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Crystal structure and Hirshfeld surface analysis of (*E*)-1-[2,2-dichloro-1-(4-nitrophenyl)ethenyl]-2-(4-fluorophenyl)diazene

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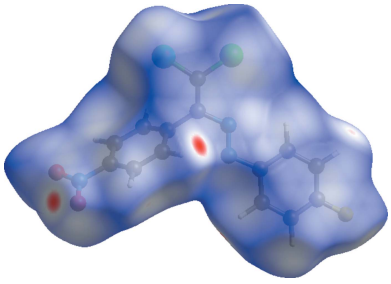
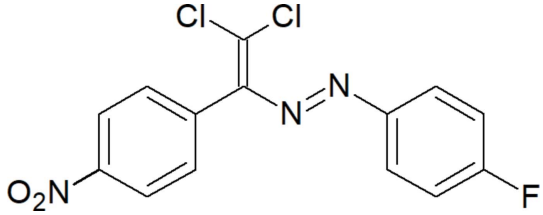
In the title compound, $C_{14}H_8Cl_2FN_3O_2$, the 4-fluorophenyl ring and the nitro-substituted benzene ring form a dihedral angle of 63.29 (8)°. In the crystal, molecules are linked by C—H···O hydrogen bonds into chains running parallel to the *c* axis. The crystal packing is further stabilized by C—Cl···π, C—F···π and N—O···π interactions. The Hirshfeld surface analysis of the crystal structure indicates that the most important contributions to the crystal packing are from H···O/O···H (15.5%), H···H (15.3%), Cl···H/H···Cl (13.8%), C···H/H···C (9.5%) and F···H/H···F (8.2%) interactions.

1. Chemical context

Non-covalent interactions, such as hydrogen, aerogen, halogen, chalcogen, pnicogen, tetrel and icosagen bonds, as well as *n*–π*, π–π stacking, π–cation, π–anion and hydrophobic interactions, can control or organize the conformation, aggregation, tertiary and quaternary structures of the molecule, its stabilization and particular properties (Akbari Afkhami *et al.*, 2017; Desiraju, 1995; Gurbanov *et al.*, 2018; Hazra *et al.*, 2018; Jlassi *et al.*, 2014; Kvyatkovskaya *et al.*, 2017; Legon, 2017, Maharramov *et al.*, 2009, 2018; Mahmoudi *et al.*, 2018*a*,*b*,*c*; Mahmudov *et al.*, 2014, 2017; Mahmudov & Pombeiro, 2016; Scheiner 2013; Shikhaliyev *et al.*, 2013, 2018). On the other hand, azo dyes and related hydrazone ligands and their complexes have attracted attention over the past decades because of their potential biological, pharmacological and analytical applications (Borisova *et al.*, 2018; Gadzhieva *et al.*, 2006; Gurbanov *et al.*, 2017; Shetnev & Zubkov, 2017). Herein we report the structure and non-covalent interactions of the title compound.





2. Structural commentary

The molecular conformation of the title compound (Fig. 1) is not planar, the 4-fluorophenyl ring and the nitro-substituted benzene ring forming a dihedral angle of 63.29 (8)°. The C2—C1—N1—N2, C1—N1—N2—C7, N1—N2—C7—C8, N2—C7—C8—Cl1, N2—C7—C8—Cl2, Cl1—C8—C7—C9 and C8—C7—C9—C14 torsion angles are −1.1 (2), 178.86 (13), 174.62 (14), −176.19 (11), 2.9 (2), 5.1 (2) and 63.4 (2)°, respectively. Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to those observed in related structures, *viz:* (2*E*)-1-(2-hydroxy-5-methylphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (Fun *et al.*, 2011*a*), (2*E*)-3-(3-benzyloxyphenyl)-1-(2-hydroxy-5-methylphenyl)prop-2-en-1-one (Fun *et al.*, 2011*b*), (2*E*)-3-[3-(benzyloxy)phenyl]-1-(2-hydroxyphenyl)prop-2-en-1-one (Fun *et al.*, 2011*c*), (2*E*)-1-(2,5-dimethoxyphenyl)-3-(3-nitrophenyl)prop-2-en-1-one (Fun *et al.*, 2011*d*) and (2*E*)-3-(3-nitrophenyl)-1-[4-(piperidin-1-yl)phenyl]prop-2-en-1-one (Fun *et al.*, 2012).

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, molecules are linked by C—H···O hydrogen bonds into chains parallel to the *c* axis (Table 1; Fig. 2). The crystal packing is further stabilized by weak C—Cl···π [Cl···*Cg*2(x, $\frac{5}{2}-y$, $\frac{1}{2}+z$) = 3.6792 (8) Å], C—F···π [F···*Cg*1($1-x$, $2-y$, $2-z$) = 3.5408 (16) Å] and N—O···π interactions [O···*Cg*1(x, $\frac{3}{2}-y$, $-\frac{1}{2}+z$) = 3.9815 (16) Å] where *Cg*1 and *Cg*2 are the centroids of the C1–C6 and C9–C14 rings, respectively.

