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(*Z*)-Ethyl 2-[2-(3-bromophenyl)hydrazono]-4-chloro-3-oxobutanoate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.032
wR factor = 0.070
Data-to-parameter ratio = 15.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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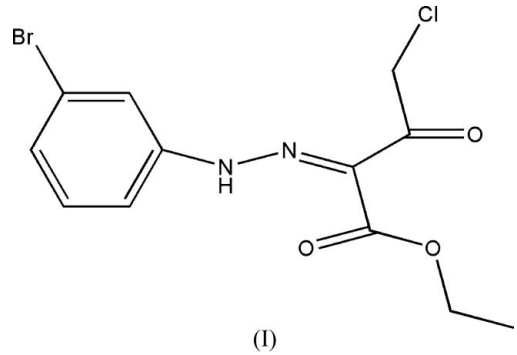
There are two crystallographically independent molecules in the asymmetric unit of the title compound, $C_{12}H_{12}ClBrN_2O_3$. The molecules adopt a keto–hydrazo tautomeric form, stabilized by an intramolecular hydrogen bond. The aromatic ring and aliphatic chain have a *trans* configuration about the N—N bond. The molecules pack *via* weak intermolecular C—H···O hydrogen bonds which, together with an intramolecular N—H···O bond, form an $S(6)[R_2^2(14)]S(6)$ motif.

Comment

1,3-Diketones are used in the synthesis of many organic compounds, such as diazepines (Khudina *et al.*, 2004), pyrazoles, pyrimidines and their derivatives (Saleh *et al.*, 2003). 3-Phenylhydrazono-2,4-diones and their derivatives are used for the treatment of cancer and AIDS (Monga & Sausville, 2002). The chemistry of hydrazones has been intensively investigated in recent years, owing to their coordinating capability, pharmacological activity and antibacterial and antifungal properties, and their use in analytical chemistry as highly selective extractants (Domiano *et al.*, 1984; Li *et al.*, 1988; Sakamoto *et al.*, 1993).



(I)

The asymmetric unit of (I) consists of two crystallographically independent, but nearly identical, molecules linked by a C—H···O hydrogen bonds (Fig. 2 and Table 1). Compound (I) consists of an aromatic ring and an aliphatic chain linked through a hydrazone group (Fig. 1). The molecules adopt a *trans* configuration about the N1—N2 bond, as evidenced by the C1—N1—N2—C7 [179.9 (2)°] and C21—N11—N12—C27 [177.3 (3)°] torsion angles.

The two molecules in the asymmetric unit are roughly planar, the dihedral angles between the benzene rings and the mean planes defined by the C7–C12/O1–O3/Cl1 and C27–C32/O11–O13/Cl2 aliphatic chains being 2.16 (6) and 15.70 (8)°, respectively. There are strong intramolecular N—H···O hydrogen bonds (Table 1). In addition, there are $[R_2^2(14)]$ ring

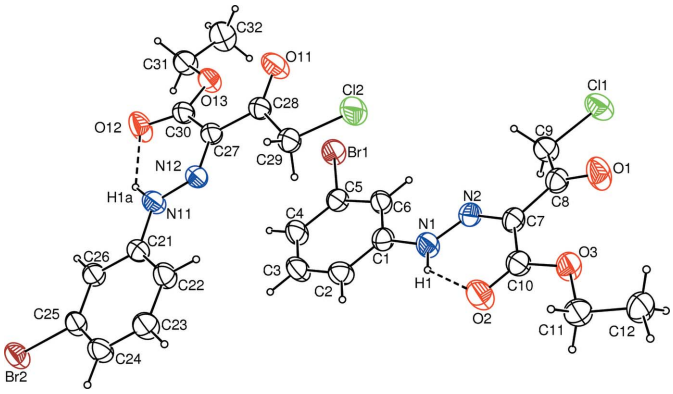


Figure 1
The asymmetric unit of (I) with the atom-numbering scheme, showing the intramolecular N—H···O hydrogen bonds (dashed lines). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

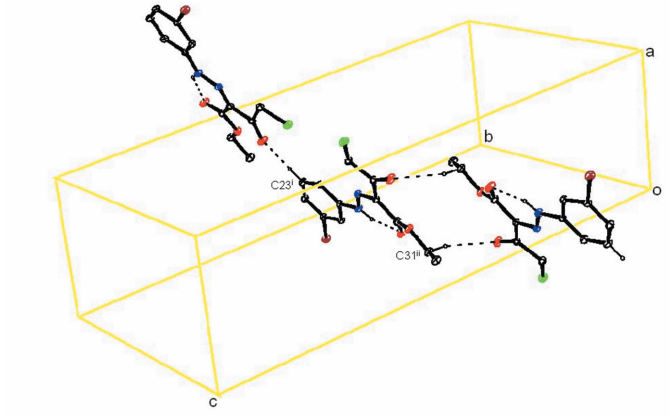


Figure 2
Part of the crystal structure of (I), showing the formation of a hydrogen-bonded $S(6)[R_2^2(14)]S(6)$ motif. Dashed lines indicate hydrogen bonds. H atoms not involved in these interactions have been omitted for clarity [Symmetry codes: (i) $\frac{5}{2}-x, y+\frac{1}{2}, \frac{3}{2}-z$; (ii) $1-x, -y, 1-z$.]

motifs constructed by intermolecular C—H···O hydrogen bonds (Fig. 2).

Experimental

The title compound was prepared as described by Odabaşoğlu *et al.* (2005), using *m*-bromoaniline and ethyl 4-chloroacetoacetate as starting materials (yield 74%, m.p. 369–371 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an absolute ethyl alcohol solution at room temperature.

Crystal data

$C_{12}H_{12}BrClN_2O_3$
M_r = 347.60
Monoclinic, $P2_1/n$
a = 7.4266 (3) Å
b = 14.1636 (7) Å
c = 26.6760 (10) Å
β = 95.354 (3)°
V = 2793.7 (2) Å^3
Z = 8
D_x = 1.653 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 3.14 mm^{-1}
T