

# A new triclinic polymorph of 6,6'- $\{$ (1*E*,1*E'*)- $\{$ (1,2-diphenylethane-1,2-diyl)bis(azaneylylidene)}-bis(methaneylylidene)}bis(2-chlorophenol)

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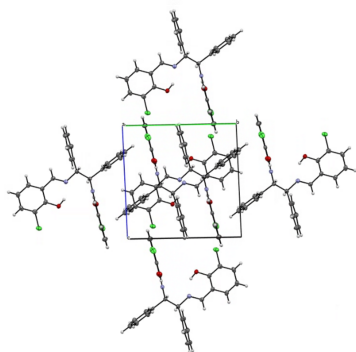
The redetermined structure of a new polymorph of 6,6'- $\{$ (1*E*,1*E'*)- $\{$ (1,2-diphenylethane-1,2-diyl)bis(azaneylylidene)}bis(methaneylylidene)}bis(2-chlorophenol), C<sub>28</sub>H<sub>22</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>2</sub> was reported. The title compound was synthesized under microwave irradiation at 90 K, within 10 min. The asymmetric unit comprises a potentially tetradentate Schiff base ligand that crystallizes in the triclinic system with space group  $P\bar{1}$ . The structure displays intramolecular O—H...N hydrogen bonding, forming an *S*(6) ring. Comparisons are made with the monoclinic polymorph [Shen *et al.* (2017). *RSC Adv.* **7**, 40640–40649].

## 1. Chemical context

Schiff base complexes have played a central role in the development of coordination chemistry, as evidenced by their vast number, ease and flexibility of synthetic procedures, diverse properties, and applications as bioactive compounds such as antitumor, antibacterial, antifungal, and various other biological applications. Numerous reports have shown that salen-type metal complexes exhibit high activity against various diseases, including cancer (Nworie *et al.*, 2016). Polymorphism is known to exist in crystals of Schiff base complex ligands and, as reported by Suda *et al.* (2021), in particular the angle of the phenyl group bonded to the nitrogen atom was different from that previously reported. Salen-type compounds are synthesized from diamines and salicylaldehyde and contain two azomethine groups that are used as organic ligands in complex formation (Akitsu *et al.*, 2011). In our laboratory, we have been studying salen-type metal complexes, which have the potential to be useful in a variety of applications, including as a new concept dye for dye-sensitized solar cells (DSSC), flame retardant in heat-stabilized PVC sheets and artificial metalloenzymes that mimic the catalytic efficiency of natural metalloenzymes (Yamane *et al.*, 2017; Soni *et al.*, 2020; Kashiwagi *et al.*, 2019). Salen-type metal complexes are known to exhibit ligand disorder (Akitsu *et al.*, 2005*b*). However, there are also examples of non-disordered salen-type metal complexes (Akitsu *et al.*, 2005*a*). In this report, we describe a new polymorphic crystal structure of the title compound.

## 2. Structural commentary

The title compound (Fig. 1) crystallizes in the triclinic system with  $P\bar{1}$  space group. The crystals obtained were racemic, with one molecule being an *RR* isomer (optically active). The previously reported crystal was monoclinic, had a space group

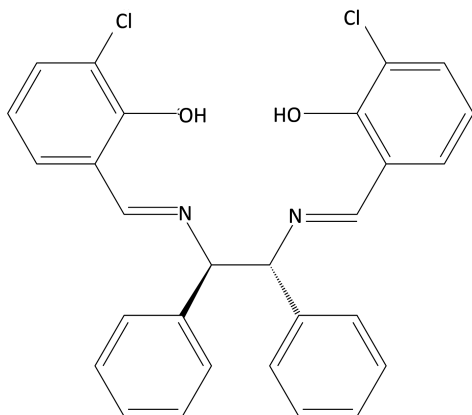


**Table 1**

Hydrogen-bond geometry (Å, °).

| <i>D</i> –H··· <i>A</i> | <i>D</i> –H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> –H··· <i>A</i> |
|-------------------------|-------------|---------------|-----------------------|-------------------------|
| O29–H29···N31           | 0.84        | 1.82          | 2.564 (3)             | 147                     |
| O30–H30···N32           | 0.84        | 1.85          | 2.597 (3)             | 147                     |

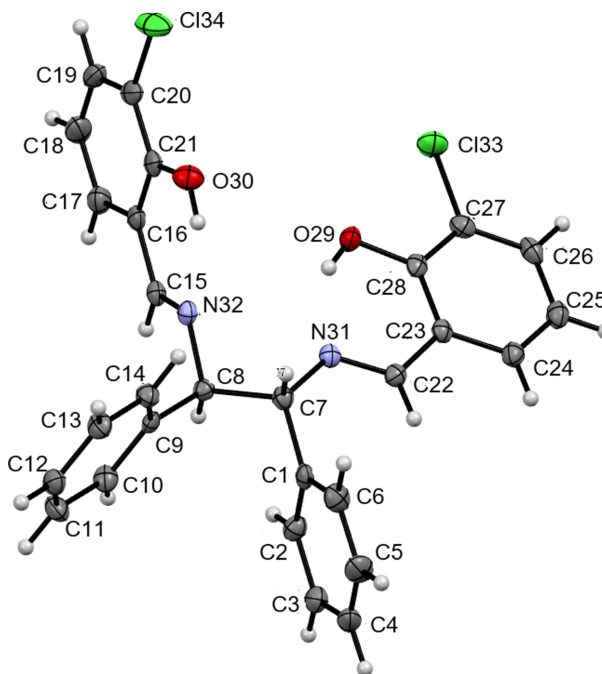
of *I2/a*, and was colorless and transparent (Shen *et al.*, 2017). Intramolecular hydrogen bonds with an *S*(6) (Bernstein *et al.*, 1995) ring formation are observed, with O···N distances of 2.564 (3) and 2.597 (3) Å (Table 1).