Hirshfeld surfaces and fingerprint plots were generated for the title compound using *CrystalExplorer* (McKinnon *et al.*, 2007) to quantify and visualize the intermolecular interactions and to explain the observed crystal packing. The Hirshfeld surface mapped over d_{norm} using a standard surface resolution with a fixed colour scale of −0.1603 (red) to 1.2420 (blue) a.u. is shown in Fig. 3. The dark-red spots on the d_{norm} surface arise as a result of short interatomic contacts (Table 2), while the other weaker intermolecular interactions appear as light-red spots. The red points, which represent closer contacts and negative d_{norm} values on the surface, correspond to the C—H···O interactions.

The percentage contributions of various contacts to the total Hirshfeld surface are shown in the two-dimensional fingerprint plots in Fig. 4. The reciprocal O···H/H···O interactions appear as two symmetrical broad wings with $d_e + d_i \simeq$

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···O1^{i}	0.93	2.52	3.369 (2)	152

Symmetry code: (i) x, $-y+\frac{5}{2}$, $z+\frac{1}{2}$.

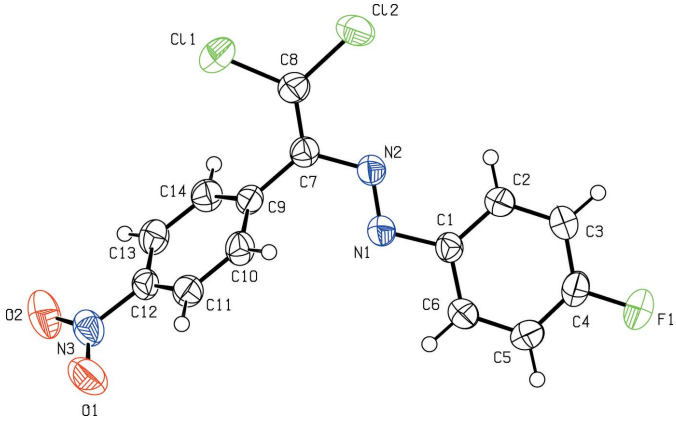


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

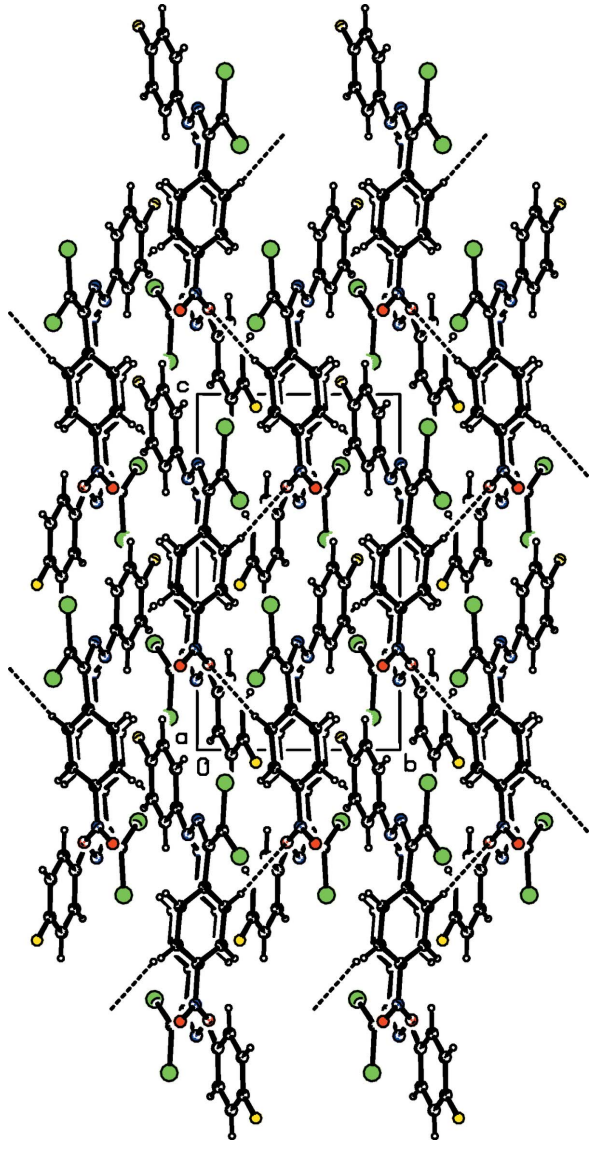


Figure 2
Crystal packing of the title compound, viewed down the *a* axis, showing the formation of chains parallel to the *c* axis through C—H···O hydrogen bonds (dashed lines).

Table 2
Summary of short interatomic contacts (Å) in the title compound.

