organic compounds

1-(4-Chlorophenyl)-3-(2-nitrophenyl)-propane-1,2-dione

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The title compound, $C_{15}H_{10}ClNO_4$, belongs to the class of 1,2-diketones, which have important applications in both synthetic organic chemistry and supramolecular chemistry. A dihedral angle of 9.03 (1)° is found between the mean planes of the two benzene rings. C—H⋯O interactions help to stabilize the crystal structure.

Related literature

For the synthesis of the title compound, see: Barnes & Gist (1950). For applications of the title compound, see: Saalfrank *et al.* (1988); Schobert (1988); van Leusen & van Leusen (1977).
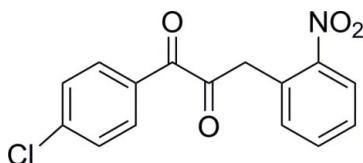
Experimental

Crystal data

$C_{15}H_{10}ClNO_4$

M_r = 303.69

Monoclinic, $P2_1/c$

a = 7.9995 (3) Å

b = 6.3730 (2) Å

c = 26.7750 (5) Å

β = 91.340 (2)°

V = 1364.64 (7) Å^3

Z = 4

Mo $K\alpha$ radiation

μ = 0.29 mm^{-1}

T = 298 (2) K

$0.20 \times 0.10 \times 0.10$ mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer

Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)

T_{min} = 0.943, T_{max} = 0.971

8762 measured reflections

2408 independent reflections

1872 reflections with $I > 2\sigma(I)$

R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047

$wR(F^2)$ = 0.133

S = 1.07

2408 reflections

190 parameters

H-atom parameters constrained

$\Delta\rho_{max}$ = 0.38 e Å^{-3}

$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C5—H5⋯O3^i	0.93	2.53	3.279 (3)	138
C15—H15⋯O1	0.93	2.59	3.244 (3)	128

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

References

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supplementary materials

1-(4-Chlorophenyl)-3-(2-nitrophenyl)propane-1,2-dione

L. Huang, S. Li and H. Li

Comment

1,2-Diketones are important intermediates in the preparation of heterocyclic compounds in synthetic chemistry (van Leusen & van Leusen, 1977; Saalfrank *et al.*, 1988). In addition, they can also be used as ligands in supramolecular chemistry (Schobert, 1988). The molecular structure of the title compound is shown in Fig. 1. The dihedral angle of the two benzene rings is 9.03 (1)°. Molecules are mainly connected by an intermolecular C—H···O interaction.

Experimental

The title compound was synthesized as previously described by Barnes & Gist (1950). Colorless crystals suitable for X-ray data collection were obtained by slow evaporation of a 5:2 ratio CH_2Cl_2:MeOH solution at 293 K.

Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding model, with $U_{iso}(H) = 1.2$ $U_{eq}(C)$ ($1.5U_{eq}(C)$ for methyl) of the parent atoms.

Figures
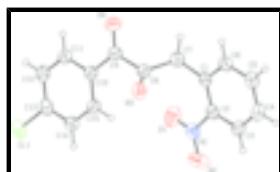
Fig. 1. View of the title molecule showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by spheres of arbitrary radius.

1-(4-Chlorophenyl)-3-(2-nitrophenyl)propane-1,2-dione

Crystal data

$C_{15}H_{10}ClNO_4$	$F_{000} = 624$
$M_r = 303.69$	$D_x = 1.478$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2433 reflections
$a = 7.9995$ (3) Å	$\theta = 2.6–23.4°$
$b = 6.3730$ (2) Å	$\mu = 0.30$ mm^{-1}
$c = 26.7750$ (5) Å	$T = 298$ (2) K
$\beta = 91.340$ (2)°	Block, colourless
$V = 1364.64$ (7) Å^3	$0.20 \times 0.10 \times 0.10$ mm

$Z = 4$

Data collection

Bruker SMART 4K CCD area-detector diffractometer	2408 independent reflections
Radiation source: fine-focus sealed tube	1872 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.027$
$T = 298(2)$ K	$\theta_{max} = 25.0°$
φ and ω scans	$\theta_{min} = 2.6°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$h = -9 \rightarrow 9$
$T_{min} = 0.943$, $T_{max} = 0.971$	$k = -7 \rightarrow 7$
8762 measured reflections	$l = -31 \rightarrow 31$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.133$	$w = 1/[\sigma^2(F_o^2) + (0.0688P)^2 + 0.2924P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
2408 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
190 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.4590 (2)	0.5187 (3)	0.67527 (7)	0.0496 (5)
C2	0.4028 (3)	0.7133 (3)	0.65828 (7)	0.0498 (5)
C3	0.2727 (3)	0.8197 (4)	0.68005 (9)	0.0606 (6)
H3	0.2382	0.9492	0.6676	0.073*
C4	0.1952 (3)	0.7326 (5)	0.72019 (9)	0.0675 (7)
H4	0.1075	0.8028	0.7351	0.081*
C5	0.2471 (3)	0.5421 (4)	0.73831 (9)	0.0669 (7)
H5	0.1947	0.4829	0.7656	0.080*
C6	0.3771 (3)	0.4379 (4)	0.71616 (8)	0.0588 (6)
H6	0.4111	0.3089	0.7291	0.071*
C7	0.6000 (3)	0.3935 (4)	0.65391 (9)	0.0602 (6)
H7A	0.6018	0.2561	0.6695	0.072*
H7B	0.5777	0.3735	0.6185	0.072*
C8	0.7691 (3)	0.4913 (3)	0.66072 (7)	0.0504 (5)
C9	0.9136 (3)	0.3769 (4)	0.63586 (8)	0.0539 (5)
C10	1.0144 (2)	0.4879 (3)	0.59876 (7)	0.0471 (5)

