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# 10,16-Dichloro-6,20-dioxa-3,23-diazatetracyclo[23.3.1.0$^{7,12}$.0$^{14,19}$]nonacosa-1(29),7,9,11,14(19),15,17,25,27-nonaene-4,22-dione methanol monosolvate

**Michaela Pojarová,[a]* Michal Dušek,[a] Zdeňka Sedláková[b] and Emanuel Makrlík[c]**

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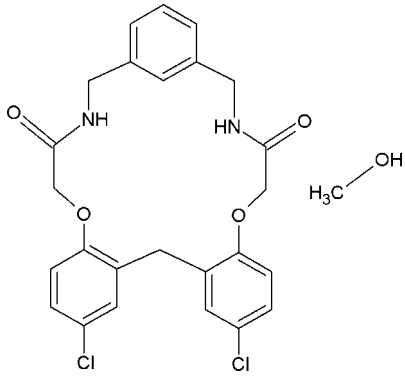
Key indicators: single-crystal X-ray study; $T$ = 120 K; mean $\sigma$(C–C) = 0.003 Å; disorder in solvent or counterion; $R$ factor = 0.033; $wR$ factor = 0.082; data-to-parameter ratio = 12.6.

In the title compound, $C_{25}H_{22}Cl_2N_2O_4\cdot CH_3OH$, the macrocyclic molecule adopts a slightly distorted $C_2$-symmetric conformation. The macrocyclic molecules are linked *via* N—H···O hydrogen bonds between the amide groups into chains extending along the [010] direction. The methanol molecules bridge these chains *via* N—H···O and O—H···O hydrogen bonds with the formation of a two-dimensional polymeric structure parallel to (001). The methanol molecule is disordered over two positions with the occupancy ratio of 9:1. The disorder of the solvent molecule is caused by weak intermolecular C—H···Cl hydrogen bonding.

## Related literature

For application of macrocycles, see: Hayvali & Hayvali (2005); Kleinpeter *et al.* (1997); Jaiyu *et al.* (2007); Christensen *et al.* (1997); Alexander (1995). For the synthetic procedure, see: Ertul *et al.* (2009).




## Experimental

### Crystal data

$C_{25}H_{22}Cl_2N_2O_4\cdot CH_4O$
$M_r$ = 517.39
Orthorhombic, *Pbca*
$a$ = 21.9905 (3) Å
$b$ = 8.1864 (1) Å
$c$ = 26.6760 (3) Å
$V$ = 4802.29 (10) Å$^3$
$Z$ = 8
Cu $K\alpha$ radiation
$\mu$ = 2.78 mm$^{-1}$
$T$ = 120 K
0.30 × 0.11 × 0.08 mm

### Data collection

Oxford Diffraction Xcalibur A Gemini Ultra diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
$T_{min}$ = 0.826, $T_{max}$ = 1.000
42959 measured reflections
4099 independent reflections
3364 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.070

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.082
$S$ = 1.03
4099 reflections
326 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1N1···O5 | 0.98 | 2.31 | 3.039 (3) | 130 |
| N2—H1N2···O1$^{i}$ | 0.93 | 2.20 | 2.860 (2) | 128 |
| O5—H1O5···O4$^{ii}$ | 0.82 | 2.05 | 2.789 (3) | 150 |
| C26*A*—H26*F*···Cl2$^{iii}$ | 0.96 | 2.74 | 3.616 (3) | 149 |

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *publCIF* (Westrip, 2010).

This study was financially supported by the Institutional Research Plan No. AVOZ10100521 of the Institute of Physics, the project Praemium Academiae of the Academy of Science of the Czech Republic, the Grant Agency of the Faculty of Environmental Sciences, Czech University of Life Sciences, Prague (project No. 42900/1312/3114 'Environmental Aspects of Sustainable Development of Society'), and the Czech Ministry of Education, Youth and Sports (Project MSM 6046137307).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2457).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o805–o806 [doi:10.1107/S1600536812007052]

## 10,16-Dichloro-6,20-dioxa-3,23-diazatetracyclo-[23.3.1.$0^{7,12}$.$0^{14,19}$]nonacosa-1(29),7,9,11,14(19),15,17,25,27-nonaene-4,22-dione methanol monosolvate