Comparing the bond distances, bond angles, and torsion angles with monoclinic polymorph the bond distances and bond angles are almost the same, but there are significant differences in the torsion angles. The torsion angles C6–C7–N31–C22 and C9–C8–N32–C15 are  $-22.8$  (3) and  $-105.8$  (2)°, respectively, in the title compound compared 108.3 (2)° in the monoclinic polymorph. In the title compound, N32–C8–C7–N31 is  $-50.7$  (2)° [43.3 (2) and  $-42.6$  (2)° in the monoclinic polymorph] while N32–C8–C9–C14 and N31–C7–C6–C1 are  $-60.7$  (3) and  $-72.1$  (3)°, respectively [43.2 (2) and  $-85.8$  (2)° in the monoclinic polymorph].

### 3. Supramolecular features

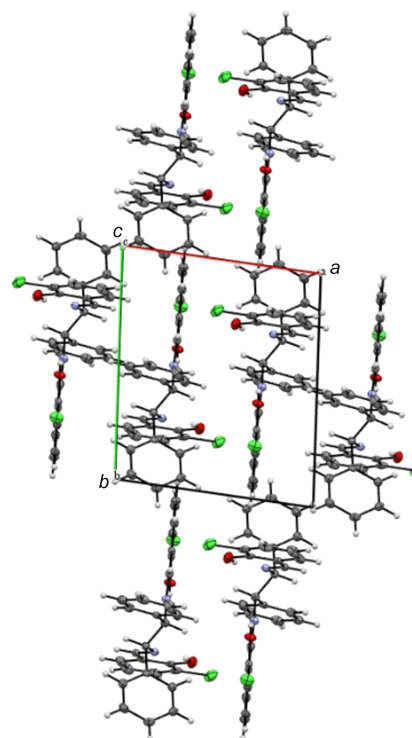
No intermolecular hydrogen bonds are observed in the crystal, but a Cl···Cl halogen interaction with length of 3.4122 (11) Å is found. There are two molecules in the unit cell. In contrast, the unit cell of the monoclinic polymorph contains 12 molecules, with an intermolecular O···Cl hydrogen bond of 3.176 Å in length, weak O···H interactions (2.688 and 2.570 Å) and a C···Cl interaction (3.225 Å; Shen *et al.*, 2017). An interesting feature of the crystal packing (Figs. 2–4) of the title compound is the presence of centrosymmetric Cl33···Cl34 [3.4122 (11) Å] and *n*··· $\pi^*$  (Echeverriá *et al.*, 2018) [O30···C22 = 3.029 (3) Å] interactions, which are shorter than sum of the van der Waals radii of the involved atoms (Bondi *et al.*, 1964). These interactions are supported by a short C4···C19 [3.361 (3) Å] contact, forming a one-dimensional extended chain of neighboring molecules parallel to the *bc*-plane.



**Figure 1**

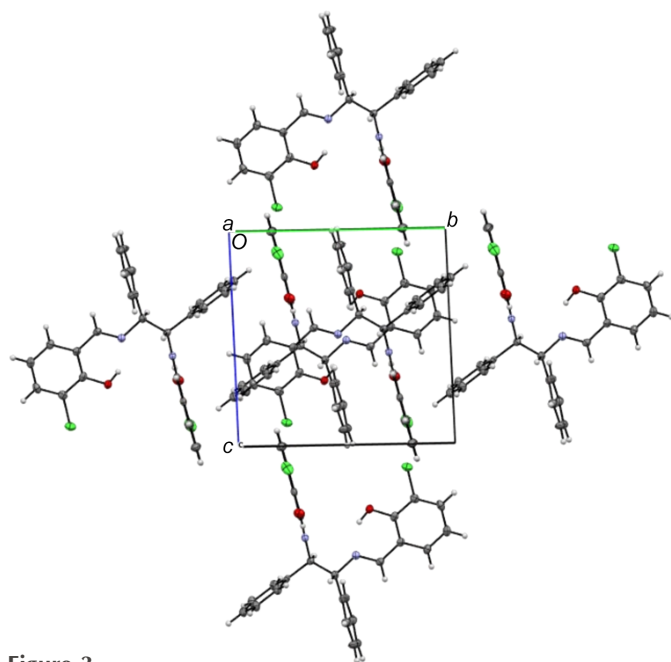
The title compound with ellipsoids drawn at the 50% probability level.

