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2-(1*H*-1,2,3-Benzotriazol-1-yl)-1-(4-ethylbenzoyl)ethyl 2,4-dichlorobenzoate

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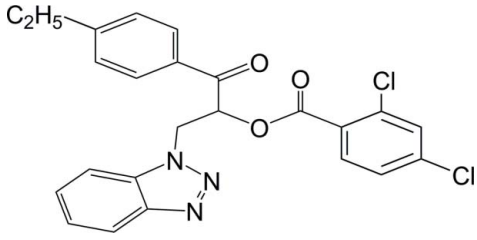
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.046; wR factor = 0.124; data-to-parameter ratio = 12.8.

In the title molecule, $C_{24}H_{19}Cl_2N_3$, the dihedral angles between the benzotriazole group and the ethyl- and dichloro-substituted benzene rings are 16.53 (1) and 82.09 (1)°, respectively. The crystal structure is stabilized by weak intermolecular C—H···O interactions.

Related literature

For related literature, see: Chen & Wu (2005). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{24}H_{19}Cl_2N_3O_3$
M_r = 468.32
Triclinic, $P\overline{1}$
a = 9.251 (3) Å
b = 10.904 (4) Å
c = 11.057 (4) Å
α = 88.327 (6)°
β = 86.442 (6)°
γ = 83.304 (5)°
V = 1105.4 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 298 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.938, T_{max} = 0.991
5375 measured reflections
3700 independent reflections
2701 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.124
S = 1.03
3700 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O3^{i}	0.93	2.51	3.425 (3)	169
C12—H12A···O1^{ii}	0.93	2.52	3.378 (4)	154

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x+1, y, z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2614).

References

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Chen, Z.-Y. & Wu, M.-J. (2005). *Org. Lett.* **7**, 475–477.
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supplementary materials

Acta Cryst. (2008). E**64**, o942 [**doi:10.1107/S1600536808011951**]

2-(1*H*-1,2,3-Benzotriazol-1-yl)-1-(4-ethylbenzoyl)ethyl 2,4-dichlorobenzoate

W.-L. Zeng

Comment

1*H*-Benzotriazole and its derivatives are an important class of compounds because they exhibit a broad spectrum of pharmacological activities such as antifungal, antitumor and antineoplastic activities (Chen & Wu, 2005). All bond lengths and angles in the title molecule (I) are within normal ranges (Allen *et al.*, 1987). The benzotriazole ring system is essentially planar, with a dihedral angle of 1.05 (1)° between the triazole ring (atoms N1—N3/C10/C16) and the benzene ring (C10—C16). The dihedral angles between the mean planes of the benzotriazole system and ring atoms C1—C6 and C17—C22 are 82.09 (1) and 16.53 (1), respectively. The dihedral angle between rings atoms C1—C6 and C17—C22 is 89.47 (2). In the crystal structure, weak intermolecular C—H···O hydrogen bonds (Table 1) link molecules into chains extended along the *a* axis.

Experimental

Bromine (3.2 g,0.02 mol) was added dropwise to a solution of 3-(1*H*-benzo[*d*][1,2,3]triazol-1-yl)-1-(4-ethylphenyl)propan-1-one (5.58 g,0.02 mol)and sodium acetate(1.6 g,0.02 mol) in acetic acid (50 ml). The reaction proceeded for 7 h. Water (50 ml) and chloroform (20 ml) were then added. The organic layer was washed successively with saturated sodium bicarbonate solution and brine, dried over anhydrous magnesium sulfate and the chloroform solution filtered. It was cooled with ice-water, and then an acetone solution (10 ml) of 2,4-dichlorobenzoic acid (3.8 g,0.02 mol) and tri ethylamine (2.8 ml) was added. The mixture was stirred with ice-water for about 6 h. The solution was then filtered and concentrated. Single crystals were obtained by slow evaporation of anacetone-ethylacetate(1:1 *v*/*v*) solution of (I) at room temperature over a period of one week.

Refinement

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(methyl C) H atoms.

Figures

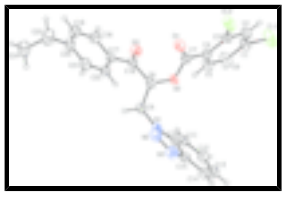

Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids.

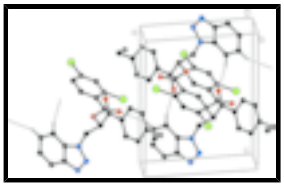

Fig. 2. Part of the crystal structure of (I) showing hydrogen bonds as dashed lines. Only H atoms involved in hydrogen bonds are shown.

