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Crystal structures and Hirshfeld surface analyses of methyl 4-{2,2-dichloro-1-[(*E*)-phenyldiazenyl]ethenyl}benzoate, methyl 4-{2,2-dichloro-1-[(*E*)-(4-methylphenyl)diazenyl]ethenyl}benzoate and methyl 4-{2,2-dichloro-1-[(*E*)-(3,4-dimethylphenyl)-diazenyl]ethenyl}benzoate

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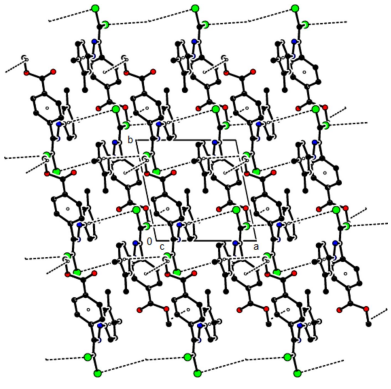
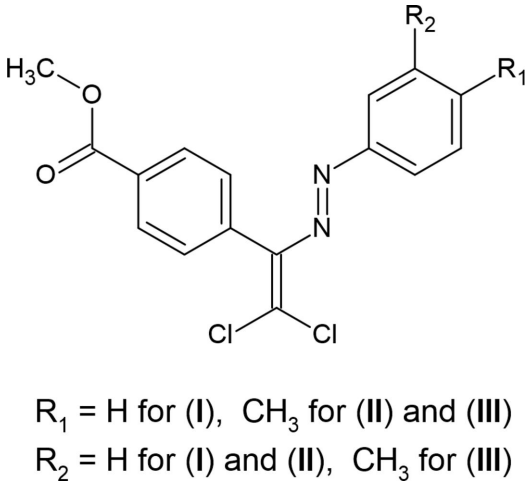
The crystal structures and Hirshfeld surface analyses of three similar azo compounds are reported. Methyl 4-{2,2-dichloro-1-[(*E*)-phenyldiazenyl]ethenyl}benzoate, $C_{16}H_{12}Cl_2N_2O_2$, (**I**), and methyl 4-{2,2-dichloro-1-[(*E*)-(4-methylphenyl)diazenyl]ethenyl}benzoate, $C_{17}H_{14}Cl_2N_2O_2$, (**II**), crystallize in the space group $P2_1/c$ with $Z = 4$, and methyl 4-{2,2-dichloro-1-[(*E*)-(3,4-dimethylphenyl)-diazenyl]ethenyl}benzoate, $C_{18}H_{16}Cl_2N_2O_2$, (**III**), in the space group $P\overline{1}$ with $Z = 2$. In the crystal of (**I**), molecules are linked by C—H···N hydrogen bonds, forming chains with $C(6)$ motifs parallel to the *b* axis. Short intermolecular Cl···O contacts of 2.8421 (16) Å and weak van der Waals interactions between these chains stabilize the crystal structure. In (**II**), molecules are linked by C—H···O hydrogen bonds and C—Cl···π interactions, forming layers parallel to (010). Weak van der Waals interactions between these layers consolidate the molecular packing. In (**III**), molecules are linked by C—H···π and C—Cl···π interactions forming chains parallel to [011]. Furthermore, these chains are connected by C—Cl···π interactions parallel to the *a* axis, forming $(0\overline{1}1)$ layers. The stability of the molecular packing is ensured by van der Waals forces between these layers.



1. Chemical context

When manufacturing new insecticides and pesticides, it is important that they are harmless to the environment and humans. This condition is fulfilled for most biopesticides. For example, methylbenzoate is considered to be a bio-insecticide (Mostafiz *et al.*, 2022; Chen *et al.*, 2015; Damalas & Eleftherohorinos, 2011; Goulson, 2013; Naqqash *et al.*, 2016; Zikankuba *et al.*, 2019) and is reported to be less harmful to the human body and the environment. Methylbenzoate is also found as a metabolite in plants and has an attractive odour to insects. At the same time, methyl benzoate is very effective as a pesticide against agricultural and warehouse pests (Isman, 2015, 2020; Pavela, 2016; Pavela & Benelli, 2016). It can therefore be concluded that methyl benzoate and its derivatives might exhibit applications as pesticides and insecticides,

and the synthesis of such or related biopesticides is an urgent issue. Taking this into account, we focused on phenylhydrazones that were obtained from the reaction of methyl 4-formylbenzoate with the corresponding phenylhydrazines (Maharramov *et al.*, 2018; Nenajdenko *et al.*, 2020, Shikhaliyev *et al.*, 2018, 2019*a*,*b*, 2021*a*,*b*,*c*), and the synthesis of methyl (*E*)-4-{2,2-dichloro-1-[(substitutedphenyl)diazenyl]vinyl}benzoate derivatives was carried out from the reaction of the latter with CCl_4. The here synthesized dichlorodiazadiene derivatives (**I**), (**II**) and (**III**) and arylhydrazo derivatives of α-keto esters obtained from their solvolysis are intended to be studied in future research as compounds with the above-mentioned properties (Fig. 1).



2. Structural commentary

The central molecular fragment of (**I**), C1/C2/N1/N2/C3/C11/Cl1/Cl2, is almost planar (Fig. 2), with a root-mean-square (r.m.s.) deviation of fitted atoms from the least-squares plane of 0.0471 Å. This plane forms dihedral angles of 23.39 (6) and 56.98 (4)°, respectively, with the planes of the phenyl (C11–C16) and methyl benzoate (C3–C8) rings. The central molecular fragment of (**II**), C1/C2/N2/N1/C3/C11/Cl1/Cl2, is likewise planar with an r.m.s. deviation of fitted atoms of 0.0680 Å (Fig. 3) and makes dihedral angles of 14.87 (8) and 70.88 (3)°, respectively, with the planes of the 4-methylphenyl (C11–C16) and methyl benzoate (C3–C8) rings. The central molecular fragment of (**III**), C1/C2/N1/N2/C3/C11/Cl1/Cl2 (r.m.s. deviation of fitted atoms = 0.0261 Å; Fig. 4) forms dihedral angles of 7.59 (6) and 69.58 (3)°, respectively, with the planes of the 3,4-dimethylphenyl (C11–C16) and methyl benzoate (C3–C8) rings.

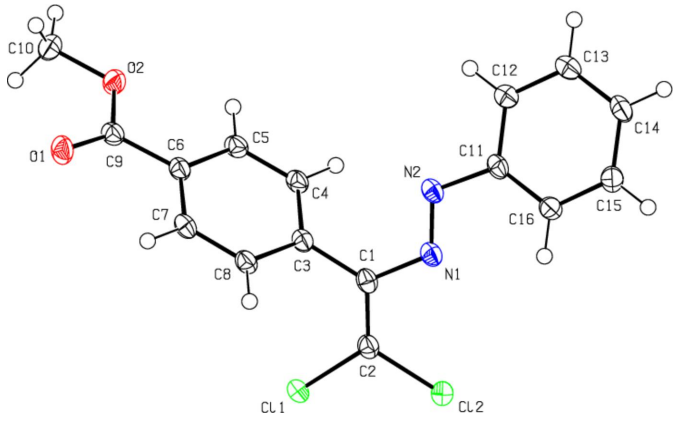


Figure 2
The molecular structure of (**I**), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

All bond lengths and angles in (**I**), (**II**) and (**III**) are in agreement with those reported for the related azo compounds discussed in the *Database survey* section.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal of (**I**), molecules are linked by C—H···N hydrogen bonds, forming chains with *C*(6) motifs (Bernstein *et al.*, 1995) extending parallel to the *b* axis. Short intermolecular Cl1···O1($-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$) contacts of 2.8421 (16) Å and weak van der Waals interactions between these chains stabi-

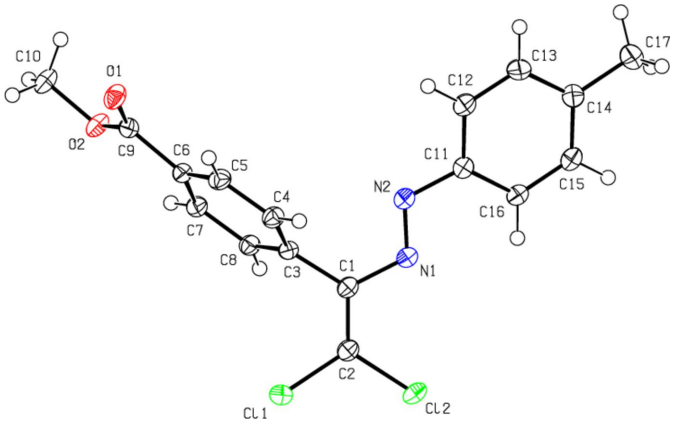


Figure 3
The molecular structure of (**II**), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

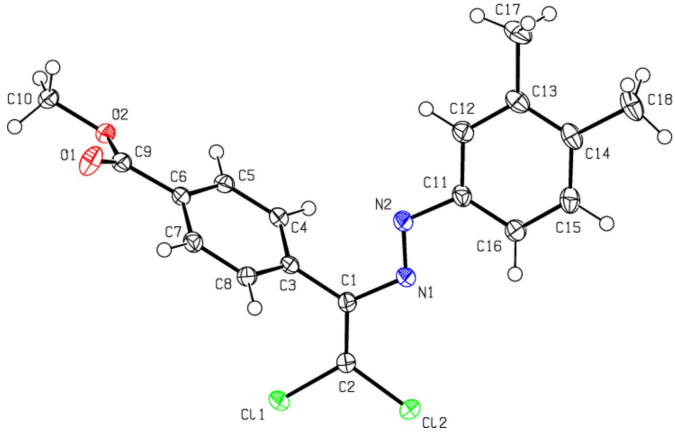


Figure 4
The molecular structure of (**III**), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