A Hirshfeld surface analysis (McKinnon *et al.*, 2004) was performed to further investigate the intermolecular interactions and contacts using *Crystal Explorer 17.5* (McKinnon *et al.*, 2007; Turner *et al.*, 2017) (Fig. 5). It indicates that the most important contributions to the packing are from H···H (35.2%), C···H/H···C (18.4%) and Cl···H/H···Cl (10.3%)



**Figure 2**

Packing of the title compound, viewed along the *b*-axis direction.



**Figure 3**  
Packing of the title compound viewed along the *a*-axis direction.

contacts. The intermolecular C—H...C hydrogen bonds are indicated by bright-red spots on the Hirshfeld surfaces mapped over  $d_{\text{norm}}$  and by two sharp spikes of almost the same length in the region  $1.6 \text{ \AA} < (d_c + d_i) < 2.4 \text{ \AA}$  in the 2D finger plots (Fig. 3).

The contributions to the packing from H...H, C...C, C...H/H...C, Cl...H/H...Cl, N...H/H...N, and H...O/O...H contacts are 35.2, 1.7, 33.0, 16.5, 1.4 and 8.6%, respectively. The structure is characterized by a high proportion of H...H interactions, which are van der Waals interactions. The high C...H/H...C value is due to the presence of aromatic rings in the compound. The low C...C value is due to the lack of overlapping aromatic rings in the structure.

#### 4. Database survey

A search in the Cambridge Structural Database (CSD, Version 5.41, update of January 2024; Groom *et al.* 2016) for similar structures gave 6,6'-((1*E*,1*E'*)-[(1,2-diphenylethane-1,2-diyl)-

**Table 2**

Experimental details.

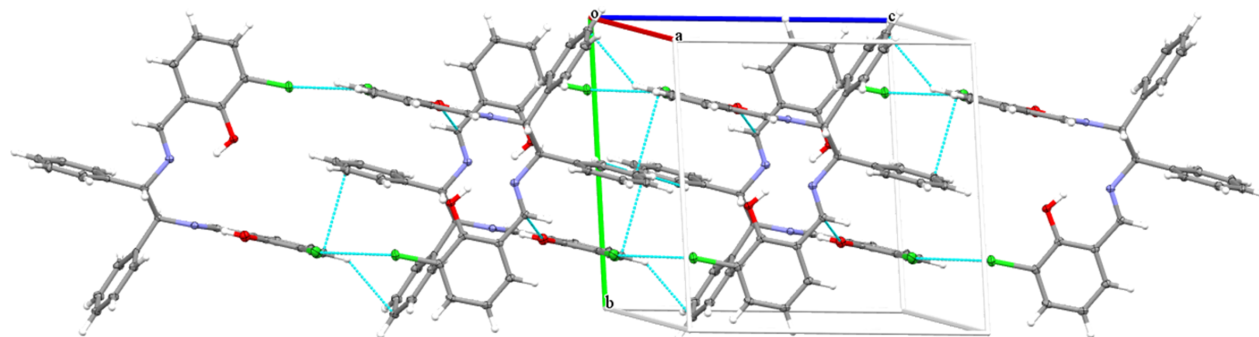
|  |  |
|--|--|
| Crystal data   |  |
| Chemical formula   | $\text{C}_{28}\text{H}_{22}\text{Cl}_2\text{N}_2\text{O}_2$            |
| $M_r$  | 489.37   |
| Crystal system, space group  | Triclinic, $P\bar{1}$  |
| Temperature (K)  | 90   |
| $a, b, c$ (Å)  | 9.8543 (15), 11.1140 (17),<br>11.4288 (18)                             |
| $\alpha, \beta, \gamma$ (°)  | 86.563 (5), 74.417 (5), 82.951 (5)                                     |
| $V$ (Å <sup>3</sup> )  | 1196.1 (3)   |
| $Z$  | 2  |
| Radiation type   | Mo $K\alpha$   |
| $\mu$ (mm <sup>-1</sup> )  | 0.30   |
| Crystal size (mm)  | 0.80 × 0.30 × 0.30   |
| Data collection  |  |
| Diffractometer   | Bruker D8 QUEST  |
| Absorption correction  | Multi-scan ( <i>SADABS</i> ; Krause <i>et al.</i> , 2015)              |
| $T_{\text{min}}, T_{\text{max}}$   | 0.70, 0.92   |
| No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections | 7500, 2909, 2808   |
| $R_{\text{int}}$   | 0.057  |
| $\theta_{\text{max}}$ (°)  | 22.4   |
| $(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )                    | 0.536  |
| Refinement   |  |
| $R[F^2 > 2\sigma(F^2)], wR(F^2), S$  | 0.078, 0.178, 1.14   |
| No. of reflections   | 2909   |
| No. of parameters  | 309  |
| H-atom treatment   | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )    | 0.78, -0.56  |

Computer programs: *APEX4* and *SAINT* (Bruker, 2019), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL2019/1* (Sheldrick, 2015b) and *shelXle ShelXle* (Hübschle *et al.*, 2011).

