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(*Z*)-3-[(3,5-Dichloroanilino)methylidene]-5-(4-methylphenyl)furan-2(3*H*)-one

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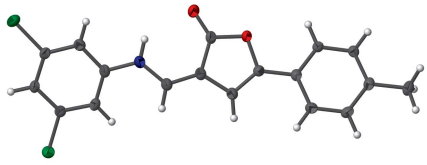
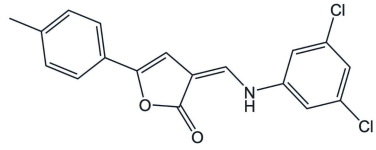
Structural data: full structural data are available from iucrdata.iucr.org

The crystal structure of the title compound, $C_{18}H_{13}Cl_2NO_2$, at 120 K has triclinic ($P\overline{1}$) symmetry. The molecule demonstrates non-planarity in the solid state and a *Z* configuration for the exocyclic C=C bond. The *Z* form is stabilized by the presence of an intramolecular N—H···O hydrogen bond with an O···H interatomic distance of 2.18 (2) Å.

3D view



Chemical scheme



Structure description

Enamine derivatives of furan-2(3*H*)-ones have a push–pull character and may be of interest for the creation of molecular switches (Osipov *et al.*, 2017). The title molecule is non-planar (Fig. 1) with the *p*-tolyl substituent rotated about the mean plane of the furanone ring by approximately −10° [C18—C17—C6—O1 torsion angle = −9.68 (19)°]. The C4=C7 bond adopts the *Z* configuration. The benzene ring of the 3,5-dichlorophenyl substituent is also out of the plane of the molecule [the dihedral angle between the mean planes of the furanone and 3,5-dichlorophenyl rings is 14.74 (7)°], which is a consequence of the repulsion of hydrogen atoms H16 of the aromatic substituent and H7 of enamine fragment (the interatomic distance is 2.12 Å, which is less than the sum of van der Waals radii of 2.38 Å). An additional stabilization factor of the molecule in the *Z*-configuration is the intramolecular hydrogen bond with an O3···H8 interatomic distance of 2.18 (2) Å (Table 1, Fig. 1).

Molecules in the crystal are oriented in a head-to-tail fashion (Fig. 2). The interplanar distances between identically oriented molecules are more than 7 Å. The shortest intercentroid distance in the crystal is between the furanone and 3,5-dichlorophenyl rings [$Cg1\cdots Cg2^{i}$ = 3.5817 (9) Å; symmetry code: (i) $-x+1, -y+1, -z+1$; *Cg*1 and *Cg*2 are the centroids of the furanone and 3,5-dichlorophenyl rings, respectively].

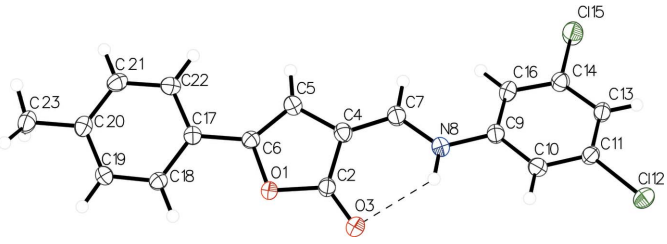


Figure 1
The molecular structure of the title compound with the atom labelling and displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

The synthesis of title compound was performed according to the method described by Osipov *et al.* (2017). Briefly, about 7 ml of benzene, 1.78 g (12.02 mmol) of triethyl orthoformate, 0.40 g (1.67 mmol) of 5-(*p*-tolyl)furan-2(3*H*)-one and 0.27 g (1.67 mmol) of 3,5-dichloroaniline were placed into a round-bottom flask equipped with a Liebig reflux condenser, and the reaction mixture was refluxed for 2 h. The precipitate of 3-[(3,5-dichloroanilino)methylidene]-5-(4-methylphenyl)furan-2(3*H*)-one was filtered off, washed with benzene and then with chloroform, dried, and recrystallized from DMF. Yield 0.45 g (78%), yellow crystals. A suitable single-crystal for X-ray analysis was obtained by slow cooling of a saturated solution of the title compound in benzene.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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