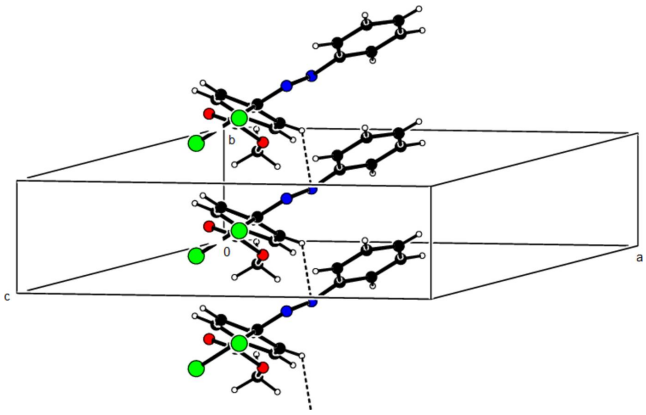


Figure 5
A general view of the C—H···N hydrogen bonds in the crystal structure of (**I**).

lize the crystal structure (Table 1; Fig. 5). In the crystal of (**II**), molecules are linked by C—H···O hydrogen bonds and C—Cl···π interactions [C2—Cl2···$Cg1^a$: Cl2···$Cg1^a$ = 3.5596 (8) Å, C2—Cl2···$Cg1^a$ = 101.11 (6)°; symmetry code (*a*) $x, \frac{3}{2}-y, -\frac{1}{2}+z$; *Cg*1 is the centroid of the C3–C8 benzene ring], forming layers parallel to (010) (Table 2; Fig. 6). Weak van der Waals interactions between these layers stabilize the molecular packing. In the crystal of (**III**), molecules are linked by C—H···π and C—Cl···π interactions, forming chains parallel to [011]. Furthermore, these chains are connected by C—Cl···π interactions [C2—Cl1···$Cg2^a$: Cl1···$Cg2^a$ = 3.5398 (8) Å, C2—Cl1···$Cg2^a$ = 92.51 (5)°; C2—Cl2···$Cg2^b$: Cl2···$Cg2^b$ = 3.9545 (8) Å, C2—Cl2···$Cg2^b$ = 88.18 (5)°; symmetry codes (*a*) $-x, -y, 1-z$; (*b*) $1-x, -y, 1-z$; *Cg*2 is the centroid of the 3,4-dimethylphenyl ring (C11–C16)] parallel to the *a* axis, forming layers parallel to $(0\bar{1}1)$ (Table 3; Figs. 7, 8 and 9). The stability of the molecular packing is ensured by van der Waals forces between these layers.

To quantify intermolecular interactions between the molecules in the crystal structures of (**I**), (**II**) and (**III**), Hirshfeld surface analyses were performed, together with two-dimensional fingerprint plots by using *CrystalExplorer* (Spackman *et al.*, 2021). The two-dimensional fingerprint plots are shown in Fig. 10. Comparative interactions calculated for each compound are given in Table 3. The dominant interactions in the crystal packing of the title compounds are H···H [(**I**): 33.5%, (**II**): 39.7% and (**III**): 37.0%], Cl···H/H···Cl [(**I**): 20.5%, (**II**): 14.4% and (**III**): 19.1%], C···H/H···C [(**I**): 14.3%, (**II**): 14.5% and (**III**):16.0%] and O···H/H···O [(**I**): 8.1%, (**II**): 6.6% and (**III**): 8.7%]. These interactions play a crucial role in the overall stabilization of the crystal packing. The presence of different functional groups in the compounds leads to some differences in the remaining weak interactions.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···N2^{i}	0.95	2.54	3.191 (3)	126

Symmetry code: (i) $x, y-1, z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1^{i}	0.95	2.43	3.268 (2)	148
C13—H13···O1^{ii}	0.95	2.40	3.309 (3)	159

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

4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.42, update of September 2021; Groom *et al.*, 2016) for the

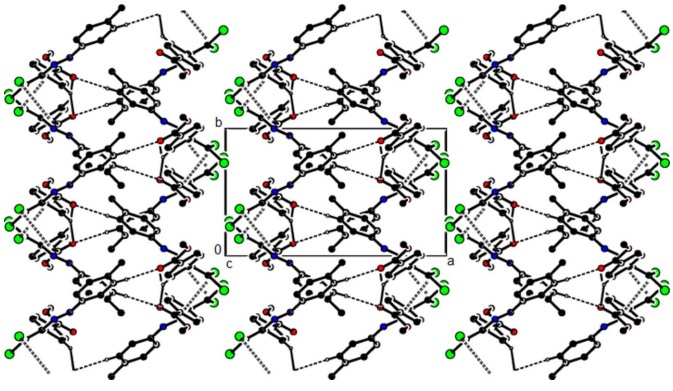


Figure 6
The crystal packing of (**II**) viewed along the *c* axis with intermolecular C—H···O and C—Cl···π interactions shown as dashed lines.

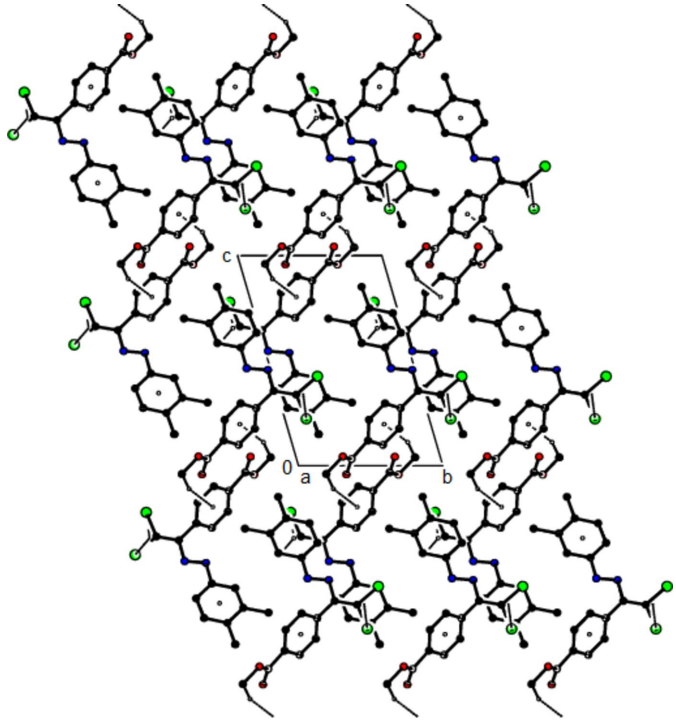


Figure 7
The crystal packing of (**III**) viewed along the *a* axis with C—H···π and C—Cl···π interactions shown as dashed lines.

Table 3
Percentage contributions of interatomic contacts to the Hirshfeld surface for the title compounds.

Contact	Percentage contribution		
	(I)	**(II)**	**(III)**
H···H	33.5	39.7	37.0
Cl···H/H···Cl	20.5	14.4	19.1
C···H/H···C	14.3	14.5	16.0
O···H/H···O	8.1	6.6	8.7
C···C	6.0	4.0	2.1
N···H/H···N	4.2	5.2	4.9
N···C/C···N	4.0	0.3	2.0
Cl···O/O···Cl	3.7	2.6	1.5
Cl···C/C···Cl	3.3	2.8	5.3
O···C/C···O	1.7	4.6	1.4
Cl···Cl	0.6	4.0	1.0
O···C/C···O	–	1.1	1.4
Cl···N/N·Cl	–	0.8	0.4
O···C/C···O	–	–	0.2

(E)-1-(2,2-dichloro-1-phenylethenyl)-2-phenyldiazene moiety resulted in 36 hits. Eighteen compounds are closely related to the title compound, *viz*. those with CSD refcodes NIKXEO (Maharramov *et al.*, 2023), NIKXIS (Maharramov *et al.*, 2023), NIKXOY (Maharramov *et al.*, 2023), NIKXUE (Maharramov *et al.*, 2023), TAZDIL (Atioğlu *et al.*, 2022*a*), HEHKEO (Akkurt *et al.*, 2022), ECUDAL (Atioğlu *et al.*, 2022*b*), PAXDOL (Çelikesir *et al.*, 2022), CANVUM (Shikhaliyev *et al.*, 2021*d*), EBUCUD (Shikhaliyev *et al.*, 2021*a*), GUPHIL (Özkaraca *et al.*, 2020*a*), DULTAI (Özkaraca *et al.*, 2020*b*), XIZREG (Atioğlu *et al.*, 2019), HODQAV (Shikhaliyev *et al.*, 2019*a*), HONBUK (Akkurt *et al.*, 2019), HONBOE (Akkurt *et al.*, 2019), LEQXOX (Shikhaliyev *et al.*, 2018) and LEQXIR (Shikhaliyev *et al.*, 2018).