Contact	Distance	Symmetry operation
(C8) Cl1···C8 (Cl1)	3.6040 (16)	$2-x, \frac{1}{2}+y, \frac{3}{2}-z$
(C13) H13···Cl1 (C8)	3.08	$2-x, 2-y, 1-z$
(C8) Cl2···Cl2 (C8)	3.6506 (7)	$2-x, 2-y, 2-z$
(C10) H10···O1 (N3)	2.52	$x, \frac{5}{2}-y, \frac{1}{2}+z$
(C4) F1···H11 (C11)	2.60	$1-x, -\frac{1}{2}+y, \frac{3}{2}-z$
(C4) F1···H6 (C6)	2.56	$x, \frac{3}{2}-y, \frac{1}{2}+z$
(N3) O1···H3 (C3)	2.67	$x, y, -1+z$
(C5) H5···O1 (N3)	2.74	$1-x, 2-y, 1-z$
(N3) O1···H10 (C10)	2.52	$x, \frac{5}{2}-y, -\frac{1}{2}+z$
(F1) C4···C4 (F1)	3.541 (3)	$1-x, 2-y, 2-z$

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

Contact	Percentage contribution
O···H/H···O	15.5
H···H	15.3
Cl···H/H···Cl	13.8
C···H/H···C	9.5
F···H/H···F	8.2
Cl···Cl	6.4
N···H/H···N	5.6
Cl···C/C···Cl	5.5
C···C	4.1
O···C/C···O	3.7
Cl···O/O···Cl	3.1
F···C/C···F	3.1
N···C/C···N	2.2
O···N/N···O	2.1
F···F	0.9
N···N	0.8

2.2 Å and contribute 15.5% to the Hirshfeld surface (Fig. 5*b*). The reciprocal Cl···H/H···Cl, C···H/H···C and F···H/H···F interactions (13.8, 9.5 and 8.2% contributions, respectively) are present as sharp symmetrical spikes at diagonal axes $d_e + d_i \simeq 2.9$, 3.0 and 2.4 Å, respectively (Fig. 5*d*–*f*). The small percentage contributions to the Hirshfeld surfaces from the various other interatomic contacts are listed in Table 3.

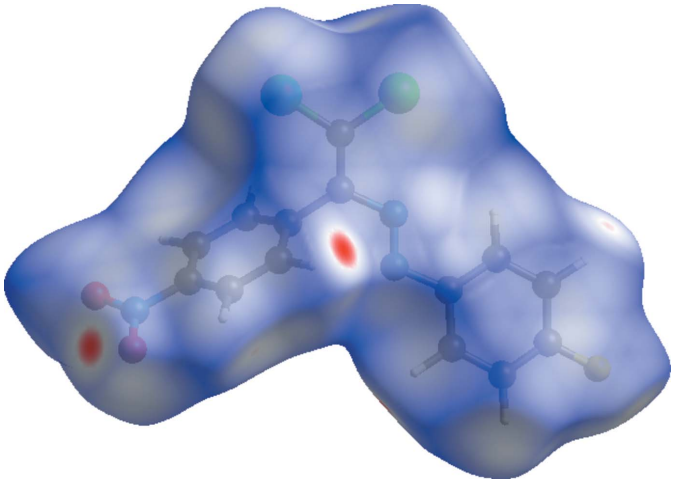


Figure 3
View of the three-dimensional Hirshfeld surface of the title compound plotted over d_{norm} in the range −0.1603 to 1.2420 a.u.

