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# 1-(3,5-Dichlorophenyl)-3-(2-methoxyphenyl)triaz-1-ene

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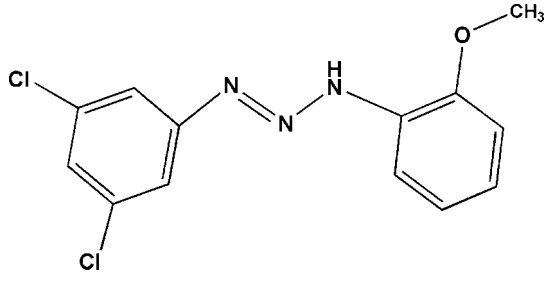
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.066; $wR$ factor = 0.137; data-to-parameter ratio = 20.7.

The title molecule, $C_{13}H_{11}Cl_2N_3O$, is almost planar and adopts a *trans* conformation with respect to the –N=N– bond; the dihedral angle between the rings is 3.47 (2)°. The N—N bond lengths indicate the presence of single- and double-bond characters and hence the –N=N—NH– moiety. In the crystal, inversion dimers linked by pairs of N—H···Cl hydrogen bonds occur, and C—H···$\pi$ and $\pi$–$\pi$ stacking interactions are also observed.

## Related literature

For background literature and the synthesis of related compounds, see: Rofouei *et al.* (2009). For the synthesis and molecular structure of a similar monochloro-substituted triazene, see: Rofouei *et al.* (2012).




## Experimental

### Crystal data

$C_{13}H_{11}Cl_2N_3O$
$M_r$ = 296.15
Monoclinic, $C2/c$
$a$ = 15.422 (3) Å
$b$ = 23.068 (5) Å
$c$ = 7.6141 (15) Å
$\beta$ = 92.60 (3)°
$V$ = 2706.0 (9) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.47 mm$^{-1}$
$T$ = 298 K
0.5 × 0.3 × 0.15 mm

### Data collection

Stoe IPDS 2T diffractometer
15133 measured reflections
3659 independent reflections
2178 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.162

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.137
$S$ = 1.07
3659 reflections
177 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

*Cg*1 is the centroid of the C2–C7 ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···Cl1$^{i}$ | 0.85 (2) | 2.69 (2) | 3.529 (2) | 170 (3) |
| C1—H1*C*···*Cg*1$^{ii}$ | 0.96 | 2.76 | 3.553 (4) | 140 |

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2511).

## References

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o724 [doi:10.1107/S1600536812005132]

## 1-(3,5-Dichlorophenyl)-3-(2-methoxyphenyl)triaz-1-ene

**Mohammad Reza Melardi, Maryam Aghamohamadi, Jafar Attar Gharamaleki, Mohammad Kazem Rofouei and Behrouz Notash**

### Comment

In continuation of our studies on the synthesis and characterization of trizene compounds as ligands in our laboratory (Rofouei *et al*., 2012; Rofouei *et al.*, 2009), we now report the crystal structure of the title compound.

The title molecule (Fig. 1) adopts *trans* configuration about the (–N2═N3–) bond and is almost planar with the dihedral angel between two aromatic rings 3.47 (2) °. Non-Classic N—H···Cl hydrogen bond with D···A distance of 3.529 (2) Å connect the individual molecules into dimers. The N1—N2 and N2—N3 bond distances are 1.323 (3) and 1.256 (3) Å, which indicate the presence of a single and a double bond characters, respectively. Another interesting feature of the title compound is the presence of $\pi$–$\pi$ [*Cg*1···*Cg*1 distance of 3.757 (2) Å] and edge-to-face C1—H1C···*Cg*1 stacking interactions between the methoxy hydrogen and the phenyl ring with H···$\pi$ distance of 2.76 Å, in which *Cg*1 is the center of (C2—C7) ring. Unit cell packing diagram of the title compound is presented in Fig. 2, showing N—H···Cl hydrogen bonds.

The bond distances and bond angles in the title compoiund are in agreement with the corresponding bond distances and bond angles reported for a closely related structure, [1-(2-methoxyphenyl)-3-(4-chlorophenyl)]triazene (Rofouei *et al.*, 2012).