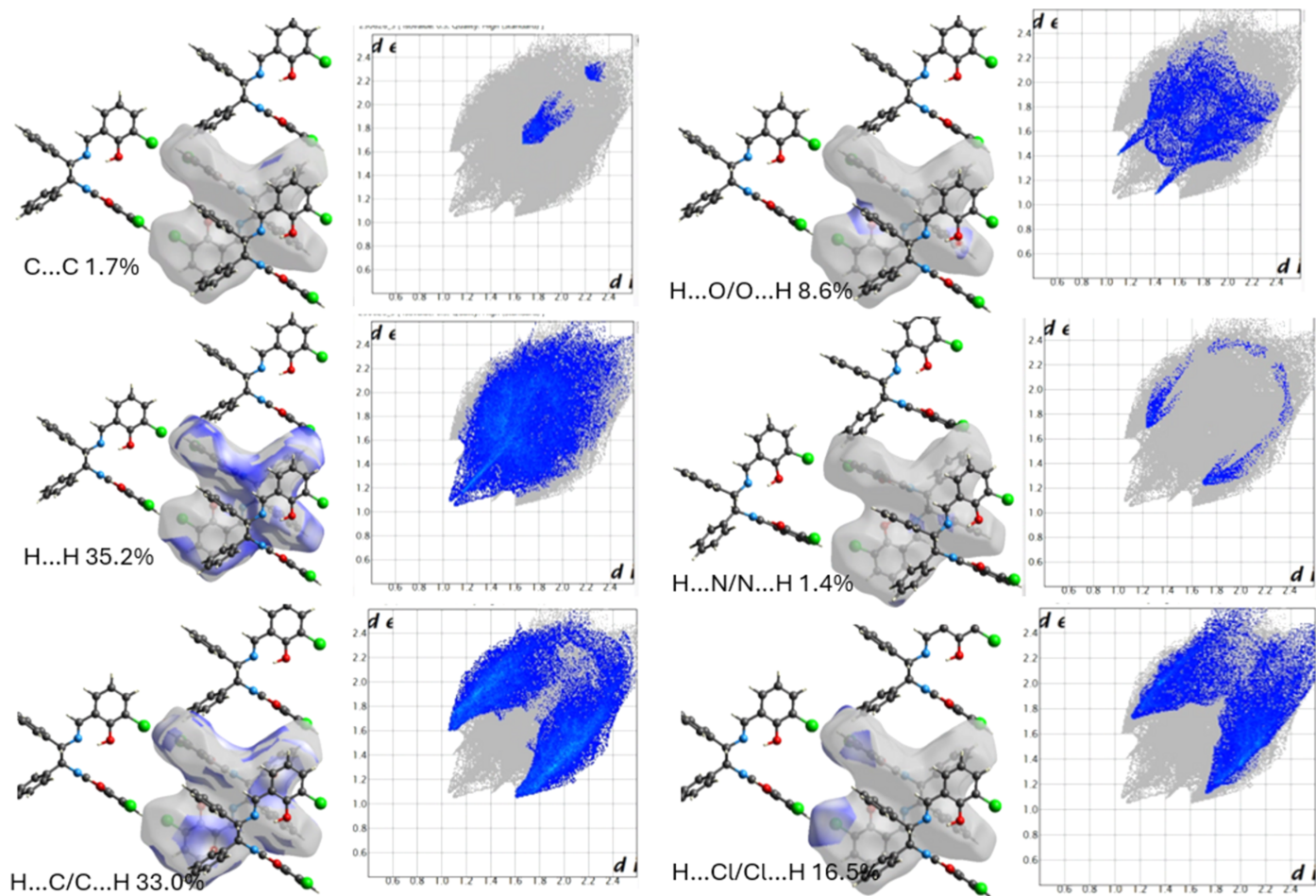
bis(azanelylidene)]bis(methaneylylidene)]bis(2-fluorophenol), which is the monoclinic polymorph (refcode LECYUQ; Shen *et al.*, 2017). Besides this, there are several similar compounds, such as 6,6'-((1*E*,1*E'*)-[(1*R*,2*R*)-1,2-diphenylethane-1,2-diyl]bis(azanelylidene)]bis(methaneylylidene)]bis(2-ethylphenol) (refcode OWIJAI; Xu *et al.*, 2021) and *N,N*-salsicylidene-(*R,S*)-1,2-ethanediamine (refcode DSPEDN01; Ramazani *et al.*, 2006).