**Michaela Pojarová, Michal Dušek, Zdeňka Sedláková and Emanuel Makrlík**

### Comment

Polyazalactones together with polyoxalactones and polyethers are studied for their ability to act as multidentate ligands to complex various cations. Polyazalactones can incorporate transition metals into their cavities *via* an ion-dipole interaction (Hayvali & Hayvali, 2005; Kleinpeter *et al.*, 1997). They are studied for their role in bioprocesses, catalysis, material science, transport and separation (Jaiyu *et al.*, 2007; Christensen *et al.*, 1997; Alexander, 1995). In this paper, we report the crystal structure of a lactam ionophore (Fig.1). The macrocycle consists of three benzene rings, two of them substituted with chlorine atom in *para* position to O atom. The neighboring molecules are connected *via* hydrogen bonds between amide groups (Table 1). The crystal contains methanol molecule disordered over two positions with partial occupancies of 0.90 and 0.10. The hydroxyl group of the solvent forms hydrogen bond to the oxygen atom of the amide group (Table 1). The methyl group of the methanol in second position is also weakly bound to the chlorine atoms of neighboring molecule. This weak interaction competes with the stronger hydrogen bond to amide group and causes the solvent disorder.

### Experimental

All chemicals used were purchased from Fluka and used without further purification. The title compound was synthesized according to the method reported by Ertul *et al.* (2009). Single crystals were prepared by slow evaporation of methanol solution.

### Refinement

Positions of disordered groups were found from electron density maps. The disordered fragments were then placed in appropriate positions, and all distances between neighbouring atoms were restrained to 1.406 (20) Å. Site occupancies were refined for the different parts with the common displacement parameters for corresponding atoms in various fragments. At the end of the refinement, site occupancies were fixed at the values 0.9 and 0.1 and hydrogen atoms were placed in calculated positions. All hydrogen atoms of the macrocyclic molecule were found from electron density difference maps. H atoms attached to C atoms were placed in calculated positions. N—H distances were initially restrained to 1.00 Å with $\sigma$=0.02 and then fixed. The isotropic displacement parameters of H atoms were calculated as 1.2$U_{eq}$ of the parent atom.

### Computing details

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO* (Agilent, 2010); data reduction: *CrysAlis PRO* (Agilent, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine

structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2006) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *publCIF* (Westrip, 2010).

