0.0007 (6)	0.0042 (6)	−0.0014 (6)
C10	0.0236 (7)	0.0207 (8)	0.0285 (8)	−0.0058 (6)	0.0093 (6)	−0.0045 (6)
C11	0.0297 (8)	0.0227 (8)	0.0269 (8)	−0.0032 (7)	0.0088 (6)	0.0052 (6)
C12	0.0233 (7)	0.0210 (8)	0.0231 (7)	−0.0001 (6)	0.0040 (6)	0.0028 (6)

Geometric parameters (Å, º)

Cl1—C5	1.7826 (14)	C4—C5	1.549 (2)
Cl2—C5	1.7923 (16)	C5—C6	1.549 (2)
F1—C10	1.3534 (18)	C6—C7	1.491 (2)
O2—C2	1.241 (2)	C7—C8	1.400 (2)
O4—H4	0.78 (3)	C7—C12	1.401 (2)
O4—C4	1.4032 (19)	C8—H8	0.9500
O6—C6	1.2103 (19)	C8—C9	1.389 (2)
N1—H1A	0.90 (3)	C9—H9	0.9500
N1—H1B	0.86 (2)	C9—C10	1.380 (2)
N1—C2	1.344 (2)	C10—C11	1.379 (2)
N3—H3	0.87 (2)	C11—H11	0.9500
N3—C2	1.366 (2)	C11—C12	1.385 (2)
N3—C4	1.431 (2)	C12—H12	0.9500
C4—H4A	1.0000		

C4—O4—H4	108.1 (19)	O6—C6—C5	116.71 (13)
H1A—N1—H1B	119 (2)	O6—C6—C7	121.55 (14)
C2—N1—H1A	116.4 (17)	C7—C6—C5	121.69 (13)
C2—N1—H1B	124.6 (17)	C8—C7—C6	124.60 (14)
C2—N3—H3	117.2 (17)	C8—C7—C12	119.10 (14)
C2—N3—C4	122.60 (14)	C12—C7—C6	116.26 (14)
C4—N3—H3	120.1 (17)	C7—C8—H8	119.7
O2—C2—N1	122.99 (15)	C9—C8—C7	120.56 (15)
O2—C2—N3	121.50 (15)	C9—C8—H8	119.7
N1—C2—N3	115.50 (15)	C8—C9—H9	120.9
O4—C4—N3	111.59 (13)	C10—C9—C8	118.28 (15)
O4—C4—H4A	109.0	C10—C9—H9	120.9
O4—C4—C5	106.95 (12)	F1—C10—C9	118.43 (15)
N3—C4—H4A	109.0	F1—C10—C11	118.56 (15)
N3—C4—C5	111.33 (13)	C11—C10—C9	123.01 (15)
C5—C4—H4A	109.0	C10—C11—H11	120.9
Cl1—C5—Cl2	110.35 (8)	C10—C11—C12	118.29 (15)
C4—C5—Cl1	109.49 (10)	C12—C11—H11	120.9
C4—C5—Cl2	107.43 (10)	C7—C12—H12	119.6
C6—C5—Cl1	110.36 (10)	C11—C12—C7	120.75 (15)

C6—C5—Cl2	107.22 (10)	C11—C12—H12	119.6
C6—C5—C4	111.93 (12)		