D—H···A	D—H	H···A	D···A	D—H···A
N8—H8···O3	0.89 (2)	2.18 (2)	2.8553 (16)	132.2 (19)

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{13}Cl_2NO_2$
M_r	346.19
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	120
a, b, c (Å)	7.4005 (8), 10.4272 (11), 10.5704 (11)
α, β, γ (°)	86.076 (2), 72.382 (2), 81.325 (2)
V (Å^3)	768.30 (14)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.43
Crystal size (mm)	0.6 × 0.1 × 0.1
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.670, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14989, 3956, 3576
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.102, 1.05
No. of reflections	3956
No. of parameters	213
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.65, −0.26

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

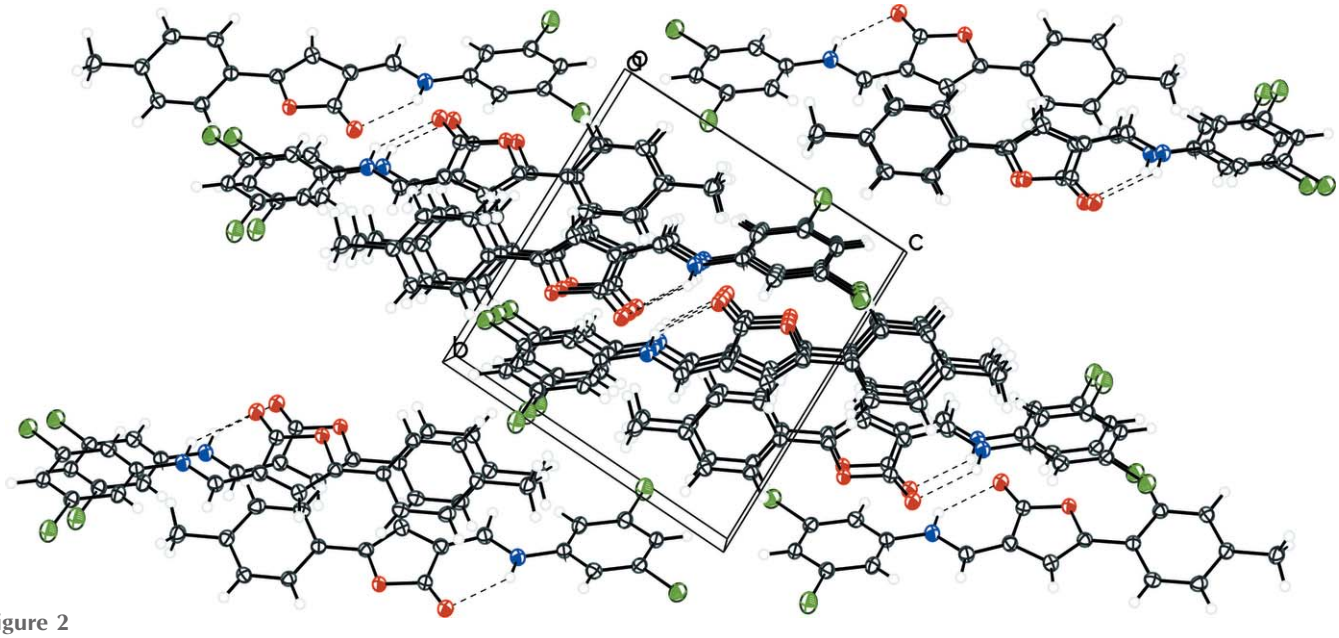


Figure 2
The crystal packing of the title compound, viewed down the *a* axis.