In the crystal structures of NIKXEO and NIKXIS, molecules are linked by C—H···π and C—Cl···π interactions, forming layers parallel to ($\bar{1}$01), while molecules of NIKXOY are linked by C—H···O contacts, C—H···π and C—Cl···π interactions, forming layers parallel to ($\bar{3}$02). The stability of the molecular packing is ensured by van der Waals forces between these layers. In the crystal structure of NIKXUE, molecules are linked by C—H···π and C—Cl···π interactions, forming a tri-periodic network. The molecules in TAZDIL are joined into layers parallel to (011) by C—H···O and C—H···F hydrogen bonds. C—Br···π and C—F···π contacts, as well as π—π stacking interactions strengthen the crystal packing. C—H···Br interactions connect the molecules in the crystal of the polymorph-1 of HEHKEO, resulting in zigzag *C*(8) chains parallel to [100]. These chains are connected by C—Br···π interactions into layers parallel to (001). van der Waals interactions between the layers contribute to the crystal cohesion. In the crystals of ECUDAL, C—H···O hydrogen bonds link molecules into chains. These chains are linked by face-to-face π–π stacking interactions, resulting in a layered structure. Short intermolecular Br···O contacts and van der Waals interactions between the layers aid in the cohesion of the crystal packing. The molecules in the crystal of PAXDOL are connected into chains running parallel to [001] by C—H···O hydrogen bonds. C—F···π contacts and π–π stacking interactions help to consolidate the crystal packing, and short Br···O [2.9828 (13) Å] distances are also observed. In CANVUM, the molecules are linked by C—H···N interactions along [100], forming a *C*(6) chain. The molecules are further connected by C—Cl···π interactions and face-to-face π–π stacking interactions, resulting in ribbons along [100]. The crystal structure of EBUCUD features short C—H···Cl and C—H···O contacts and C—H···π and van der Waals inter-

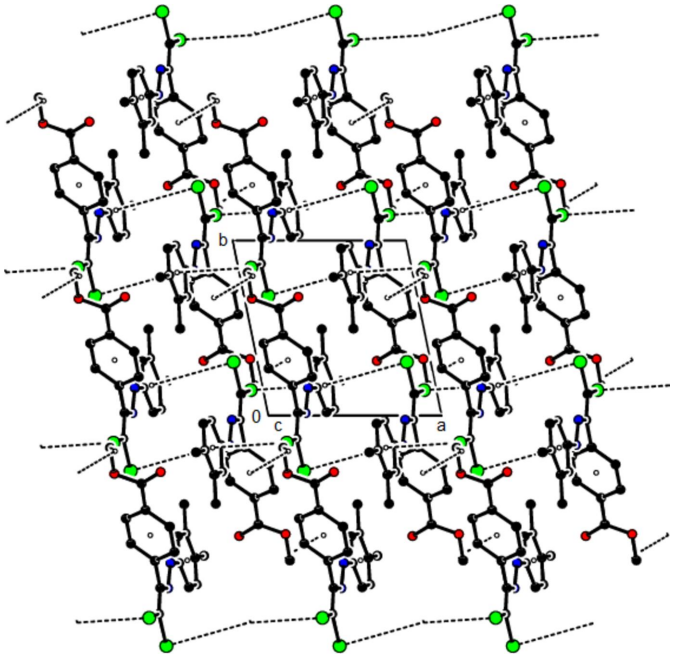


Figure 9
The crystal packing of (**III**) viewed along the *c* axis with C—H···π and C—Cl···π interactions.

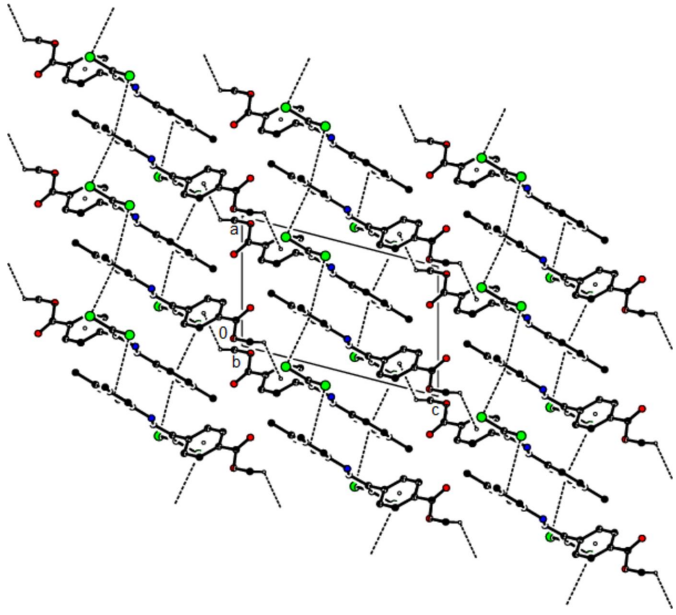


Figure 8
The crystal packing of (**III**) viewed along the *b* axis with C—H···π and C—Cl···π interactions shown as dashed lines.

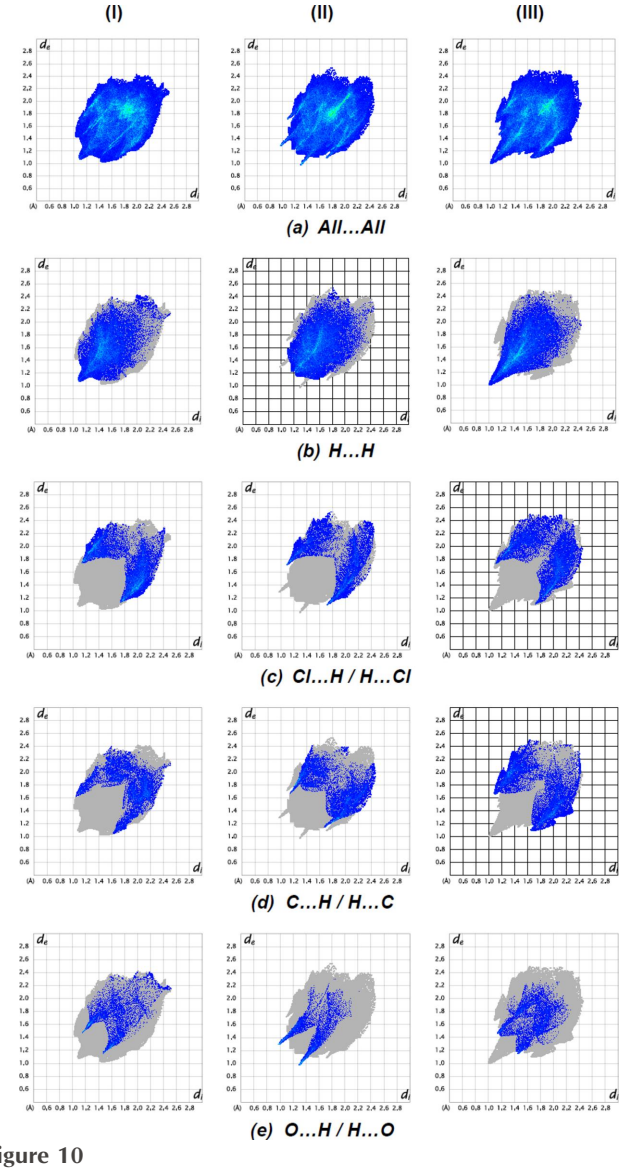


Figure 10
The full two-dimensional fingerprint plots for (**I**), (**II**) and (**III**), showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) Cl···H/H···Cl, (*d*) C···H/H···C, and (*e*) O···H/H···O interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

actions. In GUPHIL, molecules are associated into inversion dimers *via* short Cl···Cl contacts [3.3763 (9) Å]. In DULTAI, the crystal structure is stabilized by a short C—H···Cl contact, C—Cl···π and van der Waals interactions. In XIZREG, the molecules are linked by C—H···O hydrogen bonds into zigzag chains running along [001]. The crystal packing also features C—Cl···π, C—F···π and N—O···π interactions. In HODQAV, molecules are stacked in columns along [100] *via* weak C—H···Cl hydrogen bonds and face-to-face π—π stacking interactions. The crystal packing is further consolidated by short Cl···Cl contacts. In HONBUK and HONBOE, molecules are linked through weak *X*···Cl contacts (*X* = Cl for HONBUK and Br for HONBOE), C—H···Cl and C—Cl···π interactions into sheets parallel to (001). Additional van der Waals interactions consolidate the three-dimensional packing. In the crystals of LEQXOX, C—H···N and short Cl···Cl contacts are observed and in LEQXIR, C—H···N and C—H···O hydrogen bonds and short C—Cl···O contacts occur.

5. Synthesis and crystallization

Dyes (**I**), (**II**) and (**III**) were synthesized according to a literature protocol (Maharramov *et al.*, 2018).

For dye (**I**), a 20 ml screw-neck vial was charged with DMSO (10 ml), methyl (*E*)-4-[(2-phenylhydrazineylidene) methyl]benzoate (254 mg, 1 mmol), tetramethylethylenediamine (TMEDA) (295 mg, 2.5 mmol), CuCl (2 mg, 0.02 mmol) and CCl_4 (1 mmol). After 1–3 h (until TLC analysis showed complete consumption of the corresponding Schiff base), the reaction mixture was poured into a ∼0.01 *M* solution of HCl (100 ml, pH = 2–3), and extracted with dichloromethane (3 × ∼20 ml). The combined organic phase was washed with water (3× ∼50 ml), brine (30 ml), dried over anhydrous Na_2SO_4 and concentrated *in vacuo* using a rotary evaporator. The residue was purified by column chromatography on silica gel using appropriate mixtures of hexane and dichloromethane (*v*/*v*: 5/1–3/1–1/1). A red solid was obtained (yield 72%); m.p. 375 K. ^{1}H NMR (300 MHz, chloroform-*d*) δ 8.16–8.10 (*m*, 2H), 7.77 (*dd*, *J* = 6.8, 3.0 Hz, 2H), 7.48–7.43 (*m*, 3H), 7.29 (*d*, *J* = 8.3 Hz, 2H), 3.97 (*s*, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.2, 137.4, 132.0, 131.8, 130.1, 129.3, 129.1, 123.6, 123.2, 121.3, 120.0, 52.2.

For dye (**II**), the procedure was the same as that for (**I**) using methyl (*E*)-4-{[2-(*p*-tolyl)hydrazineylidene]methyl} benzoate (268 mg, 1 mmol). A red solid was obtained (yield 78%); m.p. 399 K. ^{1}H NMR (300 MHz, chloroform-*d*) δ 8.13 (*d*, *J* = 8.3 Hz, 2H), 7.69 (*d*, *J* = 8.2 Hz, 2H), 7.32–7.22 (*m*, 4H), 3.95 (*s*, 3H), 2.40 (*s*, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 151.5, 150.9, 142.6, 137.6, 134.8, 130.2, 129.8, 129.3, 123.3, 52.2, 21.6.