Cl1—C5—C6—O6	125.15 (13)	C4—N3—C2—O2	−4.3 (2)
Cl1—C5—C6—C7	−57.44 (16)	C4—N3—C2—N1	175.81 (14)
Cl2—C5—C6—O6	−114.62 (13)	C4—C5—C6—O6	2.95 (19)
Cl2—C5—C6—C7	62.78 (15)	C4—C5—C6—C7	−179.65 (13)
F1—C10—C11—C12	−179.17 (15)	C5—C6—C7—C8	−1.6 (2)
O4—C4—C5—Cl1	−56.04 (14)	C5—C6—C7—C12	−179.34 (13)
O4—C4—C5—Cl2	−175.90 (10)	C6—C7—C8—C9	−177.53 (15)
O4—C4—C5—C6	66.65 (15)	C6—C7—C12—C11	177.33 (15)
O6—C6—C7—C8	175.69 (15)	C7—C8—C9—C10	0.5 (2)
O6—C6—C7—C12	−2.1 (2)	C8—C7—C12—C11	−0.5 (2)
N3—C4—C5—Cl1	66.10 (14)	C8—C9—C10—F1	178.80 (15)
N3—C4—C5—Cl2	−53.75 (14)	C8—C9—C10—C11	−0.8 (3)
N3—C4—C5—C6	−171.20 (12)	C9—C10—C11—C12	0.4 (3)
C2—N3—C4—O4	−124.46 (15)	C10—C11—C12—C7	0.3 (3)
C2—N3—C4—C5	116.12 (15)	C12—C7—C8—C9	0.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4^{i}	0.90 (3)	2.06 (3)	2.9488 (19)	172 (2)
N1—H1*B*···O2^{ii}	0.86 (2)	2.48 (2)	3.297 (2)	158 (2)
N3—H3···O6^{iii}	0.87 (2)	2.06 (2)	2.9055 (18)	167 (2)
O4—H4···O2^{iv}	0.78 (3)	1.91 (3)	2.6596 (16)	161 (3)

Symmetry codes: (i) $x, y+1, z$; (ii) $x, -y+5/2, z-1/2$; (iii) $x, -y+3/2, z-1/2$; (iv) $-x+2, -y+2, -z+1$.

1-[2,2-Dichloro-3-(4-fluorophenyl)-1-hydroxy-3-oxopropyl]urea (II)

Crystal data

$C_{11}H_{12}Cl_2N_2O_3$
$M_r = 291.13$
Monoclinic, $P2_1/c$
$a = 12.62183\ (13)$ Å
$b = 8.77585\ (9)$ Å
$c = 11.59930\ (11)$ Å
$\beta = 94.8294\ (9)°$
$V = 1280.26\ (2)$ Å^3
$Z = 4$

$F(000) = 600$
$D_x = 1.510$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 11708 reflections
$\theta = 3.5–80.3°$
$\mu = 4.60$ mm^{-1}
$T = 100$ K
Prism, colourless
0.45 × 0.35 × 0.16 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2024)

$T_{min} = 0.277$, $T_{max} = 1.000$
17344 measured reflections
2808 independent reflections
2738 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 80.7°$, $\theta_{min} = 3.5°$
$h = -13 \rightarrow 16$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.104$
$S = 1.06$
2808 reflections
180 parameters
0 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.6302P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.51$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
Cl1	0.22629 (3)	0.88241 (4)	0.40687 (3)	0.02066 (13)
Cl2	0.24819 (3)	0.70279 (5)	0.61915 (3)	0.02309 (13)
O2	0.02392 (10)	0.37516 (13)	0.31405 (10)	0.0221 (3)
O4	0.03286 (9)	0.72384 (13)	0.48926 (10)	0.0192 (2)
O6	0.27591 (10)	0.45882 (14)	0.39418 (11)	0.0259 (3)
N1	0.03826 (12)	0.49974 (17)	0.14562 (12)	0.0212 (3)
N3	0.08327 (11)	0.62287 (15)	0.31617 (12)	0.0180 (3)
C2	0.04683 (12)	0.49344 (18)	0.26185 (13)	0.0181 (3)
C4	0.10682 (12)	0.63031 (17)	0.43899 (13)	0.0176 (3)
H4A	0.103457	0.525355	0.472057	0.021*
C5	0.22071 (13)	0.69474 (17)	0.46569 (13)	0.0185 (3)
C6	0.30485 (13)	0.58690 (19)	0.41747 (13)	0.0204 (3)
C7	0.41441 (13)	0.6404 (2)	0.40165 (14)	0.0225 (3)
C8	0.46819 (15)	0.5634 (2)	0.31877 (16)	0.0279 (4)
H8	0.433483	0.484415	0.274165	0.033*
C9	0.57245 (15)	0.6024 (2)	0.30150 (17)	0.0321 (4)
H9	0.607516	0.551826	0.243057	0.039*
C10	0.62617 (15)	0.7138 (2)	0.36820 (18)	0.0325 (4)
C11	0.57243 (15)	0.7900 (2)	0.45141 (18)	0.0326 (4)
H11	0.608274	0.866470	0.497716	0.039*
C12	0.46717 (15)	0.7555 (2)	0.46742 (16)	0.0275 (4)
H12	0.431028	0.810101	0.523000	0.033*
C13	0.74130 (16)	0.7514 (3)	0.3535 (2)	0.0440 (5)
H13A	0.762750	0.702446	0.283178	0.066*
H13B	0.786149	0.713990	0.420656	0.066*
H13C	0.749543	0.862022	0.347188	0.066*
H1A	0.059 (2)	0.574 (3)	0.110 (2)	0.030 (6)*
H1B	0.0183 (19)	0.420 (3)	0.110 (2)	0.028 (6)*