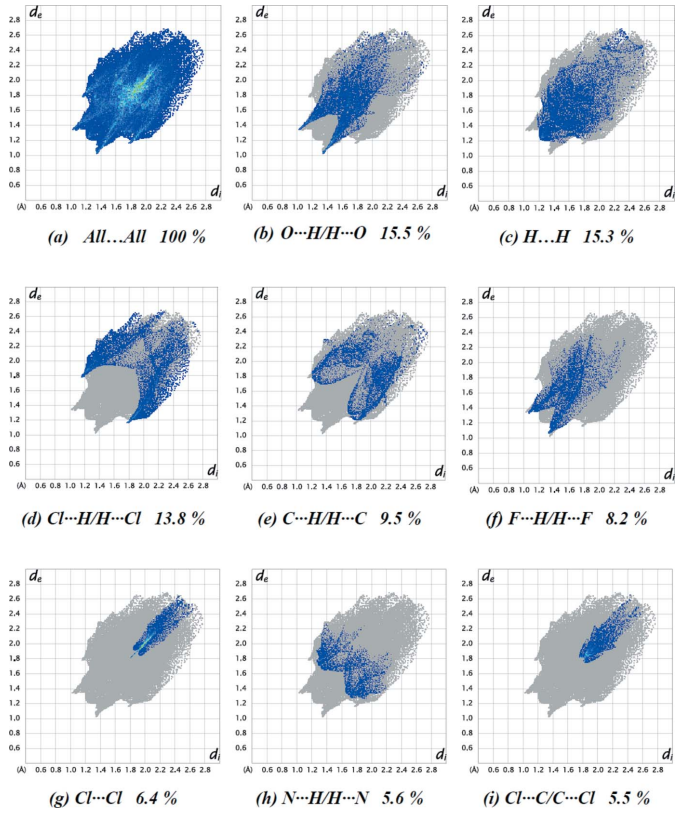


Figure 4
The full two-dimensional fingerprint plots for the title compound, showing (*a*) all interactions, and delineated into (*b*) O···H/H···O, (*c*) H···H, (*d*) Cl···H/H···Cl, (*e*) C···H/H···C, (*f*) F···H/H···F, (*g*) Cl···Cl, (*h*) N···H/H···N and (*i*) Cl···C/C···Cl interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

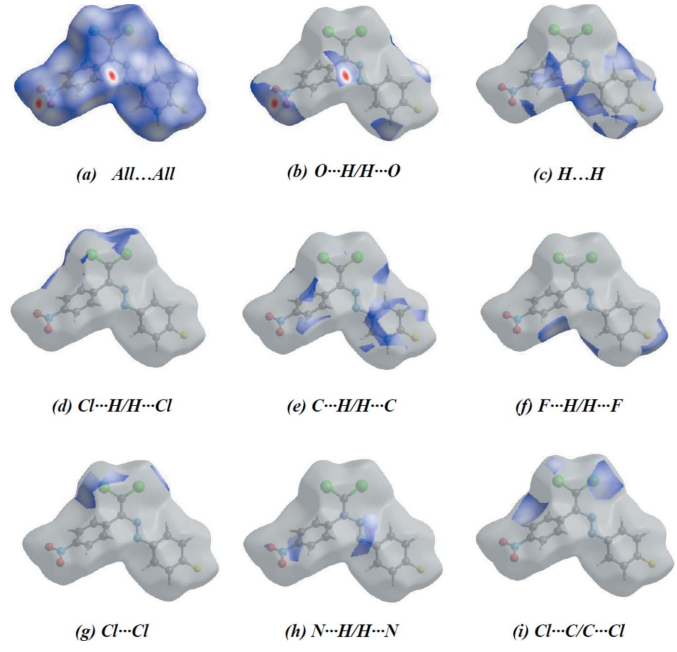


Figure 5
Hirshfeld surface representations with the function d_{norm} plotted onto the surface for (*a*) all interactions, (*b*) O···H/H···O, (*c*) H···H, (*d*) Cl···H/H···Cl, (*e*) C···H/H···C, (*f*) F···H/H···F, (*g*) Cl···Cl, (*h*) N···H/H···N and (*i*) Cl···C/C···Cl interactions.

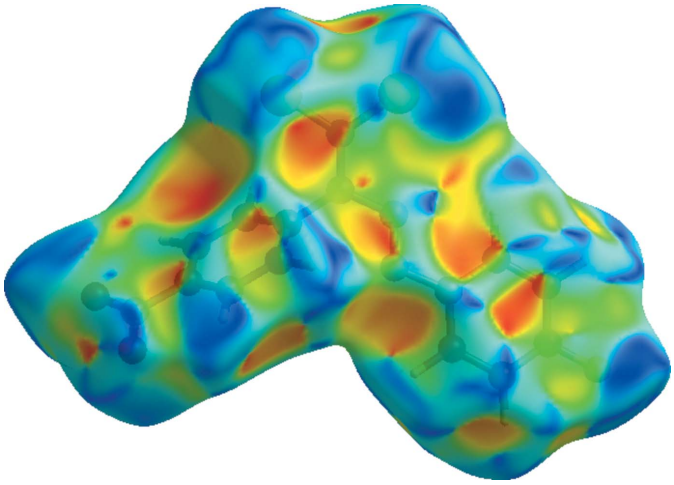


Figure 6
Hirshfeld surface of the title compound plotted over shape-index.

Hirshfeld surface representations with the function d_{norm} plotted onto the surface for all interactions are shown in Fig. 5. The large number of O···H/H···O, H···H, Cl···H/H···Cl, C···H/H···C, F···H/H···F, Cl···Cl, N···H/H···N and Cl···C/C···Cl interactions suggest that van der Waals interactions and hydrogen bonding play a major role in the crystal packing (Hathwar *et al.*, 2015). The shape-index of the Hirshfeld surface is a tool for visualizing the π–π stacking by the presence of adjacent red and blue triangles; if there are no such triangles, then there are no π–π interactions. The plot of the Hirshfeld surface mapped over shape-index shown in Fig. 6 clearly suggests that there are no π–π interactions in the title compound.

