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2-[2-(3-Chlorophenyl)hydrazinylidene]-1,3-diphenylpropane-1,3-dione

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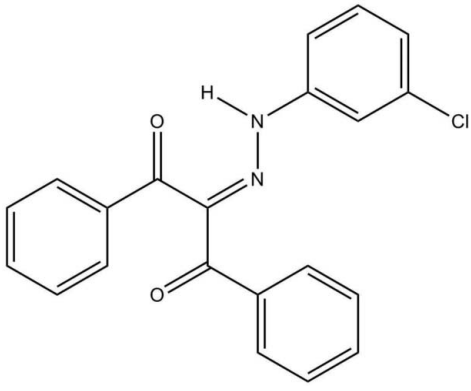
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.119; data-to-parameter ratio = 14.9.

The molecular structure of the title compound, $C_{21}H_{15}ClN_2O_2$, features one strong intramolecular N—H···O resonance-assisted hydrogen bond (RAHB). In the crystal, molecules form inversion-related dimers *via* pairs of weak intermolecular N—H···O contacts. These dimers are further stabilized *via* three weak C—H···O contacts, developing the three-dimensional structure.

Related literature

For resonance-assisted hydrogen bonds, see: Bertolasi *et al.* (1993, 1994*a*,*b*); Inabe (1991); Krygowski *et al.* (1997); Olivieri *et al.* (1989). For details of the synthesis, see: Bustos *et al.* (2007, 2009); Yao (1964).



Experimental

Crystal data

$C_{21}H_{15}ClN_2O_2$
M_r = 362.80
Monoclinic, $P2_1/n$
a = 10.2970 (9) Å
b = 10.3526 (9) Å
c = 16.8926 (15) Å
β = 102.131 (1)°
V = 1760.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 297 K
0.60 × 0.31 × 0.23 mm

Data collection

Bruker D8 Discover with SMART CCD area-detector diffractometer
11123 measured reflections
3573 independent reflections
2657 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.119
S = 0.98
3573 reflections
239 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.922 (17)	1.930 (18)	2.6205 (18)	130.1 (14)
C12—H12···N2	0.93	2.55	3.016 (2)	111
N1—H1···O1^{i}	0.922 (17)	2.628 (17)	3.344 (3)	135.0 (13)
C6—H6···O1^{i}	0.93	2.63	3.390 (2)	140

Symmetry code: (i) $-x, -y, -z+2$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2006).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2324).

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supplementary materials

Acta Cryst. (2011). E**67**, o1450-o1451 [**doi:10.1107/S1600536811017909**]

2-[2-(3-Chlorophenyl)hydrazinylidene]-1,3-diphenylpropane-1,3-dione

C. Bustos, L. Alvarez-Thon, J.-G. Cárcamo, A. Ibañez and C. Sánchez

Comment

In recent years, much attention has been devoted to structural studies on heterodienic systems forming strong intramolecular hydrogen bonds, N—H···O, assisted by resonance (RAHB) which, *inter alia*, could have potential technological applications as bistate molecular switches (Olivieri *et al.*, 1989; Inabe, 1991; Bertolasi *et al.*, 1993; Bertolasi *et al.*, 1994*a*; Bertolasi *et al.*, 1994*b*; Krygowski *et al.*, 1997; Bustos *et al.*, 2007). On the other hand, it is well known that the phenyl diazonium salts are capable of coupling with a series of β-diketones that may yield the N—H···O moiety (Yao, 1964; Bustos *et al.*, 2007; Bustos *et al.*, 2009). In this report we present the crystal and molecular structure of the title compound 2-(2-(3-chlorophenyl)hydrazono)-1,3-diphenylpropane-1,3-dione, of formula 3-Cl—C_6H_4—NHN=C(COC_6H_5)$_2$.