2-(1*H*-1,2,3-Benzotriazol-1-yl)-1-(4-ethylbenzoyl)ethyl 2,4-dichlorobenzoate

Crystal data

$C_{24}H_{19}Cl_2N_3O_3$	$Z = 2$
$M_r = 468.32$	$F_{000} = 484$
Triclinic, $P\overline{1}$	$D_x = 1.407$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.251$ (3) Å	Cell parameters from 3700 reflections
$b = 10.904$ (4) Å	$\theta = 1.9$–25.0º
$c = 11.057$ (4) Å	$\mu = 0.33$ mm^{-1}
$\alpha = 88.327$ (6)º	$T = 298$ (2) K
$\beta = 86.442$ (6)º	Block, colorless
$\gamma = 83.304$ (5)º	0.20 × 0.20 × 0.10 mm
$V = 1105.4$ (6) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3700 independent reflections
Radiation source: fine-focus sealed tube	2701 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.016$
$T = 298$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.9$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -7 \rightarrow 10$
$T_{min} = 0.938$, $T_{max} = 0.991$	$k = -12 \rightarrow 12$
5375 measured reflections	$l = -10 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.124$	$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.3617P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
3700 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
289 parameters	$\Delta\rho_{min} = -0.34$ e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.63408 (12)	0.54323 (8)	0.16704 (8)	0.0986 (4)
Cl2	0.17075 (9)	0.52373 (8)	0.46851 (10)	0.0910 (3)
N1	0.5431 (2)	0.10230 (19)	0.83967 (17)	0.0471 (5)
N2	0.4769 (3)	0.1341 (2)	0.9489 (2)	0.0609 (6)
N3	0.5560 (3)	0.2042 (2)	1.0028 (2)	0.0672 (7)
O1	0.1957 (2)	0.28033 (18)	0.58441 (18)	0.0651 (6)
O2	0.41844 (18)	0.18987 (15)	0.61852 (15)	0.0468 (4)
O3	0.3025 (2)	0.01135 (18)	0.51586 (16)	0.0608 (5)
C1	0.5439 (3)	0.4634 (3)	0.2799 (2)	0.0596 (8)
C2	0.4102 (3)	0.5146 (2)	0.3249 (2)	0.0577 (7)
H2B	0.3685	0.5899	0.2942	0.069*
C3	0.3380 (3)	0.4532 (2)	0.4161 (2)	0.0501 (6)
C4	0.4002 (3)	0.3419 (2)	0.4646 (2)	0.0436 (6)
C5	0.5355 (3)	0.2945 (2)	0.4167 (2)	0.0568 (7)
H5A	0.5794	0.2204	0.4483	0.068*
C6	0.6073 (4)	0.3530 (3)	0.3245 (3)	0.0694 (9)
H6A	0.6977	0.3184	0.2924	0.083*
C7	0.3240 (3)	0.2712 (2)	0.5603 (2)	0.0456 (6)
C8	0.3506 (3)	0.1039 (2)	0.6972 (2)	0.0439 (6)
H8A	0.2831	0.1483	0.7571	0.053*
C9	0.4734 (3)	0.0290 (2)	0.7597 (2)	0.0482 (6)
H9A	0.4352	−0.0383	0.8059	0.058*
H9B	0.5452	−0.0065	0.6990	0.058*
C10	0.6686 (3)	0.1556 (2)	0.8221 (2)	0.0431 (6)
C11	0.7727 (3)	0.1540 (2)	0.7266 (2)	0.0503 (6)
H11A	0.7664	0.1095	0.6570	0.060*
C12	0.8851 (3)	0.2213 (3)	0.7409 (3)	0.0601 (8)
H12A	0.9578	0.2228	0.6790	0.072*
C13	0.8951 (3)	0.2882 (3)	0.8449 (3)	0.0700 (9)
H13A	0.9738	0.3331	0.8504	0.084*
C14	0.7928 (4)	0.2892 (3)	0.9380 (3)	0.0692 (8)