### Experimental

To a 1 L flask in an ice bath, was added dichloroaniline (6.36 g, 0.05 mol) and HCl (4.68 g, 0.13 mol; d = 1.18 g.ml$^{-1}$). To the obtained solution was added dropwise a solution of sodium nitrite (4.14 g in 25 ml $H_2O$). Then, a diluted solution of *o*-anisidine (6.15 g, 0.05 mol) in methanol (10 ml) was added to the solution. The pH of the solution was adjusted at about 7–8 by adding a solution of sodium acetate ( 14.76 g, 0.18 mol) in 45 ml $H_2O$ as solvent. The solution was stirred for about 45 minutes, giving an orange precipitate. It was then filtered off and dried under vacuum. After dissolving in dichloromethane and recrystallization, orange crystals of the title compound were obtained.

### Refinement

N—H hydrogen atom were found in a difference Fourier map and refined isotropically with distance restraint of 0.85 (2) Å. All C—H hydrogen atoms were positioned geometrically and refined as riding atoms with C—H = 0.93 and 0.96 Å, $U_{iso}$(H) = 1.2$U_{eq}$(C) and 1.5$U_{eq}$(C) for aryl and methyl H atoms, respectively.

### Computing details

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA* (Stoe & Cie, 2005); data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

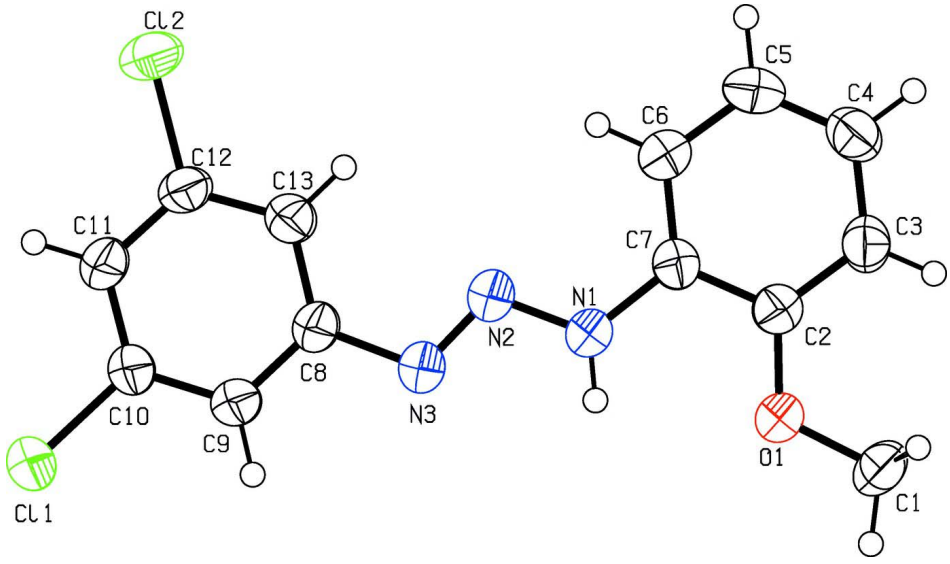


**Figure 1**

Molecular structure of the title compound with ellipsoids drawn at 50% probability level.

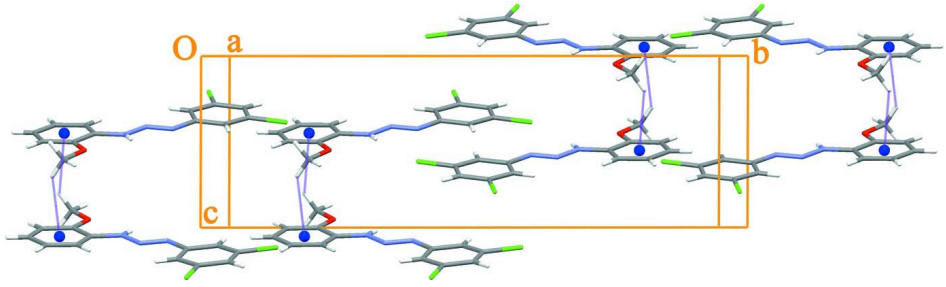


**Figure 2**

Part of the unit-cell packing of the title compound showing C—H···$\pi$ interactions between a methoxy H atom and the phenyl ring with a H···$\pi$ distance of 2.76 Å.