The molecular structure of the title compound exhibits one weak intramolecular hydrogen bond (C12–H12···N2) and one strong intramolecular hydrogen bond (N1–H1···O2) assisted by resonance (RAHB), see Fig. 1 and Table 2. The molecules form an inversion dimer *via* a pair of weak intermolecular contacts N1–H1···O1$^{\#}$ and C6–H6···O1$^{\#}$ hydrogen bonds [Symmetry code: (#) -*x*, -*y*, 2 - *z*], (see Fig. 2). These dimers are further stabilized *via* three weak contacts of the type shown in Fig. 3, to develop the three-dimensional structure.

Experimental

Chemicals: 1,3-diphenylpropane-1,3-dione, 3-chloroaniline and sodium nitrite were procured from Sigma-Aldrich, and sodium hydroxide, sodium acetate, solvents and hydrochloric acid from Merck. Chemicals were used without further purification.

Procedure: In a 500 ml beaker were dissolved 2.24 g (0.01 mole) of 1,3-diphenylpropane-1,3-dione in 100 ml of an ethanol solution containing 0.4 g (0.01 mole) of sodium hydroxide and 3.65 g of sodium acetate. The resulting β-diketonate solution was kept at -5°C and diluted with water to around 220 mL with vigorous stirring. In another beaker, a diazonium ion solution was prepared adding 1.06 ml (0.01 mole) of 3-chloroaniline (99%, density 1.215 g/ml) in 8 ml of hydrochloric acid (5 mol/*L*), cooling at -5 °C, and adding a saturated aqueous solution containing 0.69 g (0.01 mole) of sodium nitrite. This solution was then added dropwise, with vigorous stirring, into the β-diketonate solution. During the addition, a yellow solid precipitate was observed. This precipitate was filtered by suction and washed with an abundant quantity of water. Single crystals suitable for X-ray studies were obtained by recrystallization from a saturated solution of the compound in a 3:1 ethanol/acetone mixture.

Refinement

All hydrogen atoms were found in difference Fourier maps. The hydrogen attached to N1 was refined freely against the diffraction data, but all other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

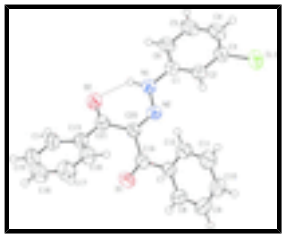

Fig. 1. View of the title compound showing displacement ellipsoids for non-H atoms at the 50% probability level. The strong intramolecular hydrogen bond (N1-H1···O2) assisted by resonance (RAHB) is depicted by a dashed line.

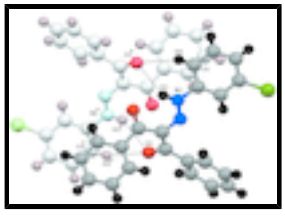

Fig. 2. View of the formation of an inversion dimer *via* two weak intermolecular contacts (dashed lines). [Symmetry code: (#) -*x*, -*y*, 2 - *z*].

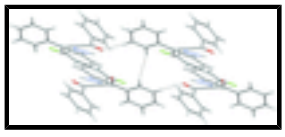

Fig. 3. The dimers are connected *via* three weak contacts (dashed lines) to develop the three-dimensional structure.

2-[2-(3-Chlorophenyl)hydrazinylidene]-1,3-diphenylpropane-1,3-dione

Crystal data

$C_{21}H_{15}ClN_2O_2$	$F(000) = 752$
$M_r = 362.80$	$D_x = 1.369$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 999 reflections
$a = 10.2970$ (9) Å	$\theta = 2.3$–$26.3°$
$b = 10.3526$ (9) Å	$\mu = 0.24$ mm^{-1}
$c = 16.8926$ (15) Å	$T = 297$ K
$\beta = 102.131$ (1)°	Polyhedron, yellow
$V = 1760.6$ (3) Å^3	0.60 × 0.31 × 0.23 mm
$Z = 4$	

Data collection

Bruker D8 Discover with SMART CCD area-detector diffractometer	2657 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.042$
graphite	$\theta_{max} = 26.3°$, $\theta_{min} = 2.3°$
φ and ω scans	$h = -12 \rightarrow 12$
11123 measured reflections	$k = -12 \rightarrow 12$
3573 independent reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.042$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.119$	H atoms treated by a mixture of independent and constrained refinement
$S = 0.98$	$w = 1/[\sigma^2(F_o^2) + (0.071P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
3573 reflections	$(\Delta/\sigma)_{max} = 0.001$
239 parameters	$\Delta\rho_{max} = 0.39$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.16$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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	0.69570 (5)	0.13603 (5)	1.18232 (3)	0.0776 (2)
O1	−0.05070 (10)	0.25054 (10)	0.91373 (7)	0.0598 (4)
O2	0.03738 (11)	−0.12050 (10)	0.92207 (7)	0.0630 (4)
N1	0.25273 (13)	−0.04080 (12)	1.02183 (8)	0.0510 (4)
N2	0.22035 (12)	0.07588 (11)	0.99272 (7)	0.0463 (4)
C1	0.36632 (15)	−0.05568 (14)	1.08450 (9)	0.0459 (5)
C2	0.46505 (14)	0.03750 (14)	1.09893 (9)	0.0477 (5)
C3	0.57101 (15)	0.01951 (15)	1.16316 (9)	0.0516 (5)
C4	0.58144 (17)	−0.08754 (18)	1.21159 (10)	0.0636 (6)
C5	0.48391 (18)	−0.18002 (18)	1.19538 (11)	0.0696 (7)
C6	0.37597 (17)	−0.16608 (16)	1.13176 (11)	0.0616 (6)
C7	0.16409 (16)	0.33878 (14)	0.92641 (8)	0.0466 (5)
C8	0.11665 (19)	0.46296 (15)	0.93282 (9)	0.0594 (6)
C9	0.1988 (2)	0.56842 (17)	0.93377 (11)	0.0743 (8)
C10	0.3278 (2)	0.55061 (18)	0.92704 (13)	0.0810 (8)
C11	0.3759 (2)	0.42915 (18)	0.91881 (12)	0.0752 (7)
C12	0.29480 (17)	0.32324 (16)	0.91906 (10)	0.0584 (6)

C13	−0.05962 (15)	0.01258 (14)	0.81261 (9)	0.0489 (5)
C14	−0.17413 (16)	−0.06169 (16)	0.79092 (10)	0.0580 (6)
C15	−0.25909 (19)	−0.0430 (2)	0.71700 (12)	0.0768 (7)
C16	−0.2295 (2)	0.0471 (2)	0.66386 (12)	0.0840 (8)
C17	−0.1161 (2)	0.1191 (2)	0.68373 (11)	0.0787 (8)
C18	−0.02983 (18)	0.10304 (17)	0.75838 (10)	0.0632 (6)
C19	0.06905 (15)	0.23015 (14)	0.92398 (8)	0.0466 (5)
C20	0.11471 (14)	0.09339 (14)	0.93512 (9)	0.0457 (5)
C21	0.02976 (14)	−0.01183 (15)	0.89255 (9)	0.0482 (5)
H1	0.1934 (18)	−0.1079 (15)	1.0080 (10)	0.062 (5)*
H2	0.46010	0.11030	1.06620	0.0570*
H4	0.65340	−0.09750	1.25480	0.0760*
H5	0.49060	−0.25330	1.22780	0.0840*
H6	0.31090	−0.22970	1.12080	0.0740*
H8	0.02860	0.47520	0.93650	0.0710*
H9	0.16670	0.65130	0.93900	0.0890*
H10	0.38330	0.62170	0.92810	0.0970*
H11	0.46310	0.41810	0.91310	0.0900*
H12	0.32800	0.24070	0.91430	0.0700*
H14	−0.19350	−0.12410	0.82630	0.0700*
H15	−0.33650	−0.09150	0.70320	0.0920*
H16	−0.28700	0.05920	0.61400	0.1010*
H17	−0.09640	0.17930	0.64710	0.0940*
H18	0.04710	0.15240	0.77180	0.0760*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0598 (3)	0.0744 (3)	0.0886 (4)	−0.0128 (2)	−0.0070 (3)	−0.0046 (2)
O1	0.0440 (7)	0.0589 (7)	0.0723 (7)	0.0061 (5)	0.0030 (5)	0.0073 (5)
O2	0.0609 (7)	0.0489 (7)	0.0717 (8)	−0.0080 (5)	−0.0030 (6)	0.0076 (6)
N1	0.0465 (7)	0.0428 (7)	0.0574 (8)	0.0001 (6)	−0.0032 (6)	0.0035 (6)
N2	0.0444 (7)	0.0431 (7)	0.0488 (7)	0.0025 (5)	0.0041 (6)	0.0035 (5)
C1	0.0420 (8)	0.0442 (8)	0.0497 (8)	0.0059 (6)	0.0057 (6)	0.0021 (6)
C2	0.0480 (9)	0.0454 (8)	0.0476 (8)	0.0038 (7)	0.0055 (7)	0.0041 (7)
C3	0.0466 (9)	0.0535 (9)	0.0522 (9)	0.0017 (7)	0.0048 (7)	−0.0048 (7)
C4	0.0539 (10)	0.0756 (12)	0.0559 (10)	0.0080 (9)	−0.0007 (8)	0.0142 (9)
C5	0.0577 (11)	0.0677 (11)	0.0798 (12)	0.0069 (9)	0.0060 (9)	0.0309 (10)
C6	0.0514 (9)	0.0528 (9)	0.0769 (12)	0.0008 (8)	0.0049 (8)	0.0174 (8)
C7	0.0525 (9)	0.0449 (8)	0.0385 (8)	0.0011 (7)	0.0005 (6)	0.0010 (6)
C8	0.0708 (11)	0.0498 (9)	0.0534 (9)	0.0075 (8)	0.0036 (8)	−0.0013 (7)
C9	0.1002 (17)	0.0449 (10)	0.0701 (12)	0.0008 (10)	0.0002 (11)	−0.0019 (8)
C10	0.0874 (16)	0.0552 (12)	0.0907 (15)	−0.0228 (10)	−0.0031 (12)	0.0091 (10)
C11	0.0633 (12)	0.0671 (12)	0.0928 (14)	−0.0129 (9)	0.0112 (10)	0.0116 (10)
C12	0.0560 (10)	0.0485 (9)	0.0690 (11)	−0.0021 (8)	0.0095 (8)	0.0032 (8)
C13	0.0444 (8)	0.0509 (9)	0.0495 (9)	0.0012 (7)	0.0059 (7)	−0.0072 (7)
C14	0.0531 (10)	0.0561 (10)	0.0618 (10)	−0.0040 (7)	0.0051 (8)	−0.0151 (8)
C15	0.0591 (11)	0.0872 (14)	0.0755 (13)	−0.0045 (10)	−0.0051 (10)	−0.0279 (12)

C16	0.0810 (15)	0.1068 (17)	0.0526 (11)	0.0086 (13)	−0.0125 (10)	−0.0128 (11)
C17	0.0879 (15)	0.0923 (15)	0.0525 (11)	0.0040 (12)	0.0071 (10)	0.0058 (10)
C18	0.0635 (11)	0.0705 (11)	0.0536 (10)	−0.0049 (9)	0.0079 (8)	0.0025 (8)
C19	0.0468 (9)	0.0509 (9)	0.0390 (8)	0.0024 (7)	0.0020 (6)	0.0035 (6)
C20	0.0424 (8)	0.0456 (8)	0.0464 (8)	−0.0002 (6)	0.0029 (6)	0.0033 (6)
C21	0.0429 (9)	0.0468 (9)	0.0540 (9)	−0.0015 (7)	0.0081 (7)	0.0001 (7)

Geometric parameters (Å, °)

Cl1—C3	1.7418 (17)	C13—C21	1.487 (2)
O1—C19	1.2269 (19)	C14—C15	1.380 (3)
O2—C21	1.2264 (19)	C15—C16	1.373 (3)
N1—N2	1.3203 (17)	C16—C17	1.366 (3)
N1—C1	1.411 (2)	C17—C18	1.392 (3)
N2—C20	1.3107 (19)	C19—C20	1.491 (2)
N1—H1	0.922 (17)	C20—C21	1.485 (2)
C1—C6	1.386 (2)	C2—H2	0.9300
C1—C2	1.385 (2)	C4—H4	0.9300
C2—C3	1.380 (2)	C5—H5	0.9300
C3—C4	1.368 (2)	C6—H6	0.9300
C4—C5	1.373 (3)	C8—H8	0.9300
C5—C6	1.382 (3)	C9—H9	0.9300
C7—C8	1.388 (2)	C10—H10	0.9300
C7—C19	1.486 (2)	C11—H11	0.9300
C7—C12	1.387 (2)	C12—H12	0.9300
C8—C9	1.379 (3)	C14—H14	0.9300
C9—C10	1.369 (3)	C15—H15	0.9300
C10—C11	1.369 (3)	C16—H16	0.9300
C11—C12	1.379 (3)	C17—H17	0.9300
C13—C14	1.391 (2)	C18—H18	0.9300
C13—C18	1.389 (2)		
Cl1···C21^{i}	3.5703 (16)	C21···Cl1^{i}	3.5703 (16)
Cl1···H10^{ii}	3.1300	C4···H12^{i}	2.9600
O1···C13	2.9882 (18)	C5···H12^{i}	3.0100
O1···C18	3.083 (2)	C8···H16^{vii}	3.0200
O1···O2^{iii}	3.0602 (16)	C8···H8^{viii}	2.9800
O1···C15^{iv}	3.386 (2)	C9···H4^{ix}	2.9800
O1···C6^{iii}	3.390 (2)	C10···H11^{ii}	3.1000
O2···O1^{iii}	3.0602 (16)	C14···H5^{x}	2.9100
O2···N1	2.6205 (18)	C15···H6^{x}	3.0300
O2···N2	2.8570 (16)	C19···H18	2.6600
O2···C19^{iii}	3.2328 (18)	C20···H12	2.7600
O2···C20^{iii}	3.1526 (19)	C20···H18	2.7700
O1···H15^{iv}	2.6400	C21···H1	2.500 (17)
O1···H1^{iii}	2.628 (17)	H1···O2	1.930 (18)
O1···H6^{iii}	2.6300	H1···C21	2.500 (17)

O1···H8	2.4700	H1···H6	2.3900
O2···H1	1.930 (18)	H1···O1^{iii}	2.628 (17)
O2···H9^{v}	2.7000	H2···N2	2.5400
O2···H14	2.5800	H4···C9^{vii}	2.9800
N1···O2	2.6205 (18)	H5···C14^{xi}	2.9100
N2···O2	2.8570 (16)	H6···H1	2.3900
N2···C12	3.016 (2)	H6···O1^{iii}	2.6300
N2···H2	2.5400	H6···C15^{xi}	3.0300
N2···H12	2.5500	H8···O1	2.4700
C1···C14^{iii}	3.402 (2)	H8···C8^{viii}	2.9800
C6···C14^{iii}	3.567 (2)	H8···H8^{viii}	2.4000
C6···O1^{iii}	3.390 (2)	H9···O2^{xii}	2.7000
C10···C11^{ii}	3.577 (3)	H10···Cl1^{ii}	3.1300
C11···C10^{ii}	3.577 (3)	H11···C10^{ii}	3.1000
C12···N2	3.016 (2)	H12···N2	2.5500
C13···O1	2.9882 (18)	H12···C20	2.7600
C14···C1^{iii}	3.402 (2)	H12···C4^{i}	2.9600
C14···C6^{iii}	3.567 (2)	H12···C5^{i}	3.0100
C15···O1^{vi}	3.386 (2)	H14···O2	2.5800
C18···C19	3.065 (2)	H15···O1^{vi}	2.6400
C18···O1	3.083 (2)	H16···C8^{ix}	3.0200
C19···O2^{iii}	3.2328 (18)	H18···C19	2.6600
C19···C18	3.065 (2)	H18···C20	2.7700
C20···O2^{iii}	3.1526 (19)		
N2—N1—C1	118.94 (12)	C19—C20—C21	119.83 (13)
N1—N2—C20	120.55 (12)	N2—C20—C19	114.55 (13)
N2—N1—H1	119.5 (11)	C13—C21—C20	120.28 (13)
C1—N1—H1	120.6 (11)	O2—C21—C13	119.97 (14)
N1—C1—C2	121.18 (13)	O2—C21—C20	119.69 (13)
N1—C1—C6	118.03 (14)	C1—C2—H2	121.00
C2—C1—C6	120.79 (15)	C3—C2—H2	121.00
C1—C2—C3	118.30 (14)	C3—C4—H4	121.00
C2—C3—C4	121.95 (15)	C5—C4—H4	121.00
Cl1—C3—C4	119.19 (12)	C4—C5—H5	119.00
Cl1—C3—C2	118.86 (12)	C6—C5—H5	119.00
C3—C4—C5	118.91 (16)	C1—C6—H6	121.00
C4—C5—C6	121.16 (17)	C5—C6—H6	121.00
C1—C6—C5	118.87 (16)	C7—C8—H8	120.00
C12—C7—C19	123.77 (14)	C9—C8—H8	120.00
C8—C7—C12	118.60 (15)	C8—C9—H9	120.00
C8—C7—C19	117.57 (15)	C10—C9—H9	120.00
C7—C8—C9	120.61 (18)	C9—C10—H10	120.00
C8—C9—C10	119.72 (17)	C11—C10—H10	120.00
C9—C10—C11	120.67 (18)	C10—C11—H11	120.00
C10—C11—C12	119.85 (19)	C12—C11—H11	120.00

C7—C12—C11	120.52 (16)	C7—C12—H12	120.00
C14—C13—C21	118.29 (14)	C11—C12—H12	120.00
C14—C13—C18	119.45 (15)	C13—C14—H14	120.00
C18—C13—C21	122.22 (14)	C15—C14—H14	120.00
C13—C14—C15	120.15 (16)	C14—C15—H15	120.00
C14—C15—C16	120.11 (18)	C16—C15—H15	120.00
C15—C16—C17	120.38 (19)	C15—C16—H16	120.00
C16—C17—C18	120.48 (18)	C17—C16—H16	120.00
C13—C18—C17	119.41 (17)	C16—C17—H17	120.00
O1—C19—C20	117.52 (13)	C18—C17—H17	120.00
O1—C19—C7	120.69 (13)	C13—C18—H18	120.00
C7—C19—C20	121.79 (13)	C17—C18—H18	120.00
N2—C20—C21	124.79 (13)		
C1—N1—N2—C20	−179.27 (14)	C8—C9—C10—C11	0.5 (3)
N2—N1—C1—C2	−20.0 (2)	C9—C10—C11—C12	−1.4 (3)
N2—N1—C1—C6	159.53 (14)	C10—C11—C12—C7	0.9 (3)
N1—N2—C20—C19	163.92 (13)	C18—C13—C14—C15	−1.8 (2)
N1—N2—C20—C21	−5.6 (2)	C21—C13—C18—C17	178.47 (16)
N1—C1—C6—C5	−177.52 (15)	C14—C13—C21—O2	30.3 (2)
C2—C1—C6—C5	2.0 (2)	C14—C13—C21—C20	−152.55 (15)
N1—C1—C2—C3	177.48 (14)	C18—C13—C21—O2	−147.25 (16)
C6—C1—C2—C3	−2.1 (2)	C18—C13—C21—C20	29.9 (2)
C1—C2—C3—Cl1	−179.79 (12)	C21—C13—C14—C15	−179.39 (16)
C1—C2—C3—C4	0.8 (2)	C14—C13—C18—C17	0.9 (3)
Cl1—C3—C4—C5	−178.97 (14)	C13—C14—C15—C16	1.4 (3)
C2—C3—C4—C5	0.4 (3)	C14—C15—C16—C17	−0.2 (3)
C3—C4—C5—C6	−0.5 (3)	C15—C16—C17—C18	−0.7 (3)
C4—C5—C6—C1	−0.8 (3)	C16—C17—C18—C13	0.3 (3)
C19—C7—C8—C9	−178.70 (14)	O1—C19—C20—N2	−136.93 (14)
C8—C7—C12—C11	0.5 (2)	C7—C19—C20—C21	−147.85 (13)
C8—C7—C19—O1	12.4 (2)	O1—C19—C20—C21	33.1 (2)
C8—C7—C19—C20	−166.55 (13)	C7—C19—C20—N2	42.09 (19)
C19—C7—C12—C11	177.53 (15)	N2—C20—C21—O2	16.7 (2)
C12—C7—C8—C9	−1.5 (2)	N2—C20—C21—C13	−160.48 (14)
C12—C7—C19—C20	16.4 (2)	C19—C20—C21—O2	−152.33 (14)
C12—C7—C19—O1	−164.61 (14)	C19—C20—C21—C13	30.5 (2)
C7—C8—C9—C10	1.0 (3)		

Symmetry codes: (i) −*x*+1, −*y*, −*z*+2; (ii) −*x*+1, −*y*+1, −*z*+2; (iii) −*x*, −*y*, −*z*+2; (iv) −*x*−1/2, *y*+1/2, −*z*+3/2; (v) *x*, *y*−1, *z*; (vi) −*x*−1/2, *y*−1/2, −*z*+3/2; (vii) *x*+1/2, −*y*+1/2, *z*+1/2; (viii) −*x*, −*y*+1, −*z*+2; (ix) *x*−1/2, −*y*+1/2, *z*−1/2; (x) *x*−1/2, −*y*−1/2, *z*−1/2; (xi) *x*+1/2, −*y*−1/2, *z*+1/2; (xii) *x*, *y*+1, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.922 (17)	1.930 (18)	2.6205 (18)	130.1 (14)
C12—H12···N2	0.93	2.55	3.016 (2)	111
N1—H1···O1^{iii}	0.922 (17)	2.628 (17)	3.3435 (27)	135.0 (13)
C6—H6···O1^{iii}	0.93	2.63	3.390 (2)	140

Symmetry codes: (iii) −*x*, −*y*, −*z*+2.

Fig. 1

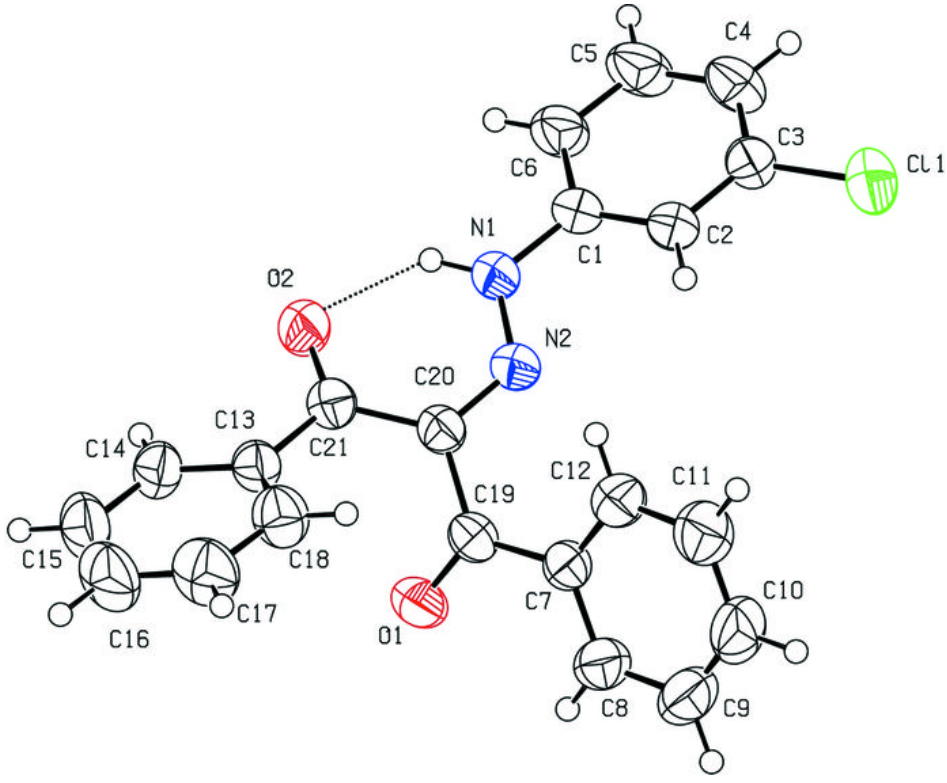
C5
C4
C6
C3
Cl1
N1
C1
C2
O2
N2
C20
C13
C21
C14
C12
C11
C19
C15
C18
C7
C10
C17
O1
C16
C8
C9

Fig. 2

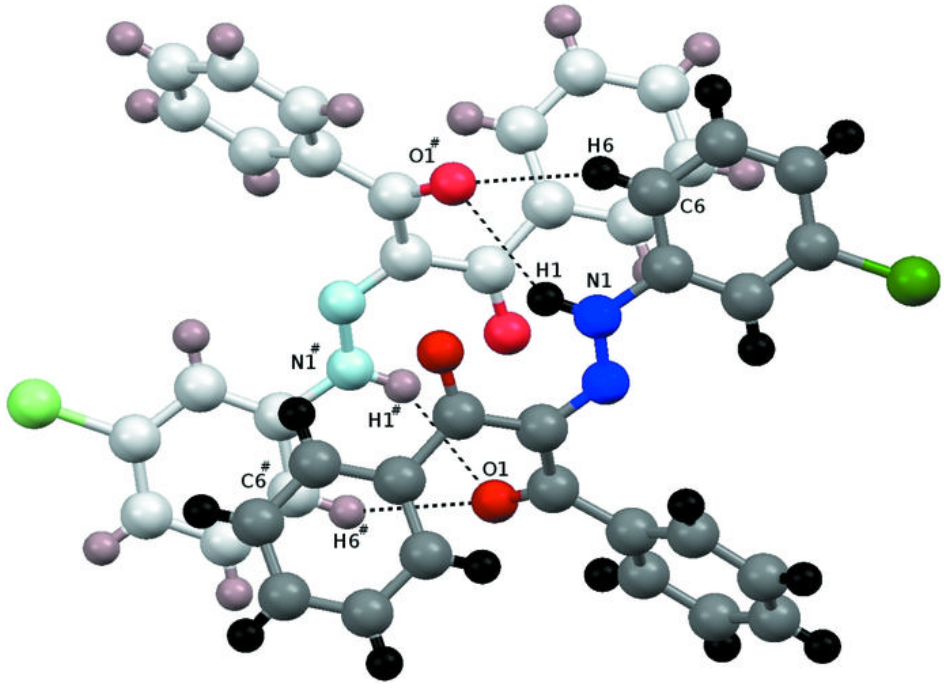

Fig. 3