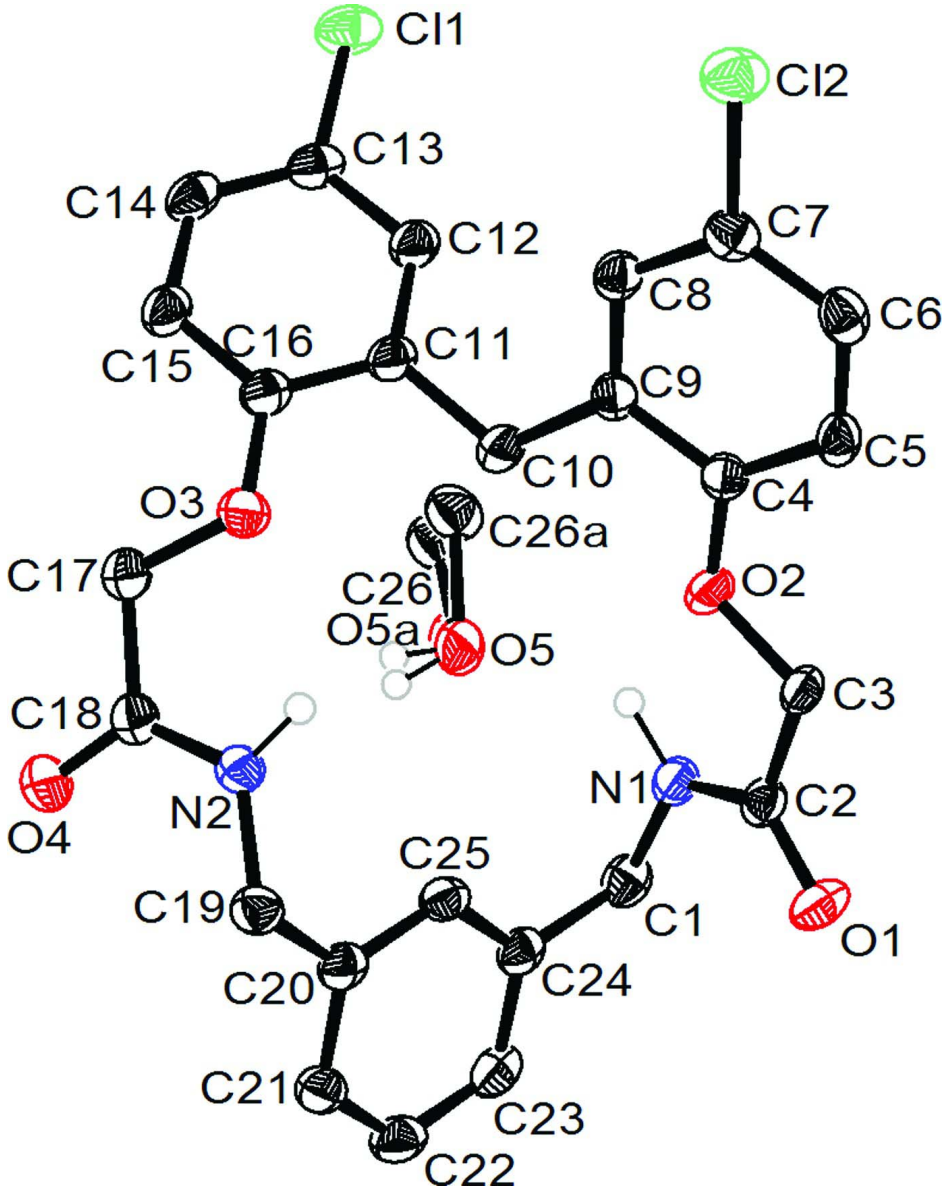


**Figure 1**

View of the asymmetric unit of the title compound with displacement ellipsoids shown at the 50% probability level. C-bound H atoms have been omitted for clarity.

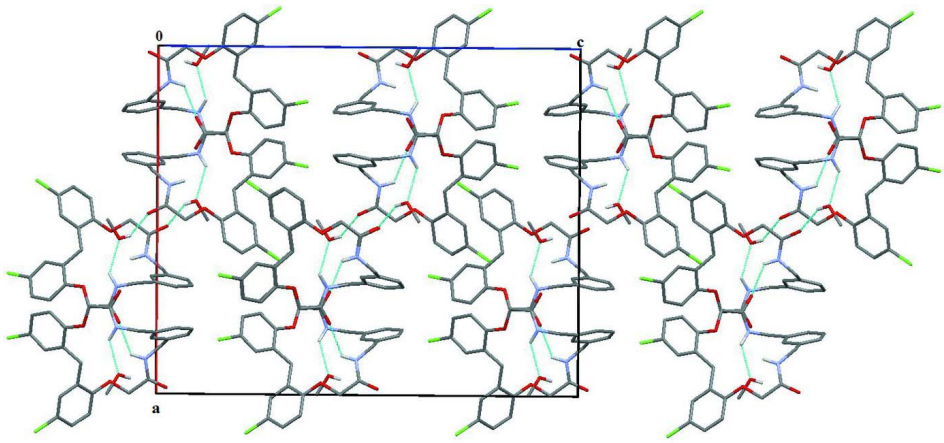


**Figure 2**

Projection along the *b* axis with highlighted hydrogen bonds between the molecules. Hydrogen atoms not involved in hydrgen bonding have been omitted.

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*Crystal data*

$C_{25}H_{22}Cl_2N_2O_4{\cdot}CH_4O$
$M_r$ = 517.39
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
*a* = 21.9905 (3) Å
*b* = 8.1864 (1) Å
*c* = 26.6760 (3) Å
*V* = 4802.29 (10) Å$^3$
*Z* = 8
*F*(000) = 2160
$D_x$ = 1.431 Mg m$^{-3}$
Melting point = 316–318 K
Cu *Kα* radiation, *λ* = 1.5418 Å
Cell parameters from 6415 reflections
*θ* = 3.3–67.1°
*μ* = 2.78 mm$^{-1}$
*T* = 120 K
Prism, colourless
0.30 × 0.11 × 0.08 mm

*Data collection*

Oxford Diffraction Xcalibur A Gemini Ultra diffractometer
Radiation source: Enhance Ultra (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3784 pixels mm$^{-1}$
*ω* scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
$T_{min}$ = 0.826, $T_{max}$ = 1.000
42959 measured reflections
4099 independent reflections
3364 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.070
$\theta_{max}$ = 65.1°, $\theta_{min}$ = 3.3°
*h* = −22→25
*k* = −9→9
*l* = −29→31

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.082
*S* = 1.03
4099 reflections
326 parameters
4 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

$w = 1/[\sigma^2(F_o^2) + (0.0358P)^2 + 2.1383P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.20$ e Å$^{-3}$
$\Delta\rho_{min} = -0.21$ e Å$^{-3}$