### 1-(3,5-Dichlorophenyl)-3-(2-methoxyphenyl)triaz-1-ene

*Crystal data*

$C_{13}H_{11}Cl_2N_3O$
$M_r$ = 296.15
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
*a* = 15.422 (3) Å
*b* = 23.068 (5) Å
*c* = 7.6141 (15) Å
$\beta$ = 92.60 (3)°
*V* = 2706.0 (9) Å$^3$
*Z* = 8

*F*(000) = 1216
$D_x$ = 1.454 Mg m$^{-3}$
Mo *K*$\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3659 reflections
$\theta$ = 1.6–29.4°
$\mu$ = 0.47 mm$^{-1}$
*T* = 298 K
Needle, orange
0.5 × 0.3 × 0.15 mm

*Data collection*

Stoe IPDS 2T diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator

Detector resolution: 0.15 pixels mm$^{-1}$
rotation method scans
15133 measured reflections
3659 independent reflections

2178 reflections with $I > 2\sigma(I)$
$R_{int} = 0.162$
$\theta_{max} = 29.4°$, $\theta_{min} = 1.6°$
$h = -21 \rightarrow 20$
$k = -31 \rightarrow 31$
$l = -10 \rightarrow 10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.137$
$S = 1.07$
3659 reflections
177 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 2.1152P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{min} = -0.28$ e Å$^{-3}$

*Special details*

**Experimental**. $^1$H-NMR (300 MHz, $d^6$-DMSO) $\delta$, p.p.m.: 3.83 (3*H*, $CH_3$), 6.73–7.69 (7*H*, aromatic groups) and 12.93(1*H*, NH group). $^{13}$C-NMR (100 MHz, DMSO) $\delta$, p.p.m.: 55.8 (O—$CH_3$), 111.9–153.8 (C atoms of aromatic rings). IR (KBr): 3314, 1601, 1566, 1473, 1255, 754 cm$^{-1}$.

**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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.15277 (6) | 0.62694 (3) | 0.38482 (14) | 0.0726 (3) |
| Cl2 | 0.38395 (5) | 0.46656 (4) | 0.20924 (14) | 0.0697 (3) |
| O1 | −0.07732 (12) | 0.25239 (8) | 0.5479 (3) | 0.0541 (5) |
| N1 | 0.04554 (15) | 0.32351 (9) | 0.4565 (4) | 0.0510 (6) |
| N2 | 0.10429 (14) | 0.36171 (9) | 0.4104 (3) | 0.0461 (6) |
| N3 | 0.07934 (15) | 0.41328 (9) | 0.4243 (3) | 0.0515 (6) |
| C1 | −0.14585 (18) | 0.21619 (13) | 0.6009 (5) | 0.0577 (8) |
| H1A | −0.1640 | 0.1912 | 0.5054 | 0.087* |
| H1B | −0.1939 | 0.2397 | 0.6335 | 0.087* |
| H1C | −0.1260 | 0.1932 | 0.6998 | 0.087* |
| C2 | −0.00257 (17) | 0.22621 (11) | 0.5001 (4) | 0.0438 (6) |
| C3 | 0.01140 (19) | 0.16686 (11) | 0.4955 (4) | 0.0494 (7) |
| H3 | −0.0323 | 0.1414 | 0.5256 | 0.059* |
| C4 | 0.0906 (2) | 0.14548 (12) | 0.4459 (4) | 0.0552 (7) |
| H4 | 0.1001 | 0.1057 | 0.4434 | 0.066* |
| C5 | 0.15531 (19) | 0.18293 (13) | 0.4004 (4) | 0.0544 (7) |
| H5 | 0.2086 | 0.1684 | 0.3685 | 0.065* |
| C6 | 0.14148 (18) | 0.24231 (12) | 0.4020 (4) | 0.0488 (7) |
| H6 | 0.1852 | 0.2674 | 0.3703 | 0.059* |