For dye (**III**), the procedure was the same as that for (**I**) using methyl (*E*)-4-{[2-(3,4-dimethylphenyl)hydrazineylidene] methyl}benzoate (282 mg, 1 mmol). A red solid was obtained (yield 73%); m.p. 405 K. ^{1}H NMR (300 MHz, chloroform-*d*) δ 8.14 (*d*, *J* = 8.2 Hz, 2H), 7.60–7.52 (*m*, 2H), 7.29 (*d*, *J* = 8.1 Hz, 2H), 7.20 (*d*, *J* = 8.0 Hz, 1H), 3.96 (*s*, 3H), 2.31 (*s*, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.5, 151.2, 141.4, 137.7, 137.4, 134.5, 130.3, 130.2, 129.3, 124.6, 120.7, 52.2, 20.0.

Compounds (**I**), (**II**), and (**III**) were dissolved in dichloromethane and then left at room temperature for slow evaporation; red crystals of all compounds suitable for X-rays started to form after *ca* 2 d.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. In all three compounds (**I**), (**II**) and (**III**), all H atoms were positioned geometrically and treated as riding atoms, with C—H = 0.95–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C-methyl).

Table 4
Experimental details.

	(**I**)	(**II**)	(**III**)
Crystal data			
Chemical formula	$C_{16}H_{12}Cl_2N_2O_2$	$C_{17}H_{14}Cl_2N_2O_2$	$C_{18}H_{16}Cl_2N_2O_2$
M_r	335.18	349.20	363.23
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$
Temperature (K)	100	100	100
a, b, c (Å)	15.47572 (16), 4.16896 (4), 23.2257 (2)	15.6177 (2), 8.47502 (11), 13.10365 (17)	8.22057 (10), 8.53211 (9), 13.08729 (14)
α, β, γ (°)	90, 100.1964 (9), 90	90, 109.6555 (15), 90	103.9484 (9), 101.9047 (10), 98.0600 (9)
V (Å^3)	1474.80 (2)	1633.34 (4)	854.09 (2)
Z	4	4	2
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	4.04	3.67	3.53
Crystal size (mm)	0.22 × 0.13 × 0.11	0.26 × 0.19 × 0.17	0.20 × 0.15 × 0.09
Data collection			
Diffractometer	XtaLAB Synergy, Dualflex, HyPix	XtaLAB Synergy, Dualflex, HyPix	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)
T_{min}, T_{max}	0.404, 0.600	0.307, 0.530	0.349, 0.700
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23940, 3151, 2823	28857, 3469, 3197	29808, 3632, 3340
R_{int}	0.060	0.054	0.063
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.634	0.634	0.634
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.103, 1.04	0.038, 0.108, 1.13	0.035, 0.097, 1.08
No. of reflections	3151	3469	3632
No. of parameters	200	211	220
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.46, −0.32	0.30, −0.48	0.31, −0.46

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

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References

Akkurt, M., Shikhaliyev, N. Q., Suleymanova, G. T., Babayeva, G. V., Mammadova, G. Z., Niyazova, A. A., Shikhaliyeva, I. M. & Toze, F. A. A. (2019). *Acta Cryst.* E**75**, 1199–1204.

Akkurt, M., Yıldırım, S. Ö., Shikhaliyev, N. Q., Mammadova, N. A., Niyazova, A. A., Khrustalev, V. N. & Bhattarai, A. (2022). *Acta Cryst.* E**78**, 732–736.

Atioğlu, Z., Akkurt, M., Shikhaliyev, N. Q., Mammadova, N. A., Babayeva, G. V., Khrustalev, V. N. & Bhattarai, A. (2022*a*). *Acta Cryst.* E**78**, 530–535.

Atioğlu, Z., Akkurt, M., Shikhaliyev, N. Q., Mammadova, N. A., Babayeva, G. V., Khrustalev, V. N. & Bhattarai, A. (2022*b*). *Acta Cryst.* E**78**, 804–808.

Atioğlu, Z., Akkurt, M., Shikhaliyev, N. Q., Suleymanova, G. T., Bagirova, K. N. & Toze, F. A. A. (2019). *Acta Cryst.* E**75**, 237–241.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Çelikesir, S. T., Akkurt, M., Shikhaliyev, N. Q., Mammadova, N. A., Suleymanova, G. T., Khrustalev, V. N. & Bhattarai, A. (2022). *Acta Cryst.* E**78**, 404–408.

Chen, M., Chang, C. H., Tao, L. & Lu, C. (2015). *Pediatrics*, **136**, 719–729.

Damalas, C. A. & Eleftherohorinos, I. G. (2011). *Int. J. Environ. Res. Publ. Heal.* **8**, 1402–1419.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Goulson, D. (2013). *J. Appl. Ecol.* **50**, 977–987.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Isman, M. B. (2015). *Pest. Manag. Sci.* **71**, 1587–1590.

Isman, M. B. (2020). *Phytochem. Rev.* **19**, 235–241.

Maharramov, A., Shikhaliyev, N. Q., Qajar, A., Atakishiyeva, G. T., Niyazova, A., Khrustalev, V. N., Akkurt, M., Yıldırım, S. Ö. & Bhattarai, A. (2023). *Acta Cryst.* E**79**, 637–643.

Maharramov, A. M., Shikhaliyev, N. Q., Suleymanova, G. T., Gurbanov, A. V., Babayeva, G. V., Mammadova, G. Z., Zubkov, F. I., Nenajdenko, V. G., Mahmudov, K. T. & Pombeiro, A. J. (2018). *Dyes Pigments*, **159**, 135–141.

Mostafiz, M. M., Hassan, E. & Lee, K. Y. (2022). *Agriculture*, **12**, 378.

Naqqash, M. N., Gökçe, A., Bakhsh, A. & Salim, M. (2016). *Parasitol. Res.* **115**, 1363–1373.

Nenajdenko, V. G., Shikhaliyev, N. G., Maharramov, A. M., Bagirova, K. N., Suleymanova, G. T., Novikov, A. S., Khrustalev, V. N. & Tskhovrebov, A. G. (2020). *Molecules*, **25**, 5013.

Özkaraca, K., Akkurt, M., Shikhaliyev, N. Q., Askerova, U. F., Suleymanova, G. T., Mammadova, G. Z. & Shadrack, D. M. (2020*a*). *Acta Cryst.* E**76**, 1251–1254.

Özkaraca, K., Akkurt, M., Shikhaliyev, N. Q., Askerova, U. F., Suleymanova, G. T., Shikhaliyeva, I. M. & Bhattarai, A. (2020*b*). *Acta Cryst.* E**76**, 811–815.
Pavela, R. (2016). *Plant Prot. Sci.* **52**, 229–241.
Pavela, R. & Benelli, G. (2016). *Trends Plant Sci.* **21**, 1000–1007.
Rigaku OD (2021). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Shikhaliyev, N. G., Maharramov, A. M., Bagirova, K. N., Suleymanova, G. T., Tsyrenova, B. D., Nenajdenko, V. G., Novikov, A. S., Khrustalev, V. N. & Tskhovrebov, A. G. (2021*b*). *Mendeleev Commun.* **31**, 191–193.
Shikhaliyev, N. G., Maharramov, A. M., Suleymanova, G. T., Babayeva, G. V., Mammadova, G. Z., Shikhaliyeva, I. M., Babazade, A. A. & Nenajdenko, V. G. (2021*c*). *Arkivoc*, pp. 67–75.
Shikhaliyev, N. G., Suleymanova, G. T., İsrayilova, A. A., Ganbarov, K. G., Babayeva, G. V., Garazadeh, K. A., Mammadova, G. Z. & Nenajdenko, V. G. (2019*b*). *Org. Chem.* pp. 64–73.
Shikhaliyev, N. Q., Ahmadova, N. E., Gurbanov, A. V., Maharramov, A. M., Mammadova, G. Z., Nenajdenko, V. G., Zubkov, F. I., Mahmudov, K. T. & Pombeiro, A. J. L. (2018). *Dyes Pigments*, **150**, 377–381.
Shikhaliyev, N. Q., Atioğlu, Z., Akkurt, M., Qacar, A. M., Askerov, R. K. & Bhattarai, A. (2021*a*). *Acta Cryst.* E**77**, 965–970.
Shikhaliyev, N. Q., Çelikesir, S. T., Akkurt, M., Bagirova, K. N., Suleymanova, G. T. & Toze, F. A. A. (2019*a*). *Acta Cryst.* E**75**, 465–469.
Shikhaliyev, N. Q., Özkaraca, K., Akkurt, M., Bagirova, X. N., Suleymanova, G. T., Abdulov, M. S. & Mlowe, S. (2021*d*). *Acta Cryst.* E**77**, 1158–1163.
Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.
Zikankuba, V. L., Mwanyika, G., Ntwenya, J. E. & James, A. (2019). *Cogent Food Agric.* **5**, 1601544.

supporting information

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Crystal structures and Hirshfeld surface analyses of methyl 4-{2,2-dichloro-1-[(*E*)-phenyldiazenyl]ethenyl}benzoate, methyl 4-{2,2-dichloro-1-[(*E*)-(4-methylphenyl)diazenyl]ethenyl}benzoate and methyl 4-{2,2-dichloro-1-[(*E*)-(3,4-dimethylphenyl)diazenyl]ethenyl}benzoate

Namiq Q. Shikhaliyev, Shafiga A. İbrahimova, Gulnar T. Atakishiyeva, Nigar E. Ahmedova, Gulnara V. Babayeva, Victor N. Khrustalev, Zeliha Atioğlu, Mehmet Akkurt and Ajaya Bhattarai

Computing details

Methyl 4-{2,2-dichloro-1-[(*E*)-phenyldiazenyl]ethenyl}benzoate (I)