Funding information

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References

Bruker (2013). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Osipov, A. K., Anis'kov, A. A., Grinev, V. S. & Yegorova, A. Yu. (2017). *Magn. Reson. Chem.* **55**, 730–737.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2018). **3**, x181224 [https://doi.org/10.1107/S2414314618012245]

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Crystal data

$C_{18}H_{13}Cl_2NO_2$
M_r = 346.19
Triclinic, $P\overline{1}$
a = 7.4005 (8) Å
b = 10.4272 (11) Å
c = 10.5704 (11) Å
α = 86.076 (2)°
β = 72.382 (2)°
γ = 81.325 (2)°
V = 768.30 (14) Å^3
Z = 2
$F(000)$ = 356
D_x = 1.496 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9480 reflections
θ = 2.8–33.6°
μ = 0.43 mm^{-1}
T = 120 K
Needle, metallic orangish yellow
0.6 × 0.1 × 0.1 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.670, T_{max} = 0.747
14989 measured reflections
3956 independent reflections
3576 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 28.7°, θ_{min} = 2.0°
h = −9→9
k = −14→14
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 1.05
3956 reflections
213 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.3381P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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.

Refinement. The structure was solved by the internal phasing method and refined by the least-squares method in the anisotropic full-matrix approximation in accordance with F^2_{hkl}. The hydrogen atom of NH group in the compound was localized from a difference electron density map and refined in the isotropic approximation.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl12	0.73598 (6)	0.17894 (4)	0.95054 (4)	0.03192 (11)
Cl15	0.28370 (5)	0.00476 (4)	0.70818 (4)	0.03283 (11)
O1	0.85703 (14)	0.63696 (9)	0.18548 (9)	0.0221 (2)
O3	0.87688 (16)	0.58521 (10)	0.39293 (10)	0.0284 (2)
N8	0.71111 (18)	0.35501 (11)	0.49406 (12)	0.0236 (2)
H8	0.782 (3)	0.412 (2)	0.506 (2)	0.041 (5)*
C2	0.8260 (2)	0.56018 (12)	0.29926 (13)	0.0218 (3)
C4	0.72893 (19)	0.45467 (13)	0.28098 (13)	0.0220 (3)
C5	0.70627 (19)	0.47396 (13)	0.15031 (13)	0.0220 (3)
H5	0.648261	0.420524	0.109156	0.026*
C6	0.78279 (18)	0.58216 (12)	0.09699 (13)	0.0192 (2)
C7	0.6746 (2)	0.36063 (13)	0.37583 (13)	0.0232 (3)
H7	0.608028	0.296280	0.357858	0.028*
C9	0.64593 (19)	0.26777 (12)	0.59953 (13)	0.0213 (3)
C10	0.7183 (2)	0.26434 (13)	0.70751 (13)	0.0232 (3)
H10	0.810977	0.318045	0.708104	0.028*
C11	0.65202 (19)	0.18079 (13)	0.81396 (13)	0.0215 (3)
C13	0.51978 (18)	0.09828 (12)	0.81629 (12)	0.0213 (3)
H13	0.477960	0.040465	0.889271	0.026*
C14	0.45206 (19)	0.10446 (13)	0.70717 (13)	0.0217 (3)
C16	0.51059 (19)	0.18805 (13)	0.59919 (13)	0.0222 (3)
H16	0.459533	0.190915	0.526541	0.027*
C17	0.80132 (18)	0.65033 (12)	−0.02995 (12)	0.0190 (2)
C18	0.86162 (18)	0.77317 (12)	−0.05282 (13)	0.0198 (2)
H18	0.891237	0.813026	0.015255	0.024*
C19	0.87821 (18)	0.83680 (12)	−0.17475 (13)	0.0206 (2)
H19	0.918467	0.920294	−0.188607	0.025*
C20	0.83712 (19)	0.78098 (13)	−0.27751 (13)	0.0213 (3)
C21	0.7763 (2)	0.65877 (13)	−0.25387 (14)	0.0237 (3)
H21	0.746755	0.619139	−0.322100	0.028*
C22	0.75842 (19)	0.59414 (13)	−0.13209 (13)	0.0222 (3)
H22	0.716687	0.511095	−0.118071	0.027*
C23	0.8603 (2)	0.85101 (14)	−0.41077 (14)	0.0274 (3)
H23A	0.748654	0.916703	−0.405238	0.041*
H23B	0.870926	0.788438	−0.478604	0.041*
H23C	0.976178	0.893178	−0.434616	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl12	0.0422 (2)	0.0348 (2)	0.02564 (18)	−0.01046 (15)	−0.01803 (15)	0.00003 (14)
Cl15	0.0342 (2)	0.0421 (2)	0.02820 (19)	−0.01988 (16)	−0.01268 (15)	0.00627 (14)
O1	0.0303 (5)	0.0196 (4)	0.0175 (4)	−0.0055 (4)	−0.0081 (4)	0.0014 (3)
O3	0.0406 (6)	0.0261 (5)	0.0205 (5)	−0.0074 (4)	−0.0107 (4)	0.0007 (4)
N8	0.0277 (6)	0.0219 (5)	0.0211 (5)	−0.0065 (4)	−0.0061 (5)	0.0020 (4)