C11	1.1423 (3)	0.3809 (4)	0.57538 (8)	0.0568 (6)
H11	1.1665	0.2433	0.5846	0.068*
C12	1.2338 (3)	0.4757 (4)	0.53865 (8)	0.0631 (6)
H12	1.3183	0.4029	0.5228	0.076*
C13	1.1976 (3)	0.6808 (4)	0.52582 (8)	0.0571 (6)
C14	1.0729 (3)	0.7893 (4)	0.54892 (8)	0.0572 (6)
H14	1.0505	0.9278	0.5401	0.069*
C15	0.9814 (3)	0.6933 (3)	0.58507 (8)	0.0536 (5)
H15	0.8965	0.7669	0.6005	0.064*
Cl1	1.31213 (9)	0.80635 (13)	0.48046 (2)	0.0813 (3)
N1	0.4783 (3)	0.8163 (4)	0.61492 (7)	0.0653 (6)
O1	0.5769 (2)	0.7162 (4)	0.58971 (7)	0.0969 (7)
O2	0.4318 (3)	0.9934 (3)	0.60410 (8)	0.0994 (7)
O3	0.79710 (19)	0.6406 (3)	0.68656 (6)	0.0693 (5)
O4	0.9394 (2)	0.1971 (3)	0.64872 (8)	0.0855 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0428 (11)	0.0539 (13)	0.0520 (11)	−0.0072 (9)	−0.0024 (9)	−0.0057 (10)
C2	0.0441 (11)	0.0588 (14)	0.0463 (11)	−0.0084 (10)	−0.0063 (9)	−0.0001 (10)
C3	0.0509 (13)	0.0614 (15)	0.0687 (15)	0.0020 (11)	−0.0109 (11)	−0.0052 (11)
C4	0.0447 (13)	0.0881 (19)	0.0697 (15)	0.0040 (12)	0.0022 (11)	−0.0143 (14)
C5	0.0516 (14)	0.0881 (19)	0.0614 (14)	−0.0112 (13)	0.0082 (11)	0.0013 (13)
C6	0.0538 (13)	0.0602 (14)	0.0623 (13)	−0.0091 (11)	−0.0032 (10)	0.0065 (11)
C7	0.0574 (14)	0.0606 (14)	0.0627 (13)	−0.0031 (11)	0.0040 (10)	−0.0117 (11)
C8	0.0533 (13)	0.0521 (13)	0.0461 (11)	0.0014 (10)	0.0050 (9)	0.0032 (10)
C9	0.0544 (13)	0.0497 (13)	0.0579 (12)	0.0040 (10)	0.0038 (10)	0.0016 (10)
C10	0.0448 (11)	0.0478 (12)	0.0485 (11)	0.0002 (9)	−0.0008 (9)	−0.0050 (9)
C11	0.0542 (13)	0.0491 (13)	0.0676 (14)	0.0044 (10)	0.0066 (10)	−0.0056 (10)
C12	0.0537 (14)	0.0705 (17)	0.0655 (14)	0.0015 (12)	0.0141 (11)	−0.0154 (12)
C13	0.0509 (13)	0.0724 (17)	0.0479 (12)	−0.0099 (11)	0.0004 (10)	−0.0030 (10)
C14	0.0580 (13)	0.0548 (13)	0.0589 (13)	−0.0014 (11)	0.0016 (10)	0.0049 (10)
C15	0.0492 (12)	0.0549 (14)	0.0568 (12)	0.0055 (10)	0.0047 (9)	0.0001 (10)
Cl1	0.0776 (5)	0.1050 (6)	0.0619 (4)	−0.0200 (4)	0.0179 (3)	0.0070 (3)
N1	0.0593 (12)	0.0772 (16)	0.0588 (12)	−0.0135 (11)	−0.0082 (10)	0.0125 (11)
O1	0.0753 (13)	0.143 (2)	0.0732 (13)	0.0231 (13)	0.0198 (10)	0.0355 (12)
O2	0.140 (2)	0.0620 (13)	0.0966 (14)	−0.0183 (13)	0.0092 (13)	0.0166 (11)
O3	0.0546 (9)	0.0763 (11)	0.0774 (11)	−0.0077 (8)	0.0083 (8)	−0.0257 (9)
O4	0.0905 (14)	0.0608 (12)	0.1068 (14)	0.0187 (10)	0.0394 (11)	0.0232 (10)