Crystal data

$C_{16}H_{12}Cl_2N_2O_2$
$M_r = 335.18$
Monoclinic, $P2_1/c$
$a = 15.47572$ (16) Å
$b = 4.16896$ (4) Å
$c = 23.2257$ (2) Å
$\beta = 100.1964$ (9)°
$V = 1474.80$ (2) Å^3
$Z = 4$
$F(000) = 688$
$D_x = 1.510$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 12282 reflections
$\theta = 2.9$–$77.0°$
$\mu = 4.04$ mm^{-1}
$T = 100$ K
Prism, red
0.22 × 0.13 × 0.11 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2021)
$T_{min} = 0.404$, $T_{max} = 0.600$
23940 measured reflections
3151 independent reflections
2823 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 77.8°$, $\theta_{min} = 2.9°$
$h = -19 \rightarrow 19$
$k = -5 \rightarrow 5$
$l = -29 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.103$
$S = 1.04$
3151 reflections
200 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 1.3375P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ 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	0.13479 (3)	0.05540 (11)	0.40331 (2)	0.02247 (13)
Cl2	0.27874 (3)	0.32473 (12)	0.48326 (2)	0.02639 (14)
O1	0.01936 (9)	0.1723 (4)	0.10978 (6)	0.0285 (3)
O2	0.14455 (9)	−0.0786 (4)	0.10100 (6)	0.0268 (3)
N1	0.34295 (10)	0.5658 (4)	0.38299 (7)	0.0216 (3)
N2	0.38242 (10)	0.6368 (4)	0.34161 (7)	0.0211 (3)
C1	0.26399 (11)	0.3911 (4)	0.36605 (8)	0.0207 (4)
C2	0.22931 (12)	0.2761 (5)	0.41127 (8)	0.0214 (4)
C3	0.22169 (11)	0.3354 (4)	0.30397 (8)	0.0197 (4)
C4	0.26794 (11)	0.1764 (5)	0.26603 (8)	0.0210 (4)
H4	0.327649	0.119411	0.279023	0.025*
C5	0.22744 (12)	0.1012 (5)	0.20969 (8)	0.0220 (4)
H5	0.259087	−0.011130	0.184514	0.026*
C6	0.14025 (11)	0.1895 (4)	0.18962 (8)	0.0203 (4)
C7	0.09485 (11)	0.3596 (4)	0.22649 (8)	0.0215 (4)
H7	0.036219	0.427119	0.212652	0.026*
C8	0.13490 (12)	0.4308 (5)	0.28326 (8)	0.0220 (4)
H8	0.103337	0.544786	0.308278	0.026*
C9	0.09401 (12)	0.0996 (5)	0.13000 (8)	0.0215 (4)
C10	0.10264 (14)	−0.1883 (6)	0.04358 (8)	0.0293 (4)
H10A	0.087522	−0.003127	0.017776	0.044*
H10B	0.049121	−0.307201	0.046928	0.044*
H10C	0.142947	−0.328838	0.027228	0.044*
C11	0.46037 (11)	0.8199 (4)	0.36040 (8)	0.0209 (4)
C12	0.51605 (12)	0.8486 (5)	0.31977 (8)	0.0228 (4)
H12	0.500723	0.751252	0.282364	0.027*
C13	0.59408 (13)	1.0194 (5)	0.33381 (9)	0.0255 (4)
H13	0.632190	1.038116	0.306098	0.031*
C14	0.61616 (12)	1.1624 (5)	0.38829 (9)	0.0258 (4)
H14	0.670157	1.274595	0.398327	0.031*
C15	0.55900 (13)	1.1414 (5)	0.42850 (8)	0.0264 (4)
H15	0.573440	1.245282	0.465361	0.032*
C16	0.48136 (12)	0.9698 (5)	0.41483 (8)	0.0233 (4)
H16	0.442743	0.954405	0.442280	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0167 (2)	0.0264 (2)	0.0244 (2)	−0.00357 (16)	0.00406 (15)	−0.00043 (16)

Cl2	0.0217 (2)	0.0355 (3)	0.0213 (2)	−0.00499 (17)	0.00171 (16)	−0.00103 (17)
O1	0.0183 (6)	0.0359 (8)	0.0291 (7)	0.0048 (6)	−0.0016 (5)	−0.0043 (6)
O2	0.0193 (6)	0.0382 (8)	0.0220 (6)	0.0047 (6)	0.0016 (5)	−0.0046 (6)
N1	0.0162 (7)	0.0232 (8)	0.0248 (7)	0.0001 (6)	0.0022 (6)	0.0017 (6)
N2	0.0153 (7)	0.0216 (7)	0.0260 (8)	0.0007 (6)	0.0025 (6)	0.0023 (6)
C1	0.0152 (8)	0.0208 (8)	0.0255 (9)	0.0019 (7)	0.0018 (7)	−0.0006 (7)
C2	0.0163 (8)	0.0233 (9)	0.0241 (9)	0.0006 (7)	0.0023 (6)	−0.0014 (7)
C3	0.0163 (8)	0.0202 (8)	0.0221 (9)	−0.0004 (7)	0.0023 (6)	0.0026 (7)
C4	0.0142 (8)	0.0244 (9)	0.0241 (9)	0.0035 (7)	0.0029 (6)	0.0038 (7)
C5	0.0166 (8)	0.0260 (9)	0.0240 (9)	0.0034 (7)	0.0049 (7)	0.0022 (7)
C6	0.0159 (8)	0.0220 (9)	0.0224 (8)	−0.0002 (7)	0.0021 (6)	0.0025 (7)
C7	0.0140 (8)	0.0221 (9)	0.0281 (9)	0.0019 (7)	0.0028 (7)	0.0016 (7)
C8	0.0153 (8)	0.0248 (9)	0.0261 (9)	0.0004 (7)	0.0044 (7)	−0.0011 (7)
C9	0.0175 (8)	0.0233 (9)	0.0237 (9)	0.0013 (7)	0.0038 (7)	0.0019 (7)
C10	0.0264 (10)	0.0391 (11)	0.0214 (9)	0.0002 (8)	0.0018 (7)	−0.0053 (8)
C11	0.0140 (8)	0.0219 (9)	0.0259 (9)	0.0017 (7)	0.0011 (6)	0.0034 (7)
C12	0.0205 (9)	0.0238 (9)	0.0246 (9)	0.0002 (7)	0.0053 (7)	0.0005 (7)
C13	0.0195 (9)	0.0274 (10)	0.0306 (10)	0.0009 (8)	0.0077 (7)	0.0020 (8)
C14	0.0155 (8)	0.0291 (10)	0.0319 (10)	−0.0030 (7)	0.0014 (7)	0.0046 (8)
C15	0.0233 (9)	0.0305 (10)	0.0241 (9)	−0.0045 (8)	0.0004 (7)	0.0017 (8)
C16	0.0189 (9)	0.0282 (9)	0.0227 (9)	−0.0010 (7)	0.0037 (7)	0.0035 (7)

Geometric parameters (Å, º)

Cl1—C2	1.7102 (19)	C7—C8	1.386 (3)
Cl2—C2	1.7231 (18)	C7—H7	0.9500
O1—C9	1.206 (2)	C8—H8	0.9500
O2—C9	1.343 (2)	C10—H10A	0.9800
O2—C10	1.450 (2)	C10—H10B	0.9800
N1—N2	1.262 (2)	C10—H10C	0.9800
N1—C1	1.417 (2)	C11—C12	1.391 (3)
N2—C11	1.429 (2)	C11—C16	1.396 (3)
C1—C2	1.349 (3)	C12—C13	1.390 (3)
C1—C3	1.492 (2)	C12—H12	0.9500
C3—C4	1.397 (3)	C13—C14	1.386 (3)
C3—C8	1.402 (2)	C13—H13	0.9500
C4—C5	1.383 (3)	C14—C15	1.398 (3)
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.397 (2)	C15—C16	1.386 (3)
C5—H5	0.9500	C15—H15	0.9500
C6—C7	1.394 (3)	C16—H16	0.9500
C6—C9	1.490 (3)		

C9—O2—C10	115.49 (15)	O1—C9—O2	123.12 (17)
N2—N1—C1	114.77 (15)	O1—C9—C6	124.62 (17)
N1—N2—C11	112.90 (15)	O2—C9—C6	112.26 (15)
C2—C1—N1	114.12 (16)	O2—C10—H10A	109.5
C2—C1—C3	121.99 (16)	O2—C10—H10B	109.5

N1—C1—C3	123.89 (16)	H10A—C10—H10B	109.5
C1—C2—Cl1	123.88 (14)	O2—C10—H10C	109.5
C1—C2—Cl2	122.96 (14)	H10A—C10—H10C	109.5
Cl1—C2—Cl2	113.11 (11)	H10B—C10—H10C	109.5
C4—C3—C8	119.01 (16)	C12—C11—C16	120.20 (17)
C4—C3—C1	119.81 (16)	C12—C11—N2	115.44 (16)
C8—C3—C1	121.15 (16)	C16—C11—N2	124.36 (16)
C5—C4—C3	120.48 (16)	C13—C12—C11	120.10 (18)
C5—C4—H4	119.8	C13—C12—H12	119.9
C3—C4—H4	119.8	C11—C12—H12	119.9
C4—C5—C6	120.35 (17)	C14—C13—C12	119.88 (18)
C4—C5—H5	119.8	C14—C13—H13	120.1
C6—C5—H5	119.8	C12—C13—H13	120.1
C7—C6—C5	119.42 (17)	C13—C14—C15	120.01 (18)
C7—C6—C9	119.19 (16)	C13—C14—H14	120.0
C5—C6—C9	121.38 (17)	C15—C14—H14	120.0
C8—C7—C6	120.28 (16)	C16—C15—C14	120.30 (18)
C8—C7—H7	119.9	C16—C15—H15	119.8
C6—C7—H7	119.9	C14—C15—H15	119.8
C7—C8—C3	120.38 (17)	C15—C16—C11	119.46 (17)
C7—C8—H8	119.8	C15—C16—H16	120.3
C3—C8—H8	119.8	C11—C16—H16	120.3