H14A	0.7999	0.3340	1.0073	0.083*
C15	0.6762 (3)	0.2204 (2)	0.9264 (2)	0.0538 (7)
C16	0.2697 (3)	0.0221 (2)	0.6232 (2)	0.0431 (6)
C17	0.1553 (3)	−0.0460 (2)	0.6839 (2)	0.0411 (6)
C18	0.0778 (3)	−0.1156 (3)	0.6140 (2)	0.0562 (7)
H18A	0.1012	−0.1206	0.5311	0.067*
C19	−0.0327 (3)	−0.1769 (3)	0.6645 (3)	0.0643 (8)
H19A	−0.0826	−0.2233	0.6154	0.077*
C20	−0.0714 (3)	−0.1716 (3)	0.7863 (3)	0.0584 (7)
C21	0.0066 (3)	−0.1038 (3)	0.8564 (3)	0.0606 (8)
H21A	−0.0172	−0.0997	0.9392	0.073*
C22	0.1188 (3)	−0.0418 (2)	0.8074 (2)	0.0514 (7)
H22A	0.1699	0.0029	0.8572	0.062*
C23	−0.1960 (4)	−0.2369 (3)	0.8398 (4)	0.0919 (12)
H23A	−0.2398	−0.1926	0.9102	0.110*
H23B	−0.2696	−0.2351	0.7808	0.110*
C24	−0.1512 (5)	−0.3659 (4)	0.8759 (4)	0.1279 (18)
H24A	−0.2344	−0.4024	0.9103	0.192*
H24B	−0.0790	−0.3684	0.9349	0.192*
H24C	−0.1110	−0.4112	0.8061	0.192*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1231 (8)	0.0765 (6)	0.0850 (6)	0.0018 (5)	0.0436 (6)	0.0298 (5)
Cl2	0.0503 (5)	0.0744 (5)	0.1377 (8)	0.0148 (4)	0.0167 (5)	0.0406 (5)
N1	0.0510 (13)	0.0529 (12)	0.0360 (11)	−0.0061 (10)	0.0035 (10)	0.0096 (9)
N2	0.0689 (16)	0.0705 (16)	0.0410 (13)	−0.0087 (13)	0.0113 (12)	0.0072 (11)
N3	0.0821 (19)	0.0766 (17)	0.0422 (13)	−0.0113 (15)	0.0051 (13)	0.0002 (12)
O1	0.0416 (12)	0.0720 (13)	0.0788 (14)	−0.0037 (10)	0.0036 (9)	0.0222 (10)
O2	0.0430 (10)	0.0462 (10)	0.0507 (10)	−0.0075 (8)	−0.0010 (8)	0.0142 (8)
O3	0.0685 (13)	0.0705 (13)	0.0441 (11)	−0.0188 (10)	0.0123 (9)	−0.0010 (9)
C1	0.074 (2)	0.0521 (16)	0.0493 (16)	−0.0051 (15)	0.0089 (14)	0.0100 (13)
C2	0.0620 (19)	0.0476 (16)	0.0618 (18)	−0.0022 (14)	−0.0054 (14)	0.0154 (13)
C3	0.0428 (15)	0.0477 (15)	0.0590 (16)	−0.0023 (12)	−0.0059 (12)	0.0075 (12)
C4	0.0450 (14)	0.0429 (14)	0.0434 (14)	−0.0080 (11)	−0.0032 (11)	0.0038 (11)
C5	0.0624 (18)	0.0464 (15)	0.0559 (17)	0.0070 (13)	0.0107 (13)	0.0119 (13)
C6	0.074 (2)	0.0603 (18)	0.0643 (19)	0.0143 (16)	0.0231 (16)	0.0125 (15)
C7	0.0416 (15)	0.0445 (14)	0.0501 (15)	−0.0042 (11)	−0.0027 (12)	0.0037 (12)
C8	0.0447 (15)	0.0441 (14)	0.0419 (13)	−0.0077 (11)	0.0042 (11)	0.0090 (11)
C9	0.0530 (16)	0.0463 (14)	0.0451 (14)	−0.0090 (12)	0.0010 (12)	0.0078 (11)
C10	0.0456 (15)	0.0429 (13)	0.0392 (13)	−0.0006 (11)	−0.0033 (11)	0.0097 (11)
C11	0.0472 (16)	0.0512 (15)	0.0501 (16)	−0.0002 (12)	0.0030 (12)	0.0073 (12)
C12	0.0459 (16)	0.0578 (17)	0.074 (2)	−0.0024 (14)	0.0039 (14)	0.0090 (15)
C13	0.0560 (19)	0.0633 (19)	0.092 (2)	−0.0130 (15)	−0.0131 (18)	0.0106 (18)
C14	0.083 (2)	0.0620 (18)	0.066 (2)	−0.0107 (17)	−0.0198 (18)	−0.0055 (15)
C15	0.0627 (18)	0.0527 (16)	0.0453 (15)	−0.0022 (14)	−0.0088 (13)	0.0043 (12)
C16	0.0414 (14)	0.0424 (13)	0.0429 (15)	0.0001 (11)	0.0055 (11)	0.0058 (11)