H3	0.0725 (18)	0.701 (3)	0.280 (2)	0.023 (5)*
H4	0.014 (2)	0.683 (3)	0.543 (3)	0.034 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0239 (2)	0.0173 (2)	0.0211 (2)	−0.00221 (12)	0.00407 (14)	0.00018 (12)
Cl2	0.0263 (2)	0.0291 (2)	0.01385 (19)	0.00153 (14)	0.00155 (14)	−0.00215 (13)
O2	0.0313 (6)	0.0194 (6)	0.0164 (5)	−0.0060 (4)	0.0065 (4)	−0.0001 (4)
O4	0.0229 (5)	0.0207 (5)	0.0150 (5)	0.0016 (4)	0.0073 (4)	0.0021 (4)
O6	0.0270 (6)	0.0222 (6)	0.0290 (6)	0.0015 (5)	0.0057 (5)	−0.0049 (5)
N1	0.0304 (7)	0.0189 (6)	0.0144 (6)	−0.0061 (5)	0.0037 (5)	−0.0009 (5)
N3	0.0253 (6)	0.0152 (6)	0.0139 (6)	−0.0010 (5)	0.0032 (5)	0.0016 (5)
C2	0.0199 (7)	0.0179 (7)	0.0168 (7)	0.0003 (5)	0.0043 (5)	0.0001 (5)
C4	0.0217 (7)	0.0176 (7)	0.0139 (7)	−0.0012 (5)	0.0042 (5)	0.0008 (5)
C5	0.0225 (7)	0.0182 (7)	0.0150 (7)	−0.0007 (6)	0.0035 (5)	−0.0005 (5)
C6	0.0235 (7)	0.0226 (7)	0.0156 (7)	0.0027 (6)	0.0039 (5)	−0.0006 (6)
C7	0.0215 (7)	0.0258 (8)	0.0203 (7)	0.0026 (6)	0.0030 (6)	0.0021 (6)
C8	0.0277 (8)	0.0307 (9)	0.0258 (8)	0.0044 (7)	0.0061 (6)	−0.0006 (7)
C9	0.0279 (9)	0.0390 (10)	0.0308 (9)	0.0081 (8)	0.0105 (7)	0.0065 (7)
C10	0.0231 (8)	0.0377 (10)	0.0372 (10)	0.0018 (7)	0.0063 (7)	0.0162 (8)
C11	0.0269 (9)	0.0346 (10)	0.0358 (10)	−0.0041 (7)	0.0000 (7)	0.0040 (8)
C12	0.0263 (8)	0.0305 (9)	0.0260 (8)	−0.0010 (7)	0.0039 (6)	−0.0010 (7)
C13	0.0258 (10)	0.0529 (14)	0.0546 (13)	−0.0018 (9)	0.0104 (9)	0.0210 (11)

Geometric parameters (Å, º)

Cl1—C5	1.7864 (16)	C6—C7	1.486 (2)
Cl2—C5	1.7861 (16)	C7—C8	1.397 (2)
O2—C2	1.248 (2)	C7—C12	1.400 (3)
O4—C4	1.4060 (18)	C8—H8	0.9500
O4—H4	0.78 (3)	C8—C9	1.390 (3)
O6—C6	1.205 (2)	C9—H9	0.9500
N1—C2	1.345 (2)	C9—C10	1.388 (3)
N1—H1A	0.82 (3)	C10—C11	1.396 (3)
N1—H1B	0.84 (3)	C10—C13	1.514 (2)
N3—C2	1.360 (2)	C11—H11	0.9500
N3—C4	1.432 (2)	C11—C12	1.390 (3)
N3—H3	0.81 (3)	C12—H12	0.9500
C4—H4A	1.0000	C13—H13A	0.9800
C4—C5	1.551 (2)	C13—H13B	0.9800
C5—C6	1.561 (2)	C13—H13C	0.9800
C4—O4—H4	109 (2)	C8—C7—C6	116.30 (16)
C2—N1—H1A	121.9 (18)	C8—C7—C12	119.18 (16)
C2—N1—H1B	117.0 (16)	C12—C7—C6	124.44 (15)
H1A—N1—H1B	121 (2)	C7—C8—H8	120.0
C2—N3—C4	122.18 (13)	C9—C8—C7	120.07 (18)