*Special details*

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger. Positions of disordered groups were found from electron density maps. The disordered fragments were then placed in appropriate positions, and all distances between neighbouring atoms were fixed. Site occupancies were refined for the different parts with the same thermal parameters for same atoms in various fragments. At the end of the refinement, site occupancies were fixed at values 0.90 and 0.10 and hydrogen atoms were placed into calculated positions. All hydrogen atoms could be found from maps of difference electron density, but those, attached to carbon atoms, were placed into calculated positions. The distance between N and H atoms were restrained to 1.00 Å with $\sigma$=0.02. The isotropic temperature parameters of hydrogen atoms were calculated as 1.2*$U_{eq}$ of the parent atom.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cl1 | 1.11069 (2) | 0.02416 (7) | 0.213524 (18) | 0.03911 (14) | |
| Cl2 | 0.84287 (2) | −0.21013 (7) | 0.147714 (18) | 0.03856 (14) | |
| O1 | 0.70542 (6) | 0.38705 (18) | 0.40933 (5) | 0.0367 (3) | |
| O2 | 0.80594 (5) | 0.19883 (17) | 0.32491 (5) | 0.0305 (3) | |
| O3 | 0.96571 (6) | −0.16498 (18) | 0.38954 (5) | 0.0322 (3) | |
| O4 | 0.98144 (6) | −0.1787 (2) | 0.52219 (5) | 0.0406 (4) | |
| N1 | 0.80833 (7) | 0.38887 (19) | 0.40351 (6) | 0.0277 (3) | |
| H1N2 | 0.8870 | −0.1436 | 0.4391 | 0.033* | |
| N2 | 0.89913 (7) | −0.14110 (19) | 0.47251 (5) | 0.0262 (3) | |
| H1N1 | 0.8439 | 0.3425 | 0.3862 | 0.031* | |
| C1 | 0.81828 (9) | 0.4614 (2) | 0.45255 (7) | 0.0315 (4) | |
| H1A | 0.7853 | 0.5366 | 0.4597 | 0.038* | |
| H1B | 0.8557 | 0.5239 | 0.4517 | 0.038* | |
| C2 | 0.75257 (8) | 0.3497 (2) | 0.38745 (7) | 0.0266 (4) | |
| C3 | 0.74788 (8) | 0.2590 (2) | 0.33841 (7) | 0.0265 (4) | |
| H3A | 0.7195 | 0.1690 | 0.3417 | 0.032* | |
| H3B | 0.7329 | 0.3318 | 0.3125 | 0.032* | |
| C4 | 0.81162 (8) | 0.1097 (2) | 0.28177 (6) | 0.0240 (4) | |
| C5 | 0.76696 (8) | 0.0977 (2) | 0.24538 (7) | 0.0267 (4) | |
| H5 | 0.7306 | 0.1547 | 0.2489 | 0.032* | |
| C6 | 0.77682 (8) | 0.0000 (2) | 0.20357 (7) | 0.0289 (4) | |
| H6 | 0.7472 | −0.0093 | 0.1788 | 0.035* | |
| C7 | 0.83113 (8) | −0.0828 (2) | 0.19935 (7) | 0.0276 (4) | |
| C8 | 0.87611 (8) | −0.0703 (2) | 0.23554 (7) | 0.0255 (4) | |
| H8 | 0.9124 | −0.1273 | 0.2317 | 0.031* | |
| C9 | 0.86718 (8) | 0.0266 (2) | 0.27735 (7) | 0.0242 (4) | |
| C10 | 0.91217 (8) | 0.0476 (3) | 0.31990 (7) | 0.0338 (5) | |