C2	0.0254 (6)	0.0195 (6)	0.0180 (6)	−0.0012 (5)	−0.0041 (5)	0.0004 (4)
C4	0.0220 (6)	0.0205 (6)	0.0217 (6)	−0.0014 (5)	−0.0047 (5)	0.0008 (5)
C5	0.0217 (6)	0.0214 (6)	0.0232 (6)	−0.0039 (5)	−0.0070 (5)	0.0019 (5)
C6	0.0188 (6)	0.0189 (6)	0.0195 (6)	−0.0010 (4)	−0.0057 (5)	−0.0013 (4)
C7	0.0242 (6)	0.0223 (6)	0.0224 (6)	−0.0037 (5)	−0.0061 (5)	0.0014 (5)
C9	0.0224 (6)	0.0191 (6)	0.0191 (6)	0.0004 (5)	−0.0029 (5)	0.0002 (4)
C10	0.0250 (6)	0.0212 (6)	0.0230 (6)	−0.0044 (5)	−0.0059 (5)	−0.0008 (5)
C11	0.0250 (6)	0.0217 (6)	0.0178 (6)	−0.0014 (5)	−0.0070 (5)	−0.0017 (5)
C13	0.0236 (6)	0.0220 (6)	0.0168 (6)	−0.0020 (5)	−0.0049 (5)	0.0014 (5)
C14	0.0202 (6)	0.0243 (6)	0.0204 (6)	−0.0042 (5)	−0.0053 (5)	0.0000 (5)
C16	0.0220 (6)	0.0264 (6)	0.0178 (6)	−0.0015 (5)	−0.0068 (5)	0.0012 (5)
C17	0.0174 (5)	0.0191 (6)	0.0191 (6)	−0.0003 (4)	−0.0043 (4)	−0.0001 (4)
C18	0.0201 (6)	0.0206 (6)	0.0194 (6)	−0.0023 (5)	−0.0067 (5)	−0.0012 (4)
C19	0.0207 (6)	0.0190 (6)	0.0219 (6)	−0.0024 (5)	−0.0062 (5)	0.0011 (5)
C20	0.0193 (6)	0.0235 (6)	0.0196 (6)	0.0023 (5)	−0.0061 (5)	0.0004 (5)
C21	0.0254 (6)	0.0248 (6)	0.0230 (6)	−0.0009 (5)	−0.0108 (5)	−0.0042 (5)
C22	0.0226 (6)	0.0201 (6)	0.0249 (6)	−0.0030 (5)	−0.0084 (5)	−0.0022 (5)
C23	0.0317 (7)	0.0290 (7)	0.0198 (6)	0.0020 (6)	−0.0083 (5)	0.0010 (5)