#### 5. Synthesis and crystallization

3-Chlorosalicylaldehyde (0.039 g, 0.25 mmol) and *racemic*-(+/-)-1,2-diphenylethylenediamine (0.027 g, 0.125 mmol) were



**Figure 4**  
The packing of the title compound, showing the chain formed by Cl...Cl and O...C contacts.



**Figure 5**  
Hirshfeld surface mapped over  $d_{\text{norm}}$  and the two-dimensional fingerprint plots.

dissolved in 18 mL of methanol. The solution was stirred 50 s and irradiated with microwaves for 10 minutes at 353 K. The resulting clear yellow solution was evaporated. Recrystallized by slow evaporation of diethyl ether solution gave clear yellow rectangular-parallelepiped-shaped single crystals suitable for single-crystal X-ray diffraction analysis within a day. IR (ATR,  $\text{cm}^{-1}$ ): 522 (*s*), 776 (*m*), 832 (*w*), 896 (*s*), 1093 (*m*, C–OH), 1413 (*w*), 1447 (*w*), 1624 (*m*, C=N)

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All C-bound H atoms were placed in geometrically calculated positions (C–H = 0.95–0.10 Å) and were refined using a riding model with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for  $\text{R}_2\text{CH}$  and  $\text{R}_3\text{CH}$  H atoms and  $1.5U_{\text{eq}}(\text{C})$  for the methyl H atoms. The O-bound H atoms H29, H30 were located based on a difference-Fourier map and refined using constraints. In the process of removing the weak reciprocal lattice points due to twins, some reflections that should be present may have been inadvertently deleted.

## Funding information

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## supporting information

*Acta Cryst.* (2025). E81, 1023-1027 [https://doi.org/10.1107/S2056989025008850]

## A new triclinic polymorph of 6,6'-{(1*E*,1*E'*)-[(1,2-diphenylethane-1,2-diyl)bis-(azaneylylidene)]bis(methaneylylidene)}bis(2-chlorophenol)

Marika Iwatani, Daisuke Nakane and Takashi Akitsu

### Computing details

6,6'-{(1*E*,1*E'*)-[(1,2-Diphenylethane-1,2-diyl)bis(azaneylylidene)]bis(methaneylylidene)}bis(2-chlorophenol)

#### Crystal data

C<sub>28</sub>H<sub>22</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>2</sub>

*M<sub>r</sub>* = 489.37

Triclinic, *P* $\bar{1}$

*a* = 9.8543 (15) Å

*b* = 11.1140 (17) Å

*c* = 11.4288 (18) Å

$\alpha$  = 86.563 (5)°

$\beta$  = 74.417 (5)°

$\gamma$  = 82.951 (5)°

*V* = 1196.1 (3) Å<sup>3</sup>

*Z* = 2

*F*(000) = 508

*D<sub>x</sub>* = 1.359 Mg m<sup>-3</sup>

Mo *K*α radiation,  $\lambda$  = 0.71073 Å

Cell parameters from 7076 reflections

$\theta$  = 2.6–22.4°

$\mu$  = 0.30 mm<sup>-1</sup>

*T* = 90 K

Plate, yellow

0.80 × 0.30 × 0.30 mm

#### Data collection

Bruker D8 QUEST

diffractometer

Detector resolution: 7.3910 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Krause *et al.*, 2015)

*T<sub>min</sub>* = 0.70, *T<sub>max</sub>* = 0.92

7500 measured reflections

2909 independent reflections

2808 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.057

$\theta_{\max}$  = 22.4°,  $\theta_{\min}$  = 1.9°

*h* = -10→10

*k* = -11→11

*l* = -12→12

#### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.078

*wR*(*F*<sup>2</sup>) = 0.178

*S* = 1.14

2909 reflections

309 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.1181*P*)<sup>2</sup> + 0.4041*P*]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> < 0.001

Δρ<sub>max</sub> = 0.78 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.56 e Å<sup>-3</sup>