C2—N3—H3	115.9 (17)	C9—C8—H8	120.0
C4—N3—H3	118.9 (17)	C8—C9—H9	119.4
O2—C2—N1	121.23 (15)	C10—C9—C8	121.10 (18)
O2—C2—N3	123.58 (14)	C10—C9—H9	119.4
N1—C2—N3	115.19 (14)	C9—C10—C11	118.69 (17)
O4—C4—N3	110.47 (13)	C9—C10—C13	121.3 (2)
O4—C4—H4A	109.1	C11—C10—C13	120.0 (2)
O4—C4—C5	109.93 (12)	C10—C11—H11	119.6
N3—C4—H4A	109.1	C12—C11—C10	120.89 (19)
N3—C4—C5	109.05 (12)	C12—C11—H11	119.6
C5—C4—H4A	109.1	C7—C12—H12	120.0
Cl2—C5—Cl1	109.48 (8)	C11—C12—C7	120.02 (17)
C4—C5—Cl1	108.99 (11)	C11—C12—H12	120.0
C4—C5—Cl2	108.20 (10)	C10—C13—H13A	109.5
C4—C5—C6	110.76 (13)	C10—C13—H13B	109.5
C6—C5—Cl1	111.85 (11)	C10—C13—H13C	109.5
C6—C5—Cl2	107.47 (11)	H13A—C13—H13B	109.5
O6—C6—C5	116.31 (14)	H13A—C13—H13C	109.5
O6—C6—C7	122.32 (15)	H13B—C13—H13C	109.5
C7—C6—C5	121.38 (14)		

Cl1—C5—C6—O6	−139.23 (13)	C4—N3—C2—N1	−173.78 (14)
Cl1—C5—C6—C7	40.85 (18)	C4—C5—C6—O6	−17.42 (19)
Cl2—C5—C6—O6	100.57 (15)	C4—C5—C6—C7	162.65 (14)
Cl2—C5—C6—C7	−79.35 (16)	C5—C6—C7—C8	−155.04 (15)
O4—C4—C5—Cl1	−60.19 (14)	C5—C6—C7—C12	28.2 (2)
O4—C4—C5—Cl2	58.79 (14)	C6—C7—C8—C9	−177.58 (17)
O4—C4—C5—C6	176.34 (12)	C6—C7—C12—C11	175.43 (17)
O6—C6—C7—C8	25.0 (2)	C7—C8—C9—C10	2.2 (3)
O6—C6—C7—C12	−151.71 (18)	C8—C7—C12—C11	−1.2 (3)
N3—C4—C5—Cl1	61.06 (14)	C8—C9—C10—C11	−1.9 (3)
N3—C4—C5—Cl2	−179.97 (10)	C8—C9—C10—C13	177.11 (18)
N3—C4—C5—C6	−62.41 (16)	C9—C10—C11—C12	−0.1 (3)
C2—N3—C4—O4	−110.90 (16)	C10—C11—C12—C7	1.6 (3)
C2—N3—C4—C5	128.18 (15)	C12—C7—C8—C9	−0.6 (3)
C4—N3—C2—O2	5.2 (2)	C13—C10—C11—C12	−179.03 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4$^{\text{i}}$	0.82 (3)	2.27 (3)	3.0261 (19)	153 (2)
N1—H1*B*···O4$^{\text{ii}}$	0.84 (3)	2.14 (3)	2.9798 (18)	177 (2)
N3—H3···O2$^{\text{iii}}$	0.81 (3)	2.19 (3)	2.9445 (18)	156 (2)
O4—H4···O2$^{\text{iv}}$	0.78 (3)	1.83 (3)	2.5979 (16)	168 (3)

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