C1—N1—N2—C11	178.44 (15)	C4—C3—C8—C7	1.9 (3)
N2—N1—C1—C2	170.71 (16)	C1—C3—C8—C7	−176.03 (17)
N2—N1—C1—C3	−9.2 (3)	C10—O2—C9—O1	−1.8 (3)
N1—C1—C2—Cl1	−179.62 (13)	C10—O2—C9—C6	177.22 (16)
C3—C1—C2—Cl1	0.3 (3)	C7—C6—C9—O1	2.5 (3)
N1—C1—C2—Cl2	−2.2 (2)	C5—C6—C9—O1	−178.85 (19)
C3—C1—C2—Cl2	177.66 (14)	C7—C6—C9—O2	−176.46 (17)
C2—C1—C3—C4	−120.9 (2)	C5—C6—C9—O2	2.2 (3)
N1—C1—C3—C4	59.0 (3)	N1—N2—C11—C12	167.20 (16)
C2—C1—C3—C8	57.0 (3)	N1—N2—C11—C16	−14.1 (3)
N1—C1—C3—C8	−123.1 (2)	C16—C11—C12—C13	1.8 (3)
C8—C3—C4—C5	−2.9 (3)	N2—C11—C12—C13	−179.43 (17)
C1—C3—C4—C5	175.04 (17)	C11—C12—C13—C14	−0.3 (3)
C3—C4—C5—C6	1.4 (3)	C12—C13—C14—C15	−1.6 (3)
C4—C5—C6—C7	1.2 (3)	C13—C14—C15—C16	2.0 (3)
C4—C5—C6—C9	−177.42 (17)	C14—C15—C16—C11	−0.5 (3)
C5—C6—C7—C8	−2.2 (3)	C12—C11—C16—C15	−1.4 (3)
C9—C6—C7—C8	176.44 (17)	N2—C11—C16—C15	179.91 (18)
C6—C7—C8—C3	0.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···N2^{i}	0.95	2.54	3.191 (3)	126

C5—H5···O2	0.95	2.40	2.726 (2)	100

Symmetry code: (i) *x*, *y*−1, *z*.

Methyl 4-{2,2-dichloro-1-[(*E*)-(4-methylphenyl)diazenyl]ethenyl}benzoate (II)

Crystal data

$C_{17}H_{14}Cl_2N_2O_2$
M_r = 349.20
Monoclinic, $P2_1/c$
a = 15.6177 (2) Å
b = 8.47502 (11) Å
c = 13.10365 (17) Å
β = 109.6555 (15)°
V = 1633.34 (4) Å^3
Z = 4
$F(000)$ = 720
D_x = 1.420 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 12932 reflections
θ = 3.0–77.0°
μ = 3.67 mm^{-1}
T = 100 K
Prism, red
0.26 × 0.19 × 0.17 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2021)
T_{min} = 0.307, T_{max} = 0.530
28857 measured reflections
3469 independent reflections
3197 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 78.0°, θ_{min} = 3.0°
h = −15→19
k = −10→10
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.108
S = 1.13
3469 reflections
211 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.7484P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Extinction correction: SHELXL-2018/3 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0012 (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_{iso}*/U_{eq}
Cl1	0.98647 (3)	0.77109 (5)	0.27683 (3)	0.02442 (14)
Cl2	0.94376 (3)	0.63249 (5)	0.06421 (3)	0.02432 (14)
O1	0.69644 (8)	0.59702 (16)	0.60175 (10)	0.0264 (3)
O2	0.81960 (8)	0.44134 (15)	0.66154 (9)	0.0245 (3)
N1	0.78451 (9)	0.49845 (18)	0.10125 (11)	0.0209 (3)
N2	0.71406 (9)	0.44374 (18)	0.11566 (11)	0.0219 (3)

C1	0.84094 (11)	0.5841 (2)	0.19136 (13)	0.0199 (3)
C2	0.91421 (11)	0.6533 (2)	0.17879 (13)	0.0210 (3)
C3	0.82373 (11)	0.5849 (2)	0.29668 (13)	0.0196 (3)
C4	0.74921 (11)	0.6634 (2)	0.30912 (13)	0.0216 (3)
H4	0.710519	0.725291	0.251576	0.026*
C5	0.73197 (11)	0.6504 (2)	0.40622 (14)	0.0220 (3)
H5	0.682177	0.705534	0.415595	0.026*
C6	0.78742 (11)	0.5568 (2)	0.48977 (13)	0.0195 (3)
C7	0.86273 (11)	0.4802 (2)	0.47817 (13)	0.0208 (3)
H7	0.901389	0.418074	0.535632	0.025*
C8	0.88054 (11)	0.4957 (2)	0.38166 (13)	0.0207 (3)
H8	0.932079	0.444724	0.373610	0.025*
C9	0.76178 (11)	0.5361 (2)	0.58862 (13)	0.0204 (3)
C10	0.79809 (13)	0.4119 (2)	0.75872 (14)	0.0275 (4)
H10A	0.839975	0.332619	0.802976	0.041*
H10B	0.735518	0.373025	0.739045	0.041*
H10C	0.804167	0.510044	0.800106	0.041*
C11	0.65821 (11)	0.3536 (2)	0.02710 (13)	0.0215 (3)
C12	0.57409 (12)	0.3086 (3)	0.03265 (15)	0.0296 (4)
H12	0.556200	0.343233	0.091445	0.036*
C13	0.51651 (13)	0.2138 (3)	−0.04704 (16)	0.0314 (4)
H13	0.458899	0.185192	−0.043069	0.038*
C14	0.54206 (12)	0.1597 (2)	−0.13325 (14)	0.0249 (4)
C15	0.62625 (12)	0.2063 (2)	−0.13829 (14)	0.0233 (4)
H15	0.644226	0.171215	−0.196914	0.028*
C16	0.68413 (11)	0.3026 (2)	−0.05973 (13)	0.0217 (3)
H16	0.741000	0.333796	−0.064785	0.026*
C17	0.48059 (13)	0.0527 (3)	−0.21805 (16)	0.0337 (4)
H17A	0.516980	−0.029287	−0.236871	0.050*
H17B	0.448259	0.114318	−0.282843	0.050*
H17C	0.436482	0.003205	−0.189705	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0228 (2)	0.0257 (2)	0.0259 (2)	−0.00466 (15)	0.00965 (16)	−0.00107 (15)
Cl2	0.0258 (2)	0.0280 (2)	0.0246 (2)	0.00138 (15)	0.01564 (16)	0.00240 (15)
O1	0.0245 (6)	0.0358 (7)	0.0220 (6)	0.0048 (5)	0.0119 (5)	−0.0007 (5)
O2	0.0266 (6)	0.0303 (7)	0.0204 (6)	0.0048 (5)	0.0131 (5)	0.0057 (5)
N1	0.0212 (6)	0.0230 (7)	0.0204 (6)	−0.0009 (5)	0.0094 (5)	0.0002 (5)
N2	0.0223 (7)	0.0248 (7)	0.0202 (7)	−0.0011 (6)	0.0093 (5)	−0.0005 (6)
C1	0.0209 (7)	0.0203 (8)	0.0200 (8)	0.0022 (6)	0.0087 (6)	0.0015 (6)
C2	0.0219 (8)	0.0212 (8)	0.0214 (8)	0.0017 (6)	0.0092 (6)	0.0008 (6)
C3	0.0206 (7)	0.0205 (8)	0.0197 (8)	−0.0039 (6)	0.0095 (6)	−0.0019 (6)
C4	0.0213 (8)	0.0228 (8)	0.0209 (8)	0.0013 (6)	0.0075 (6)	0.0026 (6)
C5	0.0209 (8)	0.0231 (8)	0.0246 (8)	0.0008 (6)	0.0112 (6)	−0.0010 (6)
C6	0.0206 (7)	0.0205 (8)	0.0188 (7)	−0.0028 (6)	0.0086 (6)	−0.0019 (6)
C7	0.0199 (7)	0.0228 (8)	0.0206 (7)	−0.0001 (6)	0.0079 (6)	−0.0009 (6)

C8	0.0200 (7)	0.0221 (8)	0.0221 (8)	−0.0009 (6)	0.0099 (6)	−0.0030 (6)
C9	0.0199 (7)	0.0223 (8)	0.0201 (7)	−0.0026 (6)	0.0081 (6)	−0.0032 (6)
C10	0.0323 (9)	0.0340 (10)	0.0210 (8)	0.0027 (8)	0.0153 (7)	0.0043 (7)
C11	0.0227 (8)	0.0230 (8)	0.0196 (8)	−0.0004 (6)	0.0081 (6)	0.0001 (6)
C12	0.0265 (9)	0.0407 (11)	0.0261 (9)	−0.0058 (8)	0.0146 (7)	−0.0065 (8)
C13	0.0238 (8)	0.0438 (12)	0.0302 (9)	−0.0093 (8)	0.0138 (7)	−0.0067 (8)
C14	0.0260 (8)	0.0256 (9)	0.0226 (8)	−0.0018 (7)	0.0076 (7)	−0.0007 (7)
C15	0.0273 (8)	0.0244 (9)	0.0206 (8)	0.0019 (7)	0.0112 (7)	0.0008 (7)
C16	0.0222 (8)	0.0236 (8)	0.0214 (8)	0.0002 (6)	0.0101 (6)	0.0015 (7)
C17	0.0319 (9)	0.0391 (11)	0.0300 (9)	−0.0093 (8)	0.0105 (8)	−0.0090 (8)

Geometric parameters (Å, º)