| | | | | | |
|---|---|---|---|---|---|
| H10A | 0.9167 | 0.1636 | 0.3263 | 0.041* | |
| H10B | 0.8947 | −0.0011 | 0.3498 | 0.041* | |
| C11 | 0.97467 (8) | −0.0239 (2) | 0.31231 (7) | 0.0283 (4) | |
| C12 | 1.00962 (8) | 0.0216 (2) | 0.27119 (7) | 0.0296 (4) | |
| H12 | 0.9936 | 0.0927 | 0.2474 | 0.035* | |
| C13 | 1.06813 (8) | −0.0383 (2) | 0.26540 (7) | 0.0298 (4) | |
| C14 | 1.09288 (8) | −0.1440 (3) | 0.29985 (7) | 0.0317 (4) | |
| H14 | 1.1320 | −0.1849 | 0.2953 | 0.038* | |
| C15 | 1.05893 (9) | −0.1890 (3) | 0.34134 (7) | 0.0313 (4) | |
| H15 | 1.0753 | −0.2604 | 0.3649 | 0.038* | |
| C16 | 1.00041 (8) | −0.1277 (2) | 0.34793 (7) | 0.0275 (4) | |
| C17 | 0.99810 (9) | −0.1964 (3) | 0.43499 (7) | 0.0345 (5) | |
| H17A | 1.0330 | −0.1243 | 0.4370 | 0.041* | |
| H17B | 1.0128 | −0.3081 | 0.4348 | 0.041* | |
| C18 | 0.95804 (9) | −0.1705 (2) | 0.48030 (7) | 0.0291 (4) | |
| C19 | 0.85686 (9) | −0.1157 (2) | 0.51386 (7) | 0.0287 (4) | |
| H19A | 0.8741 | −0.1632 | 0.5440 | 0.034* | |
| H19B | 0.8194 | −0.1736 | 0.5066 | 0.034* | |
| C20 | 0.84194 (8) | 0.0621 (2) | 0.52404 (7) | 0.0261 (4) | |
| C21 | 0.82821 (8) | 0.1118 (3) | 0.57256 (7) | 0.0307 (4) | |
| H21 | 0.8297 | 0.0368 | 0.5987 | 0.037* | |
| C22 | 0.81235 (9) | 0.2726 (3) | 0.58206 (7) | 0.0350 (5) | |
| H22 | 0.8037 | 0.3052 | 0.6147 | 0.042* | |
| C23 | 0.80922 (8) | 0.3850 (3) | 0.54350 (8) | 0.0321 (4) | |
| H23 | 0.7985 | 0.4927 | 0.5503 | 0.039* | |
| C24 | 0.82213 (8) | 0.3373 (2) | 0.49450 (7) | 0.0271 (4) | |
| C25 | 0.83856 (8) | 0.1760 (2) | 0.48543 (7) | 0.0264 (4) | |
| H25 | 0.8475 | 0.1435 | 0.4528 | 0.032* | |
| O5 | 0.94522 (11) | 0.3414 (4) | 0.40875 (11) | 0.0418 (7) | 0.90 |
| H1O5 | 0.9545 | 0.2931 | 0.4346 | 0.050* | 0.90 |
| C26 | 0.99892 (15) | 0.3791 (5) | 0.38117 (9) | 0.0415 (7) | 0.90 |
| H26A | 0.9904 | 0.4652 | 0.3578 | 0.050* | 0.90 |
| H26B | 1.0123 | 0.2838 | 0.3633 | 0.050* | 0.90 |
| H26C | 1.0303 | 0.4138 | 0.4039 | 0.050* | 0.90 |
| O5A | 0.9587 (14) | 0.374 (5) | 0.4090 (12) | 0.0418 (7) | 0.10 |
| H2O5 | 0.9634 | 0.2905 | 0.4254 | 0.050* | 0.10 |
| C26A | 0.9961 (18) | 0.367 (6) | 0.3651 (11) | 0.0415 (7) | 0.10 |
| H26D | 0.9888 | 0.4625 | 0.3449 | 0.050* | 0.10 |
| H26E | 0.9863 | 0.2711 | 0.3462 | 0.050* | 0.10 |
| H26F | 1.0381 | 0.3642 | 0.3748 | 0.050* | 0.10 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0270 (2) | 0.0524 (3) | 0.0380 (3) | −0.0081 (2) | 0.00832 (19) | −0.0091 (2) |
| Cl2 | 0.0337 (3) | 0.0480 (3) | 0.0339 (3) | −0.0041 (2) | 0.00026 (19) | −0.0116 (2) |
| O1 | 0.0250 (7) | 0.0434 (9) | 0.0417 (8) | 0.0047 (6) | 0.0079 (6) | −0.0067 (7) |
| O2 | 0.0183 (6) | 0.0437 (8) | 0.0294 (7) | 0.0064 (6) | 0.0010 (5) | −0.0059 (6) |
| O3 | 0.0221 (6) | 0.0499 (9) | 0.0245 (6) | 0.0021 (6) | −0.0020 (5) | −0.0013 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O4 | 0.0343 (8) | 0.0595 (10) | 0.0279 (7) | 0.0096 (7) | −0.0066 (6) | 0.0019 (7) |
| N1 | 0.0236 (8) | 0.0284 (8) | 0.0311 (8) | 0.0029 (7) | 0.0014 (6) | 0.0015 (7) |
| N2 | 0.0254 (8) | 0.0302 (9) | 0.0230 (7) | 0.0009 (7) | −0.0009 (6) | −0.0013 (6) |
| C1 | 0.0290 (10) | 0.0283 (10) | 0.0372 (11) | 0.0017 (8) | −0.0014 (8) | −0.0044 (8) |
| C2 | 0.0242 (9) | 0.0240 (10) | 0.0315 (9) | 0.0042 (8) | 0.0035 (8) | 0.0059 (8) |
| C3 | 0.0185 (8) | 0.0298 (10) | 0.0313 (9) | 0.0057 (8) | 0.0024 (7) | 0.0050 (8) |
| C4 | 0.0216 (9) | 0.0264 (9) | 0.0241 (9) | −0.0010 (8) | 0.0028 (7) | 0.0051 (7) |
| C5 | 0.0184 (9) | 0.0314 (10) | 0.0302 (10) | 0.0002 (8) | 0.0002 (7) | 0.0092 (8) |
| C6 | 0.0232 (9) | 0.0359 (11) | 0.0276 (10) | −0.0049 (8) | −0.0033 (7) | 0.0064 (8) |
| C7 | 0.0261 (9) | 0.0306 (10) | 0.0260 (9) | −0.0053 (8) | 0.0020 (7) | 0.0013 (8) |
| C8 | 0.0208 (9) | 0.0279 (10) | 0.0277 (9) | −0.0002 (8) | 0.0029 (7) | 0.0043 (8) |
| C9 | 0.0194 (8) | 0.0284 (10) | 0.0247 (9) | −0.0012 (8) | 0.0021 (7) | 0.0058 (7) |
| C10 | 0.0218 (9) | 0.0515 (13) | 0.0280 (10) | 0.0097 (9) | −0.0010 (8) | −0.0040 (9) |
| C11 | 0.0196 (9) | 0.0398 (11) | 0.0256 (9) | 0.0026 (8) | −0.0024 (7) | −0.0082 (8) |
| C12 | 0.0233 (9) | 0.0364 (11) | 0.0290 (9) | 0.0024 (8) | −0.0018 (8) | −0.0056 (8) |
| C13 | 0.0212 (9) | 0.0383 (11) | 0.0300 (10) | −0.0042 (8) | 0.0019 (7) | −0.0109 (8) |
| C14 | 0.0186 (9) | 0.0406 (12) | 0.0359 (10) | 0.0022 (8) | −0.0019 (8) | −0.0131 (9) |
| C15 | 0.0248 (10) | 0.0374 (11) | 0.0317 (10) | 0.0055 (8) | −0.0048 (8) | −0.0076 (8) |
| C16 | 0.0207 (9) | 0.0356 (11) | 0.0262 (9) | −0.0002 (8) | −0.0017 (7) | −0.0074 (8) |
| C17 | 0.0294 (10) | 0.0463 (13) | 0.0278 (10) | 0.0091 (9) | −0.0037 (8) | −0.0014 (9) |
| C18 | 0.0291 (10) | 0.0313 (11) | 0.0271 (10) | 0.0028 (8) | −0.0027 (8) | 0.0004 (8) |
| C19 | 0.0264 (9) | 0.0326 (11) | 0.0270 (9) | −0.0017 (8) | 0.0027 (7) | 0.0020 (8) |
| C20 | 0.0177 (8) | 0.0335 (10) | 0.0271 (9) | −0.0020 (8) | −0.0015 (7) | −0.0035 (8) |
| C21 | 0.0236 (9) | 0.0434 (12) | 0.0251 (9) | −0.0008 (9) | −0.0002 (7) | 0.0004 (8) |
| C22 | 0.0286 (10) | 0.0494 (13) | 0.0271 (10) | 0.0025 (9) | 0.0009 (8) | −0.0109 (9) |
| C23 | 0.0235 (9) | 0.0346 (11) | 0.0383 (11) | 0.0034 (9) | −0.0017 (8) | −0.0098 (9) |
| C24 | 0.0176 (9) | 0.0334 (11) | 0.0301 (10) | −0.0008 (8) | −0.0022 (7) | −0.0048 (8) |
| C25 | 0.0215 (9) | 0.0334 (11) | 0.0245 (9) | −0.0007 (8) | 0.0011 (7) | −0.0047 (8) |
| O5 | 0.0243 (15) | 0.0620 (19) | 0.0390 (8) | 0.0036 (10) | −0.0010 (10) | 0.0116 (10) |
| C26 | 0.0367 (13) | 0.0549 (17) | 0.0330 (17) | −0.0057 (12) | −0.0024 (16) | 0.0023 (19) |
| O5A | 0.0243 (15) | 0.0620 (19) | 0.0390 (8) | 0.0036 (10) | −0.0010 (10) | 0.0116 (10) |
| C26A | 0.0367 (13) | 0.0549 (17) | 0.0330 (17) | −0.0057 (12) | −0.0024 (16) | 0.0023 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C13 | 1.7470 (19) | C11—C16 | 1.395 (3) |
| Cl2—C7 | 1.7466 (19) | C12—C13 | 1.386 (3) |
| O1—C2 | 1.229 (2) | C12—H12 | 0.9300 |
| O2—C4 | 1.368 (2) | C13—C14 | 1.375 (3) |
| O2—C3 | 1.415 (2) | C14—C15 | 1.385 (3) |
| O3—C16 | 1.381 (2) | C14—H14 | 0.9300 |
| O3—C17 | 1.430 (2) | C15—C16 | 1.392 (3) |
| O4—C18 | 1.232 (2) | C15—H15 | 0.9300 |
| N1—C2 | 1.338 (2) | C17—C18 | 1.511 (3) |
| N1—C1 | 1.453 (2) | C17—H17A | 0.9700 |
| N1—H1N1 | 0.9840 | C17—H17B | 0.9700 |
| N2—C18 | 1.334 (2) | C19—C20 | 1.517 (3) |
| N2—C19 | 1.458 (2) | C19—H19A | 0.9700 |
| N2—H1N2 | 0.9295 | C19—H19B | 0.9700 |
| C1—C24 | 1.514 (3) | C20—C21 | 1.390 (3) |