4. Synthesis and crystallization

The title compound was synthesized according to the method reported by Shikhaliyev *et al.* (2018). A 20 mL screw-neck vial was charged with DMSO (10 mL), (*E*)-1-(4-fluorophenyl)-2-(4-nitrobenzylidene)hydrazine (259 mg, 1 mmol), tetramethylethylenediamine (TMEDA; 295 mg, 2.5 mmol), CuCl (2 mg, 0.02 mmol) and CCl_4 (20 mmol, 10 equiv). 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* by rotary evaporator. The residue was purified by column chromatography on silica gel using appropriate mixtures of hexane and dichloromethane (3:1-1:1 *v*/*v*). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution. Yield (62%); m.p. 421 K. Analysis calculated for $C_{14}H_8Cl_2FN_3O_2$ (M = 340.14): C, 49.44; H, 2.37; N, 12.35; found: C, 49.38; H, 2.40; N, 12.24%. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32–8.29 (*d*, 2H, J = 9.21Hz), 7.81–7.77 (*m* 2H), 7.40–7.37 (*d*, 2H, J = 9.02Hz), 7.17–7.12 (*t*, 2H, J = 9.22Hz).^{13}C NMR (75 MHz, $CDCl_3$) δ 166.69, 163.32, 150.43, 149.17, 147.95, 139.40, 131.26, 125.51, 125.39, 123.41, 116.42, 116.11. ESI–MS: *m*/*z*: 341.06 $[M + H]^+$.

Table 4
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_8Cl_2FN_3O_2$
M_r	340.13
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	15.8644 (5), 7.2242 (2), 12.7595 (4)
β (°)	97.038 (2)
V (Å^3)	1451.32 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.47
Crystal size (mm)	0.34 × 0.23 × 0.14
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2003)
T_{min}, T_{max}	0.861, 0.925
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11383, 2851, 2359
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.089, 1.05
No. of reflections	2851
No. of parameters	199
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.21

Computer programs: *APEX3* and *SAINT* (Bruker, 2007), *SHELXT2016* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. C-bound H atoms were constrained to an ideal geometry with C—H = 0.93 Å and refined as riding with U_{iso}(H) = 1.2U_{eq}(C). Three outliers (100, 110, 200) were omitted in the last cycles of refinement.