| | | | | |
|---|---|---|---|---|
| C7 | 0.06270 (17) | 0.26414 (10) | 0.4508 (4) | 0.0416 (6) |
| C8 | 0.14307 (17) | 0.45444 (11) | 0.3765 (4) | 0.0435 (6) |
| C9 | 0.12052 (17) | 0.51217 (11) | 0.3953 (4) | 0.0484 (7) |
| H9 | 0.0659 | 0.5222 | 0.4327 | 0.058* |
| C10 | 0.17985 (18) | 0.55468 (11) | 0.3581 (4) | 0.0495 (7) |
| C11 | 0.26112 (18) | 0.54131 (12) | 0.3013 (4) | 0.0520 (7) |
| H11 | 0.3009 | 0.5703 | 0.2776 | 0.062* |
| C12 | 0.28154 (17) | 0.48354 (12) | 0.2808 (4) | 0.0491 (7) |
| C13 | 0.22487 (17) | 0.43961 (11) | 0.3177 (4) | 0.0482 (7) |
| H13 | 0.2405 | 0.4010 | 0.3041 | 0.058* |
| H1 | −0.0038 (13) | 0.3373 (12) | 0.481 (4) | 0.059 (9)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0684 (5) | 0.0384 (3) | 0.1140 (8) | 0.0020 (3) | 0.0377 (5) | 0.0057 (4) |
| Cl2 | 0.0474 (4) | 0.0709 (5) | 0.0930 (7) | 0.0123 (4) | 0.0281 (4) | 0.0165 (4) |
| O1 | 0.0418 (10) | 0.0424 (9) | 0.0791 (15) | −0.0020 (8) | 0.0160 (10) | 0.0022 (9) |
| N1 | 0.0397 (12) | 0.0396 (11) | 0.0748 (18) | −0.0021 (10) | 0.0150 (12) | 0.0018 (11) |
| N2 | 0.0435 (12) | 0.0408 (11) | 0.0546 (15) | −0.0040 (9) | 0.0080 (11) | 0.0038 (10) |
| N3 | 0.0459 (12) | 0.0395 (11) | 0.0699 (17) | −0.0025 (10) | 0.0140 (11) | 0.0055 (11) |
| C1 | 0.0398 (14) | 0.0620 (17) | 0.072 (2) | −0.0061 (13) | 0.0109 (15) | 0.0102 (16) |
| C2 | 0.0407 (13) | 0.0432 (12) | 0.0476 (16) | −0.0006 (11) | 0.0034 (12) | −0.0013 (12) |
| C3 | 0.0539 (16) | 0.0411 (13) | 0.0535 (18) | −0.0062 (12) | 0.0065 (14) | 0.0026 (12) |
| C4 | 0.0650 (18) | 0.0421 (13) | 0.059 (2) | 0.0071 (13) | 0.0111 (16) | −0.0018 (13) |
| C5 | 0.0501 (15) | 0.0554 (15) | 0.0585 (19) | 0.0131 (14) | 0.0107 (14) | −0.0068 (14) |
| C6 | 0.0419 (14) | 0.0493 (14) | 0.0559 (19) | −0.0012 (12) | 0.0089 (13) | −0.0008 (13) |
| C7 | 0.0430 (14) | 0.0371 (12) | 0.0446 (16) | −0.0022 (11) | 0.0028 (12) | −0.0007 (11) |
| C8 | 0.0427 (13) | 0.0417 (12) | 0.0465 (16) | −0.0021 (11) | 0.0087 (12) | 0.0062 (11) |
| C9 | 0.0438 (14) | 0.0438 (13) | 0.0589 (19) | 0.0034 (11) | 0.0155 (13) | 0.0044 (13) |
| C10 | 0.0498 (15) | 0.0369 (12) | 0.063 (2) | 0.0025 (11) | 0.0147 (14) | 0.0079 (12) |
| C11 | 0.0457 (15) | 0.0467 (14) | 0.065 (2) | −0.0014 (12) | 0.0157 (14) | 0.0126 (13) |
| C12 | 0.0399 (13) | 0.0515 (14) | 0.0570 (19) | 0.0058 (12) | 0.0151 (13) | 0.0093 (13) |
| C13 | 0.0500 (16) | 0.0418 (13) | 0.0538 (19) | 0.0057 (12) | 0.0118 (14) | 0.0057 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C10 | 1.733 (3) | C4—C5 | 1.376 (4) |
| Cl2—C12 | 1.739 (3) | C4—H4 | 0.9300 |
| O1—C2 | 1.365 (3) | C5—C6 | 1.386 (4) |
| O1—C1 | 1.420 (3) | C5—H5 | 0.9300 |
| N1—N2 | 1.323 (3) | C6—C7 | 1.381 (4) |
| N1—C7 | 1.396 (3) | C6—H6 | 0.9300 |
| N1—H1 | 0.854 (17) | C8—C9 | 1.386 (4) |
| N2—N3 | 1.256 (3) | C8—C13 | 1.400 (4) |
| N3—C8 | 1.426 (3) | C9—C10 | 1.379 (4) |
| C1—H1A | 0.9600 | C9—H9 | 0.9300 |
| C1—H1B | 0.9600 | C10—C11 | 1.379 (4) |
| C1—H1C | 0.9600 | C11—C12 | 1.380 (4) |
| C2—C3 | 1.387 (4) | C11—H11 | 0.9300 |

| | | | |
|---|---|---|---|
| C2—C7 | 1.398 (4) | C12—C13 | 1.375 (4) |
| C3—C4 | 1.386 (4) | C13—H13 | 0.9300 |
| C3—H3 | 0.9300 | | |