C17	0.0401 (14)	0.0385 (13)	0.0428 (14)	−0.0008 (11)	0.0028 (11)	0.0042 (11)
C18	0.0565 (17)	0.0672 (18)	0.0454 (15)	−0.0129 (14)	0.0013 (13)	0.0010 (13)
C19	0.0560 (18)	0.072 (2)	0.068 (2)	−0.0215 (15)	−0.0003 (15)	−0.0026 (15)
C20	0.0497 (17)	0.0519 (16)	0.073 (2)	−0.0095 (13)	0.0081 (14)	0.0057 (14)
C21	0.0667 (19)	0.0624 (18)	0.0506 (16)	−0.0116 (15)	0.0165 (14)	0.0087 (14)
C22	0.0572 (17)	0.0512 (15)	0.0466 (15)	−0.0130 (13)	0.0014 (12)	0.0018 (12)
C23	0.078 (2)	0.094 (3)	0.106 (3)	−0.039 (2)	0.028 (2)	0.000 (2)
C24	0.146 (4)	0.114 (3)	0.137 (4)	−0.077 (3)	−0.020 (3)	0.051 (3)

Geometric parameters (Å, °)

Cl1—C1	1.729 (3)	C10—C11	1.384 (3)
Cl2—C3	1.720 (3)	C11—C12	1.360 (4)
N1—N2	1.356 (3)	C11—H11A	0.9300
N1—C10	1.358 (3)	C12—C13	1.393 (4)
N1—C9	1.438 (3)	C12—H12A	0.9300
N2—N3	1.298 (3)	C13—C14	1.354 (4)
N3—C15	1.379 (4)	C13—H13A	0.9300
O1—C7	1.194 (3)	C14—C15	1.399 (4)
O2—C7	1.349 (3)	C14—H14A	0.9300
O2—C8	1.432 (3)	C16—C17	1.479 (3)
O3—C16	1.213 (3)	C17—C18	1.384 (4)
C1—C6	1.368 (4)	C17—C22	1.388 (3)
C1—C2	1.366 (4)	C18—C19	1.368 (4)
C2—C3	1.376 (4)	C18—H18A	0.9300
C2—H2B	0.9300	C19—C20	1.372 (4)
C3—C4	1.389 (3)	C19—H19A	0.9300
C4—C5	1.378 (4)	C20—C21	1.376 (4)
C4—C7	1.482 (3)	C20—C23	1.506 (4)
C5—C6	1.365 (4)	C21—C22	1.378 (4)
C5—H5A	0.9300	C21—H21A	0.9300
C6—H6A	0.9300	C22—H22A	0.9300
C8—C9	1.511 (3)	C23—C24	1.471 (5)
C8—C16	1.517 (3)	C23—H23A	0.9700
C8—H8A	0.9800	C23—H23B	0.9700
C9—H9A	0.9700	C24—H24A	0.9600
C9—H9B	0.9700	C24—H24B	0.9600
C10—C15	1.379 (4)	C24—H24C	0.9600
?···?	?		
N2—N1—C10	109.9 (2)	C11—C12—C13	122.3 (3)
N2—N1—C9	119.9 (2)	C11—C12—H12A	118.9
C10—N1—C9	130.1 (2)	C13—C12—H12A	118.9
N3—N2—N1	109.1 (2)	C14—C13—C12	121.5 (3)
N2—N3—C15	107.8 (2)	C14—C13—H13A	119.2
C7—O2—C8	114.28 (19)	C12—C13—H13A	119.2
C6—C1—C2	121.2 (3)	C13—C14—C15	117.6 (3)
C6—C1—Cl1	120.3 (2)	C13—C14—H14A	121.2
C2—C1—Cl1	118.4 (2)	C15—C14—H14A	121.2
C1—C2—C3	119.2 (2)	N3—C15—C10	108.8 (2)