Funding information

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References

Akbari Afkhami, F., Mahmoudi, G., Gurbanov, A. V., Zubkov, F. I., Qu, F., Gupta, A. & Safin, D. A. (2017). *Dalton Trans.* **46**, 14888–14896.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Borisova, K. K., Nikitina, E. V., Novikov, R. A., Khrustalev, V. N., Dorovatovskii, P. V., Zubavichus, Y. V., Kuznetsov, M. L., Zaytsev, V. P., Varlamov, A. V. & Zubkov, F. I. (2018). *Chem. Commun.* **54**, 2850–2853.
Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Desiraju, G. R. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 2311–2327.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Fun, H.-K., Arshad, S., Sarojini, B. K., Khaleel, V. M. & Narayana, B. (2011*a*). *Acta Cryst.* E**67**, o1248–o1249.
Fun, H.-K., Arshad, S., Sarojini, B. K., Khaleel, V. M. & Narayana, B. (2011*b*). *Acta Cryst.* E**67**, o1372–o1373.
Fun, H.-K., Chia, T. S., Narayana, B., Nayak, P. S. & Sarojini, B. K. (2011*d*). *Acta Cryst.* E**67**, o3058–o3059.
Fun, H.-K., Chia, T. S., Nayak, P. S., Narayana, B. & Sarojini, B. K. (2012). *Acta Cryst.* E**68**, o974.
Fun, H.-K., Loh, W.-S., Sarojini, B. K., Khaleel, V. M. & Narayana, B. (2011*c*). *Acta Cryst.* E**67**, o1313–o1314.
Gadzhieva, S. R., Mursalov, T. M., Makhmudov, K. T. & Chyragov, F. M. (2006). *Russ. J. Coord. Chem.* **32**, 304–308.
Gurbanov, A. V., Maharramov, A. M., Zubkov, F. I., Saifutdinov, A. M. & Guseinov, F. I. (2018). *Aust. J. Chem.* **71**, 190–194.
Gurbanov, A. V., Mahmudov, K. T., Kopylovich, M. N., Guedes da Silva, F. M., Sutradhar, M., Guseinov, F. I., Zubkov, F. I., Maharramov, A. M. & Pombeiro, A. J. L. (2017). *Dyes Pigments*, **138**, 107–111.
Hathwar, V. R., Sist, M., Jørgensen, M. R. V., Mamakhel, A. H., Wang, X., Hoffmann, C. M., Sugimoto, K., Overgaard, J. & Iversen, B. B. (2015). *IUCrJ*, **2**, 563–574.
Hazra, S., Martins, N. M. R., Mahmudov, K. T., Zubkov, F. I., Guedes da Silva, M. F. C. & Pombeiro, A. J. L. (2018). *J. Organomet. Chem.* **867**, 193–200.
Jlassi, R., Ribeiro, A. P. C., Guedes da Silva, M. F. C., Mahmudov, K. T., Kopylovich, M. N., Anisimova, T. B., Naïli, H., Tiago, G. A. O. & Pombeiro, A. J. L. (2014). *Eur. J. Inorg. Chem.* pp. 4541–4550.
Kvyatkovskaya, E. A., Zaytsev, V. P., Zubkov, F. I., Dorovatovskii, P. V., Zubavichus, Y. V. & Khrustalev, V. N. (2017). *Acta Cryst.* E**73**, 515–519.
Legon, A. C. (2017). *Phys. Chem. Chem. Phys.* **19**, 14884–14896.
Maharramov, A. M., Alieva, R. A., Mahmudov, K. T., Kurbanov, A. V. & Askerov, R. K. (2009). *Russ. J. Coord. Chem.* **35**, 704–709.
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. L. (2018). *Dyes Pigments*, **159**, 135–141.
Mahmoudi, G., Seth, S. K., Bauzá, A., Zubkov, F. I., Gurbanov, A. V., White, J., Stilinović, V., Doert, Th. & Frontera, A. (2018*c*). *CrystEngComm*, **20**, 2812–2821.
Mahmoudi, G., Zangrando, E., Mitoraj, M. P., Gurbanov, A. V., Zubkov, F. I., Moosavifar, M., Konyaeva, I. A., Kirillov, A. M. & Safin, D. A. (2018*a*). *New J. Chem.* **42**, 4959–4971.
Mahmoudi, G., Zaręba, J. K., Gurbanov, A. V., Bauzá, A., Zubkov, F. I., Kubicki, M., Stilinović, V., Kinzhybalo, V. & Frontera, A. (2018*b*). *Eur. J. Inorg. Chem.* pp. 4763–4772.
Mahmudov, K. T., Kopylovich, M. N., Guedes da Silva, M. F. C. & Pombeiro, A. J. L. (2017). *Dalton Trans.* **46**, 10121–10138.
Mahmudov, K. T., Kopylovich, M. N., Sabbatini, A., Drew, M. G. B., Martins, L. M. D. R. S., Pettinari, C. & Pombeiro, A. J. L. (2014). *Inorg. Chem.* **53**, 9946–9958.
Mahmudov, K. T. & Pombeiro, A. J. L. (2016). *Chem. Eur. J.* **22**, 16356–16398.
McKinnon, J. J., Jayatilaka, D. & Spackman, M. A. (2007). *Chem. Commun.* pp. 3814–3816.
Scheiner, S. (2013). *Acc. Chem. Res.* **46**, 280–288.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Shetnev, A. A. & Zubkov, F. I. (2017). *Chem. Heterocycl. C.* **53**, 495–497.
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., Maharramov, A. M., Gurbanov, A. V., Nenajdenko, V. G., Muzalevskiy, V. M., Mahmudov, K. T. & Kopylovich, M. N. (2013). *Catal. Today*, **217**, 76–79.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2019). E**75**, 237-241 [https://doi.org/10.1107/S2056989019000707]

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Computing details

Data collection: *APEX3* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: SHELXT2016 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Crystal data

$C_{14}H_8Cl_2FN_3O_2$
M_r = 340.13
Monoclinic, $P2_1/c$
a = 15.8644 (5) Å
b = 7.2242 (2) Å
c = 12.7595 (4) Å
β = 97.038 (2)°
V = 1451.32 (8) Å^3
Z = 4
$F(000)$ = 688
D_x = 1.557 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4877 reflections
θ = 3.1–25.9°
μ = 0.47 mm^{-1}
T = 296 K
Block, orange
0.34 × 0.23 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2003)
T_{min} = 0.861, T_{max} = 0.925
11383 measured reflections
2851 independent reflections
2359 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 26.0°, θ_{min} = 3.2°
h = −14→19
k = −8→8
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.089
S = 1.05
2851 reflections
199 parameters
0 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