Geometric parameters (Å, º)

Cl12—C11	1.7338 (14)	C11—C13	1.3911 (19)
Cl15—C14	1.7362 (14)	C13—H13	0.9500
O1—C2	1.3771 (15)	C13—C14	1.3841 (18)
O1—C6	1.4079 (15)	C14—C16	1.3887 (18)
O3—C2	1.2171 (17)	C16—H16	0.9500
N8—H8	0.89 (2)	C17—C18	1.4010 (17)
N8—C7	1.3521 (18)	C17—C22	1.4000 (18)
N8—C9	1.4040 (17)	C18—H18	0.9500
C2—C4	1.4543 (19)	C18—C19	1.3886 (17)
C4—C5	1.4381 (18)	C19—H19	0.9500
C4—C7	1.3671 (18)	C19—C20	1.3968 (18)
C5—H5	0.9500	C20—C21	1.3964 (19)
C5—C6	1.3504 (18)	C20—C23	1.5158 (18)
C6—C17	1.4533 (17)	C21—H21	0.9500
C7—H7	0.9500	C21—C22	1.3907 (19)
C9—C10	1.3965 (19)	C22—H22	0.9500
C9—C16	1.3956 (19)	C23—H23A	0.9800
C10—H10	0.9500	C23—H23B	0.9800
C10—C11	1.3880 (18)	C23—H23C	0.9800
C2—O1—C6	107.58 (10)	C13—C14—Cl15	117.97 (10)
C7—N8—H8	116.8 (14)	C13—C14—C16	122.82 (12)
C7—N8—C9	125.78 (12)	C16—C14—Cl15	119.19 (10)
C9—N8—H8	117.4 (14)	C9—C16—H16	120.7
O1—C2—C4	107.75 (11)	C14—C16—C9	118.67 (12)
O3—C2—O1	121.51 (12)	C14—C16—H16	120.7
O3—C2—C4	130.74 (12)	C18—C17—C6	121.04 (12)

C5—C4—C2	106.19 (11)	C22—C17—C6	120.34 (12)
C7—C4—C2	123.25 (12)	C22—C17—C18	118.62 (12)
C7—C4—C5	130.56 (13)	C17—C18—H18	119.9
C4—C5—H5	126.4	C19—C18—C17	120.17 (12)
C6—C5—C4	107.27 (12)	C19—C18—H18	119.9
C6—C5—H5	126.4	C18—C19—H19	119.2
O1—C6—C17	115.91 (11)	C18—C19—C20	121.57 (12)
C5—C6—O1	111.20 (11)	C20—C19—H19	119.2
C5—C6—C17	132.89 (12)	C19—C20—C23	120.65 (12)
N8—C7—C4	123.02 (13)	C21—C20—C19	117.97 (12)
N8—C7—H7	118.5	C21—C20—C23	121.37 (12)
C4—C7—H7	118.5	C20—C21—H21	119.5
C10—C9—N8	117.85 (12)	C22—C21—C20	121.07 (12)
C16—C9—N8	121.80 (12)	C22—C21—H21	119.5
C16—C9—C10	120.34 (12)	C17—C22—H22	119.7
C9—C10—H10	120.7	C21—C22—C17	120.60 (12)
C11—C10—C9	118.57 (13)	C21—C22—H22	119.7
C11—C10—H10	120.7	C20—C23—H23A	109.5
C10—C11—Cl12	118.97 (11)	C20—C23—H23B	109.5
C10—C11—C13	122.77 (12)	C20—C23—H23C	109.5
C13—C11—Cl12	118.25 (10)	H23A—C23—H23B	109.5
C11—C13—H13	121.6	H23A—C23—H23C	109.5
C14—C13—C11	116.80 (12)	H23B—C23—H23C	109.5
C14—C13—H13	121.6		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H8···O3	0.89 (2)	2.18 (2)	2.8553 (16)	132.2 (19)