Cl1—C2	1.7159 (17)	C7—H7	0.9500
Cl2—C2	1.7217 (16)	C8—H8	0.9500
O1—C9	1.207 (2)	C10—H10A	0.9800
O2—C9	1.339 (2)	C10—H10B	0.9800
O2—C10	1.4438 (19)	C10—H10C	0.9800
N1—N2	1.265 (2)	C11—C12	1.393 (2)
N1—C1	1.414 (2)	C11—C16	1.398 (2)
N2—C11	1.418 (2)	C12—C13	1.383 (3)
C1—C2	1.345 (2)	C12—H12	0.9500
C1—C3	1.492 (2)	C13—C14	1.396 (3)
C3—C8	1.391 (2)	C13—H13	0.9500
C3—C4	1.397 (2)	C14—C15	1.396 (2)
C4—C5	1.391 (2)	C14—C17	1.504 (2)
C4—H4	0.9500	C15—C16	1.385 (2)
C5—C6	1.393 (2)	C15—H15	0.9500
C5—H5	0.9500	C16—H16	0.9500
C6—C7	1.396 (2)	C17—H17A	0.9800
C6—C9	1.489 (2)	C17—H17B	0.9800
C7—C8	1.389 (2)	C17—H17C	0.9800
C9—O2—C10	115.59 (13)	O2—C10—H10A	109.5
N2—N1—C1	113.13 (13)	O2—C10—H10B	109.5
N1—N2—C11	113.70 (13)	H10A—C10—H10B	109.5
C2—C1—N1	116.03 (14)	O2—C10—H10C	109.5
C2—C1—C3	122.48 (15)	H10A—C10—H10C	109.5
N1—C1—C3	121.28 (14)	H10B—C10—H10C	109.5
C1—C2—Cl1	122.31 (13)	C12—C11—C16	119.63 (16)
C1—C2—Cl2	123.43 (13)	C12—C11—N2	115.85 (15)
Cl1—C2—Cl2	114.26 (9)	C16—C11—N2	124.45 (15)
C8—C3—C4	119.85 (15)	C13—C12—C11	120.30 (16)
C8—C3—C1	118.23 (14)	C13—C12—H12	119.8
C4—C3—C1	121.77 (15)	C11—C12—H12	119.8
C5—C4—C3	119.63 (15)	C12—C13—C14	120.76 (16)
C5—C4—H4	120.2	C12—C13—H13	119.6
C3—C4—H4	120.2	C14—C13—H13	119.6

C4—C5—C6	120.25 (15)	C15—C14—C13	118.40 (16)
C4—C5—H5	119.9	C15—C14—C17	120.88 (16)
C6—C5—H5	119.9	C13—C14—C17	120.72 (16)
C5—C6—C7	120.22 (14)	C16—C15—C14	121.44 (15)
C5—C6—C9	118.22 (14)	C16—C15—H15	119.3
C7—C6—C9	121.52 (15)	C14—C15—H15	119.3
C8—C7—C6	119.28 (15)	C15—C16—C11	119.45 (15)
C8—C7—H7	120.4	C15—C16—H16	120.3
C6—C7—H7	120.4	C11—C16—H16	120.3
C7—C8—C3	120.73 (15)	C14—C17—H17A	109.5
C7—C8—H8	119.6	C14—C17—H17B	109.5
C3—C8—H8	119.6	H17A—C17—H17B	109.5
O1—C9—O2	123.68 (15)	C14—C17—H17C	109.5
O1—C9—C6	124.15 (15)	H17A—C17—H17C	109.5
O2—C9—C6	112.17 (13)	H17B—C17—H17C	109.5

C1—N1—N2—C11	−178.24 (14)	C1—C3—C8—C7	−173.98 (15)
N2—N1—C1—C2	−176.18 (15)	C10—O2—C9—O1	1.4 (2)
N2—N1—C1—C3	8.9 (2)	C10—O2—C9—C6	−178.45 (14)
N1—C1—C2—Cl1	176.23 (12)	C5—C6—C9—O1	−1.0 (3)
C3—C1—C2—Cl1	−8.9 (2)	C7—C6—C9—O1	−178.47 (17)
N1—C1—C2—Cl2	−3.7 (2)	C5—C6—C9—O2	178.82 (14)
C3—C1—C2—Cl2	171.16 (13)	C7—C6—C9—O2	1.4 (2)
C2—C1—C3—C8	−69.6 (2)	N1—N2—C11—C12	−171.71 (16)
N1—C1—C3—C8	104.94 (19)	N1—N2—C11—C16	11.5 (2)
C2—C1—C3—C4	114.81 (19)	C16—C11—C12—C13	0.1 (3)
N1—C1—C3—C4	−70.6 (2)	N2—C11—C12—C13	−176.77 (18)
C8—C3—C4—C5	−0.6 (2)	C11—C12—C13—C14	1.0 (3)
C1—C3—C4—C5	174.91 (16)	C12—C13—C14—C15	−1.3 (3)
C3—C4—C5—C6	−1.5 (3)	C12—C13—C14—C17	178.15 (19)
C4—C5—C6—C7	2.4 (3)	C13—C14—C15—C16	0.6 (3)
C4—C5—C6—C9	−175.06 (15)	C17—C14—C15—C16	−178.87 (17)
C5—C6—C7—C8	−1.3 (2)	C14—C15—C16—C11	0.5 (3)
C9—C6—C7—C8	176.06 (15)	C12—C11—C16—C15	−0.9 (3)
C6—C7—C8—C3	−0.7 (3)	N2—C11—C16—C15	175.78 (16)
C4—C3—C8—C7	1.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1^{i}	0.95	2.43	3.268 (2)	148
C13—H13···O1^{ii}	0.95	2.40	3.309 (3)	159

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

Methyl 4-{2,2-dichloro-1-[(*E*)-(3,4-dimethylphenyl)diazenyl]ethenyl}benzoate (III)

Crystal data

$C_{18}H_{16}Cl_2N_2O_2$
$M_r = 363.23$
Triclinic, $P\overline{1}$
$a = 8.22057$ (10) Å
$b = 8.53211$ (9) Å
$c = 13.08729$ (14) Å
$\alpha = 103.9484$ (9)°
$\beta = 101.9047$ (10)°
$\gamma = 98.0600$ (9)°
$V = 854.09$ (2) Å^3
$Z = 2$
$F(000) = 376$
$D_x = 1.412$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 14210 reflections
$\theta = 3.6$–$77.0°$
$\mu = 3.53$ mm^{-1}
$T = 100$ K
Prism, red
0.20 × 0.15 × 0.09 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2021)
$T_{min} = 0.349$, $T_{max} = 0.700$
29808 measured reflections
3632 independent reflections
3340 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 77.8°$, $\theta_{min} = 3.6°$
$h = -10 \rightarrow 10$
$k = -8 \rightarrow 10$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.097$
$S = 1.08$
3632 reflections
220 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.2636P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ 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_{iso}*/U_{eq}
Cl1	0.07283 (5)	−0.14861 (4)	0.77715 (3)	0.02444 (11)
Cl2	0.13915 (5)	−0.31047 (4)	0.57534 (3)	0.02637 (12)
O1	0.30977 (14)	0.68544 (14)	1.04185 (9)	0.0315 (3)
O2	0.04043 (13)	0.67433 (12)	0.95696 (8)	0.0208 (2)
N1	0.21612 (15)	0.01951 (15)	0.54534 (10)	0.0198 (2)
N2	0.26094 (15)	0.16265 (15)	0.53648 (10)	0.0208 (3)
C1	0.17177 (17)	0.02110 (17)	0.64440 (11)	0.0189 (3)
C2	0.13347 (18)	−0.12681 (18)	0.66254 (11)	0.0206 (3)
C3	0.17331 (18)	0.17742 (17)	0.72590 (11)	0.0185 (3)
C4	0.05481 (18)	0.27494 (17)	0.70373 (11)	0.0203 (3)