| | | | |
|---|---|---|---|
| C2—O1—C1 | 117.7 (2) | C7—C6—C5 | 120.0 (3) |
| N2—N1—C7 | 120.8 (2) | C7—C6—H6 | 120.0 |
| N2—N1—H1 | 116 (2) | C5—C6—H6 | 120.0 |
| C7—N1—H1 | 123 (2) | C6—C7—N1 | 122.4 (2) |
| N3—N2—N1 | 113.1 (2) | C6—C7—C2 | 119.8 (2) |
| N2—N3—C8 | 113.0 (2) | N1—C7—C2 | 117.8 (2) |
| O1—C1—H1A | 109.5 | C9—C8—C13 | 120.1 (2) |
| O1—C1—H1B | 109.5 | C9—C8—N3 | 115.7 (2) |
| H1A—C1—H1B | 109.5 | C13—C8—N3 | 124.1 (2) |
| O1—C1—H1C | 109.5 | C10—C9—C8 | 119.4 (2) |
| H1A—C1—H1C | 109.5 | C10—C9—H9 | 120.3 |
| H1B—C1—H1C | 109.5 | C8—C9—H9 | 120.3 |
| O1—C2—C3 | 125.3 (2) | C11—C10—C9 | 121.7 (2) |
| O1—C2—C7 | 115.0 (2) | C11—C10—Cl1 | 118.6 (2) |
| C3—C2—C7 | 119.8 (3) | C9—C10—Cl1 | 119.6 (2) |
| C4—C3—C2 | 119.9 (3) | C10—C11—C12 | 117.8 (2) |
| C4—C3—H3 | 120.1 | C10—C11—H11 | 121.1 |
| C2—C3—H3 | 120.1 | C12—C11—H11 | 121.1 |
| C5—C4—C3 | 120.2 (3) | C13—C12—C11 | 122.5 (2) |
| C5—C4—H4 | 119.9 | C13—C12—Cl2 | 119.5 (2) |
| C3—C4—H4 | 119.9 | C11—C12—Cl2 | 117.9 (2) |
| C4—C5—C6 | 120.3 (3) | C12—C13—C8 | 118.4 (2) |
| C4—C5—H5 | 119.8 | C12—C13—H13 | 120.8 |
| C6—C5—H5 | 119.8 | C8—C13—H13 | 120.8 |

| | | | |
|---|---|---|---|
| C7—N1—N2—N3 | −179.4 (3) | C3—C2—C7—N1 | 179.6 (3) |
| N1—N2—N3—C8 | 179.4 (3) | N2—N3—C8—C9 | −177.7 (3) |
| C1—O1—C2—C3 | 1.4 (4) | N2—N3—C8—C13 | 0.7 (4) |
| C1—O1—C2—C7 | −179.4 (3) | C13—C8—C9—C10 | −0.9 (5) |
| O1—C2—C3—C4 | −179.4 (3) | N3—C8—C9—C10 | 177.6 (3) |
| C7—C2—C3—C4 | 1.5 (5) | C8—C9—C10—C11 | 0.5 (5) |
| C2—C3—C4—C5 | −0.3 (5) | C8—C9—C10—Cl1 | −178.9 (2) |
| C3—C4—C5—C6 | −0.7 (5) | C9—C10—C11—C12 | 0.6 (5) |
| C4—C5—C6—C7 | 0.5 (5) | Cl1—C10—C11—C12 | 180.0 (3) |
| C5—C6—C7—N1 | 179.3 (3) | C10—C11—C12—C13 | −1.2 (5) |
| C5—C6—C7—C2 | 0.6 (5) | C10—C11—C12—Cl2 | −180.0 (2) |
| N2—N1—C7—C6 | 1.8 (4) | C11—C12—C13—C8 | 0.8 (5) |
| N2—N1—C7—C2 | −179.5 (3) | Cl2—C12—C13—C8 | 179.5 (2) |
| O1—C2—C7—C6 | 179.2 (3) | C9—C8—C13—C12 | 0.3 (4) |
| C3—C2—C7—C6 | −1.6 (5) | N3—C8—C13—C12 | −178.0 (3) |
| O1—C2—C7—N1 | 0.4 (4) | | |

*Hydrogen-bond geometry (Å, º)*

*Cg*1 is the centroid of the C2–C7 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···Cl1$^{i}$ | 0.85 (2) | 2.69 (2) | 3.529 (2) | 170 (3) |
| N1—H1···O1 | 0.85 (2) | 2.33 (3) | 2.624 (3) | 100 (2) |
| C1—H1*C*···*Cg*1$^{ii}$ | 0.96 | 2.76 | 3.553 (4) | 140 |

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