C1—C2—H2B	120.4	N3—C15—C14	131.5 (3)
C3—C2—H2B	120.4	C10—C15—C14	119.7 (3)
C2—C3—C4	121.0 (2)	O3—C16—C17	121.2 (2)
C2—C3—Cl2	116.5 (2)	O3—C16—C8	119.3 (2)
C4—C3—Cl2	122.5 (2)	C17—C16—C8	119.5 (2)
C5—C4—C3	117.6 (2)	C18—C17—C22	117.9 (2)
C5—C4—C7	119.7 (2)	C18—C17—C16	118.7 (2)
C3—C4—C7	122.7 (2)	C22—C17—C16	123.4 (2)
C6—C5—C4	122.1 (2)	C19—C18—C17	121.2 (3)
C6—C5—H5A	119.0	C19—C18—H18A	119.4
C4—C5—H5A	119.0	C17—C18—H18A	119.4
C5—C6—C1	118.9 (3)	C18—C19—C20	121.3 (3)
C5—C6—H6A	120.6	C18—C19—H19A	119.3
C1—C6—H6A	120.6	C20—C19—H19A	119.3
O1—C7—O2	122.2 (2)	C19—C20—C21	117.6 (3)
O1—C7—C4	126.3 (2)	C19—C20—C23	120.6 (3)
O2—C7—C4	111.5 (2)	C21—C20—C23	121.7 (3)
O2—C8—C9	105.67 (19)	C22—C21—C20	122.0 (3)
O2—C8—C16	109.74 (19)	C22—C21—H21A	119.0
C9—C8—C16	111.0 (2)	C20—C21—H21A	119.0
O2—C8—H8A	110.1	C21—C22—C17	119.9 (3)
C9—C8—H8A	110.1	C21—C22—H22A	120.0
C16—C8—H8A	110.1	C17—C22—H22A	120.0
N1—C9—C8	112.2 (2)	C24—C23—C20	113.1 (3)
N1—C9—H9A	109.2	C24—C23—H23A	109.0
C8—C9—H9A	109.2	C20—C23—H23A	109.0
N1—C9—H9B	109.2	C24—C23—H23B	109.0
C8—C9—H9B	109.2	C20—C23—H23B	109.0
H9A—C9—H9B	107.9	H23A—C23—H23B	107.8
N1—C10—C15	104.4 (2)	C23—C24—H24A	109.5
N1—C10—C11	132.5 (2)	C23—C24—H24B	109.5
C15—C10—C11	123.0 (3)	H24A—C24—H24B	109.5
C12—C11—C10	115.9 (3)	C23—C24—H24C	109.5
C12—C11—H11A	122.1	H24A—C24—H24C	109.5
C10—C11—H11A	122.1	H24B—C24—H24C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11A···O3^{i}	0.93	2.51	3.425 (3)	169
C12—H12A···O1^{ii}	0.93	2.52	3.378 (4)	154

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

Fig. 1

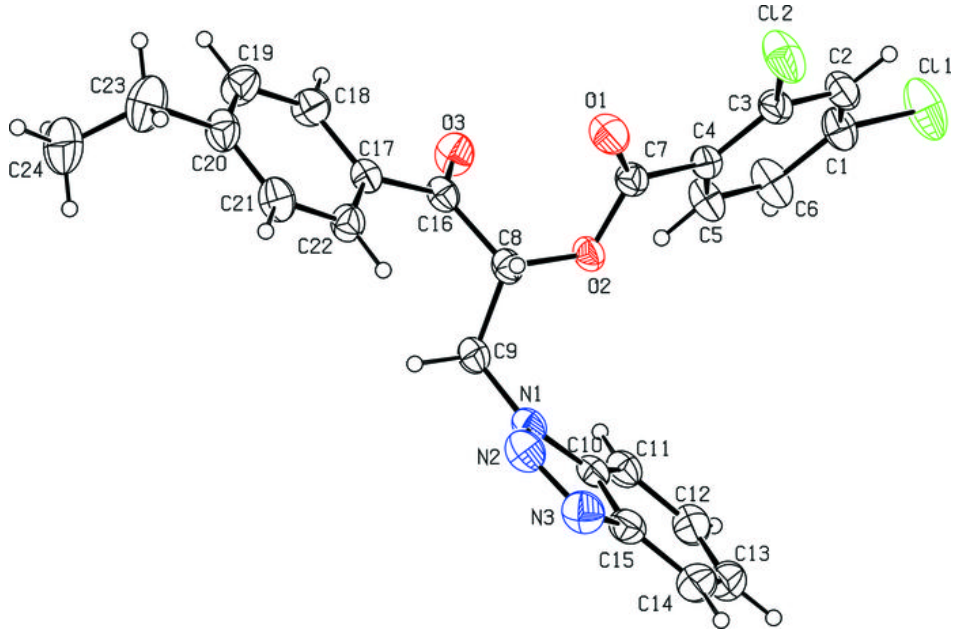
Cl2
C2
Cl1
C19
C23
C18
O1
C3
O3
C4
C7
C24
C20
C17
C1
C21
C16
C6
C22
C8
C5
O2
C9
N1
C10
C11
N2
C12
N3
C15
C13
C14

Fig. 2