H4	−0.026457	0.242988	0.635554	0.024*
C5	0.05592 (18)	0.41897 (17)	0.78162 (12)	0.0203 (3)
H5	−0.026072	0.484181	0.767260	0.024*
C6	0.17767 (17)	0.46736 (17)	0.88079 (11)	0.0188 (3)
C7	0.29835 (18)	0.37242 (17)	0.90121 (11)	0.0202 (3)
H7	0.382930	0.406872	0.968032	0.024*
C8	0.29570 (18)	0.22742 (17)	0.82428 (12)	0.0202 (3)
H8	0.377651	0.162276	0.838900	0.024*
C9	0.18624 (18)	0.61892 (17)	0.96841 (12)	0.0209 (3)
C10	0.0464 (2)	0.82424 (18)	1.03931 (12)	0.0244 (3)
H10A	−0.063270	0.858160	1.025427	0.037*
H10B	0.135720	0.911518	1.037093	0.037*
H10C	0.070772	0.804528	1.111150	0.037*
C11	0.31099 (18)	0.16259 (18)	0.43840 (11)	0.0207 (3)
C12	0.33518 (19)	0.31514 (18)	0.41637 (12)	0.0232 (3)
H12	0.318547	0.409355	0.466000	0.028*
C13	0.38330 (18)	0.3316 (2)	0.32285 (13)	0.0249 (3)
C14	0.40929 (18)	0.1914 (2)	0.25099 (12)	0.0258 (3)
C15	0.38626 (19)	0.0399 (2)	0.27461 (12)	0.0260 (3)
H15	0.403729	−0.054527	0.225581	0.031*
C16	0.33864 (19)	0.02371 (19)	0.36759 (12)	0.0231 (3)
H16	0.325038	−0.079976	0.382923	0.028*
C17	0.4088 (2)	0.4974 (2)	0.30023 (15)	0.0333 (4)
H17A	0.376239	0.578206	0.355458	0.050*
H17B	0.338263	0.488834	0.228149	0.050*
H17C	0.528407	0.532926	0.302386	0.050*
C18	0.4627 (2)	0.2029 (3)	0.14898 (14)	0.0363 (4)
H18A	0.377011	0.242730	0.103353	0.055*
H18B	0.473909	0.093688	0.108817	0.055*
H18C	0.571996	0.279612	0.168542	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0346 (2)	0.02094 (19)	0.02065 (18)	0.00298 (14)	0.01118 (14)	0.00900 (14)
Cl2	0.0402 (2)	0.01643 (18)	0.02316 (19)	0.00645 (14)	0.01064 (15)	0.00402 (13)
O1	0.0278 (6)	0.0277 (6)	0.0306 (6)	0.0074 (5)	0.0038 (5)	−0.0053 (5)
O2	0.0259 (5)	0.0182 (5)	0.0211 (5)	0.0070 (4)	0.0100 (4)	0.0059 (4)
N1	0.0231 (6)	0.0194 (6)	0.0185 (6)	0.0034 (5)	0.0077 (5)	0.0066 (5)
N2	0.0240 (6)	0.0199 (6)	0.0198 (6)	0.0025 (5)	0.0092 (5)	0.0060 (5)
C1	0.0214 (6)	0.0188 (7)	0.0171 (6)	0.0034 (5)	0.0071 (5)	0.0044 (5)
C2	0.0251 (7)	0.0191 (7)	0.0180 (6)	0.0041 (5)	0.0070 (5)	0.0049 (5)
C3	0.0234 (7)	0.0164 (6)	0.0187 (6)	0.0017 (5)	0.0108 (5)	0.0069 (5)
C4	0.0247 (7)	0.0203 (7)	0.0174 (6)	0.0040 (5)	0.0071 (5)	0.0070 (5)
C5	0.0242 (7)	0.0190 (7)	0.0216 (7)	0.0063 (5)	0.0097 (6)	0.0088 (5)
C6	0.0234 (7)	0.0170 (6)	0.0189 (7)	0.0029 (5)	0.0103 (5)	0.0067 (5)
C7	0.0216 (7)	0.0204 (7)	0.0186 (6)	0.0020 (5)	0.0063 (5)	0.0056 (5)
C8	0.0227 (7)	0.0191 (7)	0.0218 (7)	0.0060 (5)	0.0093 (5)	0.0070 (5)

C9	0.0248 (7)	0.0194 (7)	0.0219 (7)	0.0050 (5)	0.0105 (6)	0.0079 (6)
C10	0.0337 (8)	0.0179 (7)	0.0262 (7)	0.0080 (6)	0.0152 (6)	0.0066 (6)
C11	0.0212 (7)	0.0231 (7)	0.0190 (7)	0.0025 (5)	0.0085 (5)	0.0063 (6)
C12	0.0247 (7)	0.0226 (7)	0.0230 (7)	0.0022 (6)	0.0084 (6)	0.0069 (6)
C13	0.0214 (7)	0.0299 (8)	0.0252 (7)	0.0006 (6)	0.0064 (6)	0.0129 (6)
C14	0.0206 (7)	0.0376 (9)	0.0214 (7)	0.0024 (6)	0.0085 (6)	0.0114 (6)
C15	0.0245 (7)	0.0313 (8)	0.0235 (7)	0.0052 (6)	0.0119 (6)	0.0049 (6)
C16	0.0247 (7)	0.0226 (7)	0.0244 (7)	0.0042 (6)	0.0108 (6)	0.0072 (6)
C17	0.0366 (9)	0.0343 (9)	0.0335 (9)	0.0011 (7)	0.0109 (7)	0.0192 (7)
C18	0.0376 (9)	0.0506 (11)	0.0260 (8)	0.0047 (8)	0.0162 (7)	0.0157 (8)

Geometric parameters (Å, º)

Cl1—C2	1.7176 (14)	C10—H10A	0.9800
Cl2—C2	1.7148 (14)	C10—H10B	0.9800
O1—C9	1.2041 (19)	C10—H10C	0.9800
O2—C9	1.3422 (17)	C11—C16	1.396 (2)
O2—C10	1.4478 (17)	C11—C12	1.396 (2)
N1—N2	1.2651 (17)	C12—C13	1.393 (2)
N1—C1	1.4148 (17)	C12—H12	0.9500
N2—C11	1.4263 (17)	C13—C14	1.404 (2)
C1—C2	1.346 (2)	C13—C17	1.509 (2)
C1—C3	1.4901 (19)	C14—C15	1.397 (2)
C3—C8	1.390 (2)	C14—C18	1.509 (2)
C3—C4	1.397 (2)	C15—C16	1.384 (2)
C4—C5	1.392 (2)	C15—H15	0.9500
C4—H4	0.9500	C16—H16	0.9500
C5—C6	1.395 (2)	C17—H17A	0.9800
C5—H5	0.9500	C17—H17B	0.9800
C6—C7	1.390 (2)	C17—H17C	0.9800
C6—C9	1.4894 (19)	C18—H18A	0.9800
C7—C8	1.388 (2)	C18—H18B	0.9800
C7—H7	0.9500	C18—H18C	0.9800
C8—H8	0.9500		

C9—O2—C10	113.91 (11)	O2—C10—H10C	109.5
N2—N1—C1	112.85 (11)	H10A—C10—H10C	109.5
N1—N2—C11	113.33 (12)	H10B—C10—H10C	109.5
C2—C1—N1	116.08 (12)	C16—C11—C12	120.15 (13)
C2—C1—C3	121.77 (12)	C16—C11—N2	124.38 (13)
N1—C1—C3	122.12 (12)	C12—C11—N2	115.46 (13)
C1—C2—Cl2	124.19 (11)	C13—C12—C11	121.13 (14)
C1—C2—Cl1	122.44 (11)	C13—C12—H12	119.4
Cl2—C2—Cl1	113.37 (8)	C11—C12—H12	119.4
C8—C3—C4	119.86 (13)	C12—C13—C14	118.70 (14)
C8—C3—C1	119.43 (13)	C12—C13—C17	120.45 (15)
C4—C3—C1	120.70 (13)	C14—C13—C17	120.84 (14)
C5—C4—C3	119.96 (13)	C15—C14—C13	119.55 (13)

C5—C4—H4	120.0	C15—C14—C18	119.66 (15)
C3—C4—H4	120.0	C13—C14—C18	120.80 (15)
C4—C5—C6	119.88 (13)	C16—C15—C14	121.71 (14)
C4—C5—H5	120.1	C16—C15—H15	119.1
C6—C5—H5	120.1	C14—C15—H15	119.1
C7—C6—C5	119.93 (13)	C15—C16—C11	118.75 (14)
C7—C6—C9	117.03 (13)	C15—C16—H16	120.6
C5—C6—C9	123.04 (13)	C11—C16—H16	120.6
C8—C7—C6	120.22 (13)	C13—C17—H17A	109.5
C8—C7—H7	119.9	C13—C17—H17B	109.5
C6—C7—H7	119.9	H17A—C17—H17B	109.5
C7—C8—C3	120.10 (13)	C13—C17—H17C	109.5
C7—C8—H8	119.9	H17A—C17—H17C	109.5
C3—C8—H8	119.9	H17B—C17—H17C	109.5
O1—C9—O2	123.20 (13)	C14—C18—H18A	109.5
O1—C9—C6	124.17 (13)	C14—C18—H18B	109.5
O2—C9—C6	112.64 (12)	H18A—C18—H18B	109.5
O2—C10—H10A	109.5	C14—C18—H18C	109.5
O2—C10—H10B	109.5	H18A—C18—H18C	109.5
H10A—C10—H10B	109.5	H18B—C18—H18C	109.5

C1—N1—N2—C11	178.17 (11)	C10—O2—C9—O1	1.63 (19)
N2—N1—C1—C2	−175.79 (13)	C10—O2—C9—C6	−178.38 (11)
N2—N1—C1—C3	2.06 (19)	C7—C6—C9—O1	19.0 (2)
N1—C1—C2—Cl2	1.1 (2)	C5—C6—C9—O1	−161.01 (14)
C3—C1—C2—Cl2	−176.74 (11)	C7—C6—C9—O2	−161.04 (12)
N1—C1—C2—Cl1	−178.69 (10)	C5—C6—C9—O2	19.00 (18)
C3—C1—C2—Cl1	3.5 (2)	N1—N2—C11—C16	−11.1 (2)
C2—C1—C3—C8	67.37 (19)	N1—N2—C11—C12	170.08 (13)
N1—C1—C3—C8	−110.35 (15)	C16—C11—C12—C13	1.5 (2)
C2—C1—C3—C4	−113.54 (16)	N2—C11—C12—C13	−179.65 (13)
N1—C1—C3—C4	68.73 (18)	C11—C12—C13—C14	−0.7 (2)
C8—C3—C4—C5	−2.2 (2)	C11—C12—C13—C17	179.96 (14)
C1—C3—C4—C5	178.73 (12)	C12—C13—C14—C15	0.1 (2)
C3—C4—C5—C6	1.3 (2)	C17—C13—C14—C15	179.40 (14)
C4—C5—C6—C7	0.5 (2)	C12—C13—C14—C18	−179.55 (14)
C4—C5—C6—C9	−179.51 (12)	C17—C13—C14—C18	−0.2 (2)
C5—C6—C7—C8	−1.6 (2)	C13—C14—C15—C16	−0.2 (2)
C9—C6—C7—C8	178.48 (12)	C18—C14—C15—C16	179.48 (14)
C6—C7—C8—C3	0.7 (2)	C14—C15—C16—C11	0.9 (2)
C4—C3—C8—C7	1.2 (2)	C12—C11—C16—C15	−1.5 (2)
C1—C3—C8—C7	−179.74 (12)	N2—C11—C16—C15	179.72 (13)