*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 ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| Cl33 | 0.69894 (7)  | 0.77330 (6)  | 0.10865 (5)  | 0.0321 (3)                       |
| Cl34 | 0.48781 (7)  | 0.21930 (6)  | 0.10402 (6)  | 0.0386 (3)                       |
| O29  | 0.70703 (17) | 0.59076 (14) | 0.30123 (15) | 0.0236 (5)                       |
| H29  | 0.707911     | 0.541261     | 0.359594     | 0.068 (12)*                      |
| O30  | 0.58282 (17) | 0.26535 (16) | 0.31484 (15) | 0.0267 (5)                       |
| H30  | 0.614604     | 0.279211     | 0.373340     | 0.066 (12)*                      |
| N31  | 0.72307 (19) | 0.51873 (17) | 0.51494 (17) | 0.0183 (5)                       |
| N32  | 0.77316 (19) | 0.28700 (16) | 0.42902 (17) | 0.0183 (5)                       |
| C1   | 0.9058 (3)   | 0.4613 (2)   | 0.7130 (2)   | 0.0220 (6)                       |
| H1   | 0.977177     | 0.452213     | 0.638356     | 0.026000*                        |
| C12  | 0.7867 (3)   | -0.0137 (2)  | 0.7700 (2)   | 0.0238 (6)                       |
| H12  | 0.779742     | -0.084463    | 0.821244     | 0.029000*                        |
| C13  | 0.6670 (3)   | 0.0463 (2)   | 0.7423 (2)   | 0.0234 (6)                       |
| H13  | 0.577598     | 0.016854     | 0.775207     | 0.028000*                        |
| C14  | 0.6770 (2)   | 0.1492 (2)   | 0.6668 (2)   | 0.0214 (6)                       |
| H14  | 0.594582     | 0.189105     | 0.647407     | 0.026000*                        |
| C15  | 0.8672 (2)   | 0.2710 (2)   | 0.3286 (2)   | 0.0179 (6)                       |
| H15  | 0.963560     | 0.274867     | 0.326773     | 0.021000*                        |
| C16  | 0.8330 (3)   | 0.24711 (19) | 0.2164 (2)   | 0.0195 (6)                       |
| C17  | 0.9410 (3)   | 0.2246 (2)   | 0.1100 (2)   | 0.0264 (6)                       |
| H17  | 1.036817     | 0.227321     | 0.110552     | 0.032000*                        |
| C18  | 0.9104 (3)   | 0.1985 (2)   | 0.0043 (2)   | 0.0325 (7)                       |
| H18  | 0.984757     | 0.182025     | -0.067299    | 0.039000*                        |
| C19  | 0.7710 (3)   | 0.1964 (2)   | 0.0027 (2)   | 0.0307 (7)                       |
| H19  | 0.749397     | 0.178877     | -0.070156    | 0.037000*                        |
| C20  | 0.6632 (3)   | 0.2197 (2)   | 0.1067 (2)   | 0.0253 (6)                       |
| C21  | 0.6908 (3)   | 0.24488 (19) | 0.2153 (2)   | 0.0207 (6)                       |
| C22  | 0.7105 (2)   | 0.6290 (2)   | 0.5464 (2)   | 0.0180 (6)                       |
| H22  | 0.705884     | 0.646557     | 0.627801     | 0.022000*                        |
| C23  | 0.7031 (2)   | 0.7283 (2)   | 0.4572 (2)   | 0.0174 (6)                       |
| C24  | 0.6948 (2)   | 0.8487 (2)   | 0.4918 (2)   | 0.0215 (6)                       |
| H24  | 0.691537     | 0.864924     | 0.573228     | 0.026000*                        |
| C25  | 0.6914 (2)   | 0.9436 (2)   | 0.4090 (2)   | 0.0236 (6)                       |
| H25  | 0.685739     | 1.024745     | 0.433281     | 0.028000*                        |
| C26  | 0.6962 (2)   | 0.9203 (2)   | 0.2899 (2)   | 0.0236 (6)                       |
| H26  | 0.695707     | 0.985296     | 0.232084     | 0.028000*                        |
| C27  | 0.7018 (2)   | 0.8019 (2)   | 0.2556 (2)   | 0.0216 (6)                       |
| C28  | 0.7046 (2)   | 0.7043 (2)   | 0.3380 (2)   | 0.0186 (6)                       |
| C2   | 0.9419 (3)   | 0.4896 (2)   | 0.8163 (2)   | 0.0248 (6)                       |

|     |            |            |            |            |
|-----|------------|------------|------------|------------|
| H2  | 1.037668   | 0.498182   | 0.812667   | 0.030000*  |
| C3  | 0.8383 (3) | 0.5054 (2) | 0.9247 (2) | 0.0274 (6) |
| H3  | 0.862207   | 0.526466   | 0.995457   | 0.033000*  |
| C4  | 0.6999 (3) | 0.4904 (2) | 0.9294 (2) | 0.0304 (7) |
| H4  | 0.628617   | 0.500581   | 1.003883   | 0.036000*  |
| C5  | 0.6643 (3) | 0.4606 (2) | 0.8265 (2) | 0.0261 (6) |
| H5  | 0.568705   | 0.450108   | 0.831112   | 0.031000*  |
| C6  | 0.7664 (2) | 0.4459 (2) | 0.7168 (2) | 0.0185 (6) |
| C7  | 0.7240 (2) | 0.4172 (2) | 0.6047 (2) | 0.0181 (6) |
| H7  | 0.625043   | 0.394426   | 0.632597   | 0.022000*  |
| C8  | 0.8179 (2) | 0.3074 (2) | 0.5376 (2) | 0.0167 (6) |
| H8  | 0.918827   | 0.325516   | 0.512654   | 0.020000*  |
| C9  | 0.8068 (2) | 0.1941 (2) | 0.6193 (2) | 0.0171 (6) |
| C10 | 0.9253 (3) | 0.1342 (2) | 0.6481 (2) | 0.0236 (6) |
| H10 | 1.014514   | 0.164461   | 0.616680   | 0.028000*  |
| C11 | 0.9160 (3) | 0.0300 (2) | 0.7228 (2) | 0.0270 (6) |
| H11 | 0.998589   | -0.010831  | 0.741072   | 0.032000*  |