| | | | |
|---|---|---|---|
| C1—H1A | 0.9700 | C20—C25 | 1.391 (3) |
| C1—H1B | 0.9700 | C21—C22 | 1.385 (3) |
| C2—C3 | 1.507 (3) | C21—H21 | 0.9300 |
| C3—H3A | 0.9700 | C22—C23 | 1.382 (3) |
| C3—H3B | 0.9700 | C22—H22 | 0.9300 |
| C4—C5 | 1.384 (3) | C23—C24 | 1.394 (3) |
| C4—C9 | 1.404 (3) | C23—H23 | 0.9300 |
| C5—C6 | 1.389 (3) | C24—C25 | 1.391 (3) |
| C5—H5 | 0.9300 | C25—H25 | 0.9300 |
| C6—C7 | 1.378 (3) | O5—C26 | 1.425 (3) |
| C6—H6 | 0.9300 | O5—H1O5 | 0.8200 |
| C7—C8 | 1.386 (3) | C26—H26A | 0.9600 |
| C8—C9 | 1.383 (3) | C26—H26B | 0.9600 |
| C8—H8 | 0.9300 | C26—H26C | 0.9600 |
| C9—C10 | 1.516 (3) | O5A—C26A | 1.430 (19) |
| C10—C11 | 1.508 (3) | O5A—H2O5 | 0.8200 |
| C10—H10A | 0.9700 | C26A—H26D | 0.9600 |
| C10—H10B | 0.9700 | C26A—H26E | 0.9600 |
| C11—C12 | 1.390 (3) | C26A—H26F | 0.9600 |