$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 0.3915P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ 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	0.97386 (3)	1.20992 (9)	0.70124 (4)	0.07088 (18)
Cl2	0.92856 (3)	1.13756 (8)	0.90771 (4)	0.06189 (17)
F1	0.47733 (7)	0.7105 (2)	1.03511 (10)	0.0765 (4)
O1	0.68767 (10)	1.0573 (2)	0.24070 (11)	0.0720 (4)
O2	0.80699 (11)	0.9169 (2)	0.23689 (11)	0.0788 (5)
N1	0.69628 (8)	0.95452 (19)	0.76270 (10)	0.0402 (3)
N2	0.76871 (8)	1.00408 (18)	0.80363 (10)	0.0390 (3)
N3	0.75395 (11)	0.9908 (2)	0.28420 (12)	0.0521 (4)
C1	0.64239 (9)	0.8985 (2)	0.83834 (12)	0.0365 (3)
C2	0.66658 (10)	0.8947 (2)	0.94654 (12)	0.0406 (4)
H2	0.720428	0.934987	0.974163	0.049*
C3	0.61096 (11)	0.8315 (3)	1.01287 (13)	0.0481 (4)
H3	0.626481	0.828004	1.085535	0.058*
C4	0.53205 (11)	0.7736 (3)	0.96946 (14)	0.0488 (4)
C5	0.50562 (11)	0.7772 (3)	0.86378 (15)	0.0539 (5)
H5	0.451398	0.737848	0.837092	0.065*
C6	0.56182 (10)	0.8409 (3)	0.79752 (13)	0.0485 (4)
H6	0.545439	0.845134	0.725037	0.058*
C7	0.82440 (9)	1.0572 (2)	0.73073 (12)	0.0379 (3)
C8	0.89916 (10)	1.1249 (2)	0.77426 (13)	0.0440 (4)
C9	0.80338 (9)	1.0366 (2)	0.61456 (12)	0.0364 (3)
C10	0.73747 (10)	1.1361 (2)	0.55905 (13)	0.0426 (4)
H10	0.704682	1.214863	0.595145	0.051*
C11	0.72030 (10)	1.1192 (2)	0.45128 (13)	0.0433 (4)
H11	0.675811	1.184705	0.414101	0.052*
C12	0.77022 (10)	1.0035 (2)	0.39955 (12)	0.0392 (4)
C13	0.83517 (11)	0.9005 (2)	0.45209 (13)	0.0456 (4)
H13	0.867698	0.822187	0.415369	0.055*
C14	0.85078 (10)	0.9161 (2)	0.55996 (13)	0.0433 (4)
H14	0.893493	0.845300	0.596958	0.052*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0507 (3)	0.0932 (4)	0.0709 (3)	−0.0250 (3)	0.0163 (2)	−0.0040 (3)
Cl2	0.0511 (3)	0.0837 (4)	0.0477 (3)	0.0004 (2)	−0.0067 (2)	−0.0095 (2)

F1	0.0605 (7)	0.1118 (10)	0.0615 (7)	−0.0204 (7)	0.0249 (6)	0.0118 (7)
O1	0.0832 (10)	0.0843 (10)	0.0443 (7)	−0.0050 (9)	−0.0089 (7)	0.0071 (7)
O2	0.1061 (12)	0.0883 (11)	0.0464 (8)	0.0037 (9)	0.0273 (8)	−0.0124 (7)
N1	0.0380 (7)	0.0491 (8)	0.0341 (7)	0.0013 (6)	0.0068 (5)	−0.0010 (6)
N2	0.0381 (7)	0.0441 (7)	0.0352 (7)	0.0023 (6)	0.0062 (5)	0.0007 (6)
N3	0.0712 (10)	0.0461 (8)	0.0395 (8)	−0.0162 (8)	0.0090 (8)	0.0005 (7)
C1	0.0369 (8)	0.0395 (8)	0.0337 (7)	0.0030 (6)	0.0077 (6)	−0.0024 (6)
C2	0.0386 (8)	0.0453 (9)	0.0374 (8)	−0.0011 (7)	0.0031 (6)	−0.0021 (7)
C3	0.0510 (10)	0.0597 (11)	0.0342 (8)	−0.0030 (8)	0.0076 (7)	0.0020 (8)
C4	0.0464 (9)	0.0572 (11)	0.0458 (9)	−0.0041 (8)	0.0180 (8)	0.0021 (8)
C5	0.0372 (9)	0.0727 (12)	0.0517 (10)	−0.0091 (8)	0.0056 (8)	−0.0067 (9)
C6	0.0428 (9)	0.0670 (12)	0.0353 (8)	−0.0010 (8)	0.0031 (7)	−0.0046 (8)
C7	0.0361 (8)	0.0395 (8)	0.0384 (8)	0.0044 (6)	0.0065 (6)	0.0013 (7)
C8	0.0382 (8)	0.0491 (9)	0.0446 (9)	0.0023 (7)	0.0048 (7)	−0.0012 (8)
C9	0.0331 (7)	0.0395 (8)	0.0376 (8)	−0.0017 (6)	0.0079 (6)	0.0027 (7)
C10	0.0416 (8)	0.0463 (9)	0.0414 (9)	0.0092 (7)	0.0116 (7)	0.0022 (7)
C11	0.0400 (8)	0.0475 (9)	0.0422 (9)	0.0032 (7)	0.0046 (7)	0.0087 (7)
C12	0.0457 (9)	0.0389 (8)	0.0343 (8)	−0.0099 (7)	0.0093 (7)	0.0016 (7)
C13	0.0455 (9)	0.0467 (9)	0.0469 (9)	0.0038 (8)	0.0154 (7)	−0.0044 (8)
C14	0.0384 (8)	0.0473 (9)	0.0447 (9)	0.0086 (7)	0.0069 (7)	0.0031 (7)

Geometric parameters (Å, º)