*Atomic displacement parameters (Å<sup>2</sup>)*

|      | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|------|-------------|-------------|-------------|--------------|--------------|--------------|
| Cl33 | 0.0450 (5)  | 0.0330 (5)  | 0.0197 (5)  | -0.0019 (3)  | -0.0125 (3)  | 0.0016 (3)   |
| Cl34 | 0.0431 (5)  | 0.0379 (5)  | 0.0479 (6)  | -0.0175 (4)  | -0.0315 (4)  | 0.0108 (4)   |
| O29  | 0.0337 (10) | 0.0160 (9)  | 0.0221 (10) | -0.0004 (7)  | -0.0094 (8)  | -0.0029 (8)  |
| O30  | 0.0221 (10) | 0.0335 (11) | 0.0255 (11) | -0.0067 (8)  | -0.0072 (8)  | 0.0025 (8)   |
| N31  | 0.0189 (10) | 0.0160 (11) | 0.0199 (11) | -0.0015 (8)  | -0.0048 (9)  | -0.0009 (8)  |
| N32  | 0.0209 (11) | 0.0132 (10) | 0.0207 (11) | -0.0014 (8)  | -0.0053 (9)  | 0.0002 (8)   |
| C1   | 0.0250 (13) | 0.0214 (13) | 0.0189 (13) | -0.0035 (10) | -0.0039 (11) | -0.0020 (10) |
| C12  | 0.0317 (14) | 0.0166 (13) | 0.0244 (14) | -0.0037 (11) | -0.0098 (11) | 0.0019 (10)  |
| C13  | 0.0229 (13) | 0.0196 (13) | 0.0288 (14) | -0.0063 (10) | -0.0076 (11) | 0.0008 (10)  |
| C14  | 0.0198 (13) | 0.0190 (13) | 0.0270 (14) | -0.0017 (10) | -0.0090 (11) | -0.0009 (10) |
| C15  | 0.0178 (12) | 0.0128 (12) | 0.0218 (14) | -0.0009 (9)  | -0.0035 (11) | -0.0004 (9)  |
| C16  | 0.0263 (13) | 0.0103 (12) | 0.0221 (14) | -0.0002 (10) | -0.0075 (11) | 0.0006 (9)   |
| C17  | 0.0291 (14) | 0.0243 (14) | 0.0241 (15) | 0.0036 (11)  | -0.0067 (12) | -0.0030 (11) |
| C18  | 0.0443 (17) | 0.0313 (15) | 0.0205 (14) | 0.0075 (12)  | -0.0101 (12) | -0.0057 (11) |
| C19  | 0.0528 (18) | 0.0196 (14) | 0.0248 (15) | 0.0010 (12)  | -0.0204 (13) | -0.0040 (11) |
| C20  | 0.0388 (16) | 0.0120 (12) | 0.0311 (15) | -0.0058 (11) | -0.0191 (13) | 0.0036 (10)  |
| C21  | 0.0275 (14) | 0.0094 (12) | 0.0255 (14) | -0.0021 (10) | -0.0084 (11) | 0.0029 (10)  |
| C22  | 0.0150 (12) | 0.0199 (13) | 0.0184 (13) | -0.0014 (9)  | -0.0031 (10) | -0.0027 (10) |
| C23  | 0.0126 (11) | 0.0177 (12) | 0.0205 (13) | 0.0006 (9)   | -0.0026 (10) | -0.0020 (10) |
| C24  | 0.0191 (13) | 0.0194 (13) | 0.0260 (14) | -0.0001 (10) | -0.0061 (10) | -0.0034 (10) |
| C25  | 0.0231 (13) | 0.0164 (13) | 0.0317 (15) | -0.0025 (10) | -0.0076 (11) | -0.0015 (10) |
| C26  | 0.0201 (12) | 0.0187 (13) | 0.0311 (15) | -0.0008 (10) | -0.0073 (11) | 0.0058 (10)  |
| C27  | 0.0183 (12) | 0.0242 (14) | 0.0220 (14) | -0.0007 (10) | -0.0059 (10) | 0.0012 (10)  |
| C28  | 0.0117 (12) | 0.0190 (13) | 0.0244 (14) | 0.0003 (9)   | -0.0038 (10) | -0.0044 (10) |
| C2   | 0.0293 (14) | 0.0189 (13) | 0.0280 (15) | -0.0028 (11) | -0.0106 (12) | -0.0011 (10) |
| C3   | 0.0413 (17) | 0.0211 (13) | 0.0215 (14) | 0.0039 (11)  | -0.0141 (12) | -0.0028 (10) |
| C4   | 0.0343 (16) | 0.0372 (15) | 0.0156 (13) | 0.0053 (12)  | -0.0030 (11) | -0.0049 (11) |

|     |             |             |             |              |              |              |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C5  | 0.0229 (13) | 0.0287 (14) | 0.0232 (14) | 0.0006 (11)  | -0.0019 (11) | -0.0006 (11) |
| C6  | 0.0239 (13) | 0.0108 (12) | 0.0191 (13) | 0.0007 (9)   | -0.0040 (10) | 0.0009 (9)   |
| C7  | 0.0176 (12) | 0.0160 (12) | 0.0203 (13) | -0.0027 (9)  | -0.0045 (10) | 0.0004 (10)  |
| C8  | 0.0161 (12) | 0.0172 (12) | 0.0171 (13) | -0.0021 (9)  | -0.0046 (10) | -0.0015 (9)  |
| C9  | 0.0203 (12) | 0.0144 (12) | 0.0165 (12) | -0.0005 (9)  | -0.0043 (10) | -0.0063 (9)  |
| C10 | 0.0193 (13) | 0.0210 (14) | 0.0302 (14) | -0.0032 (10) | -0.0050 (11) | -0.0019 (11) |
| C11 | 0.0248 (14) | 0.0226 (14) | 0.0351 (16) | 0.0024 (11)  | -0.0132 (12) | 0.0013 (11)  |