| | | | |
|---|---|---|---|
| C4—O2—C3 | 118.82 (14) | C14—C13—C12 | 121.06 (18) |
| C16—O3—C17 | 116.49 (14) | C14—C13—Cl1 | 120.10 (14) |
| C2—N1—C1 | 121.63 (16) | C12—C13—Cl1 | 118.83 (16) |
| C2—N1—H1N1 | 119.0 | C13—C14—C15 | 119.22 (17) |
| C1—N1—H1N1 | 117.4 | C13—C14—H14 | 120.4 |
| C18—N2—C19 | 121.82 (15) | C15—C14—H14 | 120.4 |
| C18—N2—H1N2 | 115.1 | C14—C15—C16 | 120.21 (19) |
| C19—N2—H1N2 | 123.0 | C14—C15—H15 | 119.9 |
| N1—C1—C24 | 113.56 (16) | C16—C15—H15 | 119.9 |
| N1—C1—H1A | 108.9 | O3—C16—C15 | 122.18 (17) |
| C24—C1—H1A | 108.9 | O3—C16—C11 | 117.25 (16) |
| N1—C1—H1B | 108.9 | C15—C16—C11 | 120.58 (18) |
| C24—C1—H1B | 108.9 | O3—C17—C18 | 111.27 (15) |
| H1A—C1—H1B | 107.7 | O3—C17—H17A | 109.4 |
| O1—C2—N1 | 124.17 (18) | C18—C17—H17A | 109.4 |
| O1—C2—C3 | 118.49 (17) | O3—C17—H17B | 109.4 |
| N1—C2—C3 | 117.30 (16) | C18—C17—H17B | 109.4 |
| O2—C3—C2 | 109.29 (15) | H17A—C17—H17B | 108.0 |
| O2—C3—H3A | 109.8 | O4—C18—N2 | 123.83 (18) |
| C2—C3—H3A | 109.8 | O4—C18—C17 | 118.33 (17) |
| O2—C3—H3B | 109.8 | N2—C18—C17 | 117.84 (16) |
| C2—C3—H3B | 109.8 | N2—C19—C20 | 114.22 (15) |
| H3A—C3—H3B | 108.3 | N2—C19—H19A | 108.7 |
| O2—C4—C5 | 124.25 (16) | C20—C19—H19A | 108.7 |
| O2—C4—C9 | 114.12 (15) | N2—C19—H19B | 108.7 |
| C5—C4—C9 | 121.62 (17) | C20—C19—H19B | 108.7 |
| C4—C5—C6 | 119.53 (17) | H19A—C19—H19B | 107.6 |
| C4—C5—H5 | 120.2 | C21—C20—C25 | 118.80 (18) |
| C6—C5—H5 | 120.2 | C21—C20—C19 | 119.64 (17) |