Cl1—C8	1.7088 (17)	C5—C6	1.381 (2)
Cl2—C8	1.7120 (17)	C5—H5	0.9300
F1—C4	1.3569 (19)	C6—H6	0.9300
O1—N3	1.225 (2)	C7—C8	1.339 (2)
O2—N3	1.217 (2)	C7—C9	1.486 (2)
N1—N2	1.2547 (18)	C9—C10	1.389 (2)
N1—C1	1.4242 (19)	C9—C14	1.392 (2)
N2—C7	1.4123 (19)	C10—C11	1.374 (2)
N3—C12	1.466 (2)	C10—H10	0.9300
C1—C6	1.384 (2)	C11—C12	1.375 (2)
C1—C2	1.387 (2)	C11—H11	0.9300
C2—C3	1.373 (2)	C12—C13	1.377 (2)
C2—H2	0.9300	C13—C14	1.373 (2)
C3—C4	1.371 (2)	C13—H13	0.9300
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.362 (3)		
N2—N1—C1	113.22 (12)	C8—C7—C9	121.96 (14)
N1—N2—C7	114.73 (13)	N2—C7—C9	123.20 (13)
O2—N3—O1	123.66 (16)	C7—C8—Cl1	122.91 (13)
O2—N3—C12	118.56 (16)	C7—C8—Cl2	123.47 (13)
O1—N3—C12	117.78 (16)	Cl1—C8—Cl2	113.61 (9)
C6—C1—C2	119.92 (14)	C10—C9—C14	119.18 (14)
C6—C1—N1	115.71 (14)	C10—C9—C7	121.30 (14)
C2—C1—N1	124.35 (14)	C14—C9—C7	119.53 (14)

C3—C2—C1	119.97 (15)	C11—C10—C9	120.58 (15)
C3—C2—H2	120.0	C11—C10—H10	119.7
C1—C2—H2	120.0	C9—C10—H10	119.7
C4—C3—C2	118.44 (15)	C10—C11—C12	118.64 (15)
C4—C3—H3	120.8	C10—C11—H11	120.7
C2—C3—H3	120.8	C12—C11—H11	120.7
F1—C4—C5	118.34 (16)	C11—C12—C13	122.39 (15)
F1—C4—C3	118.34 (16)	C11—C12—N3	118.64 (15)
C5—C4—C3	123.32 (16)	C13—C12—N3	118.96 (15)
C4—C5—C6	117.96 (16)	C14—C13—C12	118.43 (15)
C4—C5—H5	121.0	C14—C13—H13	120.8
C6—C5—H5	121.0	C12—C13—H13	120.8
C5—C6—C1	120.38 (16)	C13—C14—C9	120.72 (15)
C5—C6—H6	119.8	C13—C14—H14	119.6
C1—C6—H6	119.8	C9—C14—H14	119.6
C8—C7—N2	114.83 (14)		

C1—N1—N2—C7	178.86 (13)	C8—C7—C9—C10	−116.26 (18)
N2—N1—C1—C6	−179.49 (15)	N2—C7—C9—C10	65.2 (2)
N2—N1—C1—C2	−1.1 (2)	C8—C7—C9—C14	63.4 (2)
C6—C1—C2—C3	0.9 (2)	N2—C7—C9—C14	−115.15 (17)
N1—C1—C2—C3	−177.43 (16)	C14—C9—C10—C11	−1.4 (2)
C1—C2—C3—C4	−0.1 (3)	C7—C9—C10—C11	178.25 (15)
C2—C3—C4—F1	179.75 (16)	C9—C10—C11—C12	−0.7 (2)
C2—C3—C4—C5	−0.6 (3)	C10—C11—C12—C13	1.8 (2)
F1—C4—C5—C6	−179.76 (17)	C10—C11—C12—N3	−177.72 (14)
C3—C4—C5—C6	0.6 (3)	O2—N3—C12—C11	166.51 (16)
C4—C5—C6—C1	0.2 (3)	O1—N3—C12—C11	−12.8 (2)
C2—C1—C6—C5	−0.9 (3)	O2—N3—C12—C13	−13.0 (2)
N1—C1—C6—C5	177.56 (16)	O1—N3—C12—C13	167.66 (16)
N1—N2—C7—C8	174.62 (14)	C11—C12—C13—C14	−0.7 (2)
N1—N2—C7—C9	−6.7 (2)	N3—C12—C13—C14	178.84 (15)
N2—C7—C8—Cl1	−176.19 (11)	C12—C13—C14—C9	−1.6 (2)
N2—C7—C8—Cl2	2.9 (2)	C10—C9—C14—C13	2.6 (2)
C9—C7—C8—Cl2	−175.78 (12)	C7—C9—C14—C13	−177.11 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O1^{i}	0.93	2.52	3.369 (2)	152

Symmetry code: (i) *x*, −*y*+5/2, *z*+1/2.