*Geometric parameters (Å, °)*

|              |             |              |             |
|--------------|-------------|--------------|-------------|
| Cl33—C27     | 1.736 (2)   | C19—C20      | 1.378 (4)   |
| Cl34—C20     | 1.738 (3)   | C20—C21      | 1.390 (4)   |
| O29—C28      | 1.349 (3)   | C22—C23      | 1.465 (3)   |
| O30—C21      | 1.341 (3)   | C23—C28      | 1.399 (3)   |
| N31—C22      | 1.280 (3)   | C23—C24      | 1.405 (3)   |
| N31—C7       | 1.479 (3)   | C24—C25      | 1.378 (4)   |
| N32—C15      | 1.273 (3)   | C25—C26      | 1.388 (4)   |
| N32—C8       | 1.463 (3)   | C26—C27      | 1.386 (4)   |
| C1—C2        | 1.385 (4)   | C27—C28      | 1.396 (4)   |
| C1—C6        | 1.394 (4)   | C2—C3        | 1.382 (4)   |
| C12—C11      | 1.379 (4)   | C3—C4        | 1.381 (4)   |
| C12—C13      | 1.385 (4)   | C4—C5        | 1.383 (4)   |
| C13—C14      | 1.388 (4)   | C5—C6        | 1.385 (4)   |
| C14—C9       | 1.390 (3)   | C6—C7        | 1.513 (3)   |
| C15—C16      | 1.457 (3)   | C7—C8        | 1.545 (3)   |
| C16—C17      | 1.397 (4)   | C8—C9        | 1.519 (3)   |
| C16—C21      | 1.409 (4)   | C9—C10       | 1.381 (3)   |
| C17—C18      | 1.376 (4)   | C10—C11      | 1.394 (4)   |
| C18—C19      | 1.382 (4)   |              |             |
| C22—N31—C7   | 121.14 (19) | C27—C26—C25  | 119.8 (2)   |
| C15—N32—C8   | 118.73 (19) | C26—C27—C28  | 121.4 (2)   |
| C2—C1—C6     | 121.0 (2)   | C26—C27—Cl33 | 119.82 (19) |
| C11—C12—C13  | 119.5 (2)   | C28—C27—Cl33 | 118.69 (18) |
| C12—C13—C14  | 120.3 (2)   | O29—C28—C27  | 119.4 (2)   |
| C13—C14—C9   | 120.5 (2)   | O29—C28—C23  | 122.2 (2)   |
| N32—C15—C16  | 122.6 (2)   | C27—C28—C23  | 118.4 (2)   |
| C17—C16—C21  | 119.7 (2)   | C3—C2—C1     | 119.9 (2)   |
| C17—C16—C15  | 120.3 (2)   | C4—C3—C2     | 119.5 (2)   |
| C21—C16—C15  | 120.1 (2)   | C3—C4—C5     | 120.6 (2)   |
| C18—C17—C16  | 120.8 (2)   | C4—C5—C6     | 120.7 (2)   |
| C17—C18—C19  | 119.7 (2)   | C5—C6—C1     | 118.3 (2)   |
| C20—C19—C18  | 120.1 (2)   | C5—C6—C7     | 119.7 (2)   |
| C19—C20—C21  | 121.6 (2)   | C1—C6—C7     | 122.0 (2)   |
| C19—C20—Cl34 | 120.1 (2)   | N31—C7—C6    | 115.14 (18) |
| C21—C20—Cl34 | 118.4 (2)   | N31—C7—C8    | 107.84 (18) |
| O30—C21—C20  | 119.6 (2)   | C6—C7—C8     | 112.28 (19) |
| O30—C21—C16  | 122.2 (2)   | N32—C8—C9    | 109.64 (17) |

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|-------------|-----------|-------------|-------------|
| C20—C21—C16 | 118.1 (2) | N32—C8—C7   | 109.18 (18) |
| N31—C22—C23 | 120.2 (2) | C9—C8—C7    | 110.89 (18) |
| C28—C23—C24 | 119.8 (2) | C10—C9—C14  | 118.8 (2)   |
| C28—C23—C22 | 120.7 (2) | C10—C9—C8   | 120.6 (2)   |
| C24—C23—C22 | 119.6 (2) | C14—C9—C8   | 120.6 (2)   |
| C25—C24—C23 | 120.8 (2) | C9—C10—C11  | 120.9 (2)   |
| C24—C25—C26 | 119.8 (2) | C12—C11—C10 | 120.0 (2)   |

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*Hydrogen-bond geometry (Å, °)*

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| <i>D—H...A</i> | <i>D—H</i> | <i>H...A</i> | <i>D...A</i> | <i>D—H...A</i> |
|----------------|------------|--------------|--------------|----------------|
| O29—H29...N31  | 0.84       | 1.82         | 2.564 (3)    | 147            |
| O30—H30...N32  | 0.84       | 1.85         | 2.597 (3)    | 147            |

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