| | | | |
|---|---|---|---|
| C7—C6—C5 | 118.96 (17) | C25—C20—C19 | 121.47 (16) |
| C7—C6—H6 | 120.5 | C22—C21—C20 | 120.21 (19) |
| C5—C6—H6 | 120.5 | C22—C21—H21 | 119.9 |
| C6—C7—C8 | 121.69 (18) | C20—C21—H21 | 119.9 |
| C6—C7—Cl2 | 119.10 (14) | C23—C22—C21 | 120.62 (18) |
| C8—C7—Cl2 | 119.19 (15) | C23—C22—H22 | 119.7 |
| C9—C8—C7 | 120.19 (17) | C21—C22—H22 | 119.7 |
| C9—C8—H8 | 119.9 | C22—C23—C24 | 120.08 (19) |
| C7—C8—H8 | 119.9 | C22—C23—H23 | 120.0 |
| C8—C9—C4 | 118.01 (16) | C24—C23—H23 | 120.0 |
| C8—C9—C10 | 125.23 (16) | C25—C24—C23 | 118.84 (18) |
| C4—C9—C10 | 116.75 (16) | C25—C24—C1 | 121.56 (17) |
| C11—C10—C9 | 116.76 (16) | C23—C24—C1 | 119.60 (18) |
| C11—C10—H10A | 108.1 | C24—C25—C20 | 121.43 (17) |
| C9—C10—H10A | 108.1 | C24—C25—H25 | 119.3 |
| C11—C10—H10B | 108.1 | C20—C25—H25 | 119.3 |
| C9—C10—H10B | 108.1 | C26—O5—H1O5 | 109.5 |
| H10A—C10—H10B | 107.3 | C26A—O5A—H2O5 | 109.5 |
| C12—C11—C16 | 118.45 (17) | O5A—C26A—H26D | 109.5 |
| C12—C11—C10 | 120.39 (18) | O5A—C26A—H26E | 109.5 |
| C16—C11—C10 | 121.00 (17) | H26D—C26A—H26E | 109.5 |
| C13—C12—C11 | 120.43 (19) | O5A—C26A—H26F | 109.5 |
| C13—C12—H12 | 119.8 | H26D—C26A—H26F | 109.5 |
| C11—C12—H12 | 119.8 | H26E—C26A—H26F | 109.5 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*1···O5 | 0.98 | 2.31 | 3.039 (3) | 130 |
| N2—H1*N*2···O1$^{i}$ | 0.93 | 2.20 | 2.860 (2) | 128 |
| O5—H1*O*5···O4$^{ii}$ | 0.82 | 2.05 | 2.789 (3) | 150 |
| C26*A*—H26*F*···Cl2$^{iii}$ | 0.96 | 2.74 | 3.616 (3) | 149 |

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