Geometric parameters (Å, °)

C1—C6	1.388 (3)	C8—C9	1.532 (3)
C1—C2	1.392 (3)	C9—O4	1.213 (3)
C1—C7	1.505 (3)	C9—C10	1.475 (3)
C2—C3	1.382 (3)	C10—C15	1.383 (3)
C2—N1	1.475 (3)	C10—C11	1.390 (3)
C3—C4	1.371 (3)	C11—C12	1.379 (3)

C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.368 (4)	C12—C13	1.380 (3)
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.380 (3)	C13—C14	1.373 (3)
C5—H5	0.9300	C13—Cl1	1.734 (2)
C6—H6	0.9300	C14—C15	1.371 (3)
C7—C8	1.497 (3)	C14—H14	0.9300
C7—H7A	0.9700	C15—H15	0.9300
C7—H7B	0.9700	N1—O2	1.221 (3)
C8—O3	1.195 (3)	N1—O1	1.229 (3)
C6—C1—C2	115.7 (2)	C7—C8—C9	115.99 (19)
C6—C1—C7	118.3 (2)	O4—C9—C10	123.5 (2)
C2—C1—C7	126.0 (2)	O4—C9—C8	116.8 (2)
C3—C2—C1	122.7 (2)	C10—C9—C8	119.63 (19)
C3—C2—N1	116.1 (2)	C15—C10—C11	118.9 (2)
C1—C2—N1	121.2 (2)	C15—C10—C9	121.91 (19)
C4—C3—C2	119.4 (2)	C11—C10—C9	119.2 (2)
C4—C3—H3	120.3	C12—C11—C10	120.9 (2)
C2—C3—H3	120.3	C12—C11—H11	119.5
C5—C4—C3	119.9 (2)	C10—C11—H11	119.5
C5—C4—H4	120.1	C11—C12—C13	118.7 (2)
C3—C4—H4	120.1	C11—C12—H12	120.6
C4—C5—C6	120.0 (2)	C13—C12—H12	120.6
C4—C5—H5	120.0	C14—C13—C12	121.0 (2)
C6—C5—H5	120.0	C14—C13—Cl1	119.0 (2)
C5—C6—C1	122.3 (2)	C12—C13—Cl1	119.99 (18)
C5—C6—H6	118.8	C15—C14—C13	119.9 (2)
C1—C6—H6	118.8	C15—C14—H14	120.0
C8—C7—C1	114.60 (19)	C13—C14—H14	120.0
C8—C7—H7A	108.6	C14—C15—C10	120.5 (2)
C1—C7—H7A	108.6	C14—C15—H15	119.7
C8—C7—H7B	108.6	C10—C15—H15	119.7
C1—C7—H7B	108.6	O2—N1—O1	123.0 (2)
H7A—C7—H7B	107.6	O2—N1—C2	118.0 (2)
O3—C8—C7	123.96 (19)	O1—N1—C2	118.8 (2)
O3—C8—C9	119.84 (19)		
C6—C1—C2—C3	−0.7 (3)	O4—C9—C10—C15	−179.9 (2)
C7—C1—C2—C3	−179.6 (2)	C8—C9—C10—C15	−0.7 (3)
C6—C1—C2—N1	−179.61 (18)	O4—C9—C10—C11	2.6 (3)
C7—C1—C2—N1	1.4 (3)	C8—C9—C10—C11	−178.20 (19)
C1—C2—C3—C4	0.3 (3)	C15—C10—C11—C12	−0.9 (3)
N1—C2—C3—C4	179.30 (19)	C9—C10—C11—C12	176.7 (2)
C2—C3—C4—C5	0.1 (3)	C10—C11—C12—C13	0.8 (3)
C3—C4—C5—C6	−0.1 (4)	C11—C12—C13—C14	−0.1 (3)
C4—C5—C6—C1	−0.3 (4)	C11—C12—C13—Cl1	179.04 (17)
C2—C1—C6—C5	0.7 (3)	C12—C13—C14—C15	−0.5 (3)
C7—C1—C6—C5	179.7 (2)	Cl1—C13—C14—C15	−179.67 (17)
C6—C1—C7—C8	−112.2 (2)	C13—C14—C15—C10	0.4 (3)

C2—C1—C7—C8	66.7 (3)	C11—C10—C15—C14	0.2 (3)
C1—C7—C8—O3	9.9 (3)	C9—C10—C15—C14	−177.2 (2)
C1—C7—C8—C9	−175.36 (19)	C3—C2—N1—O2	6.2 (3)
O3—C8—C9—O4	115.7 (3)	C1—C2—N1—O2	−174.8 (2)
C7—C8—C9—O4	−59.3 (3)	C3—C2—N1—O1	−169.5 (2)
O3—C8—C9—C10	−63.5 (3)	C1—C2—N1—O1	9.5 (3)
C7—C8—C9—C10	121.5 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O3^i	0.93	2.53	3.279 (3)	138
C15—H15···O1	0.93	2.59	3.244 (3)	128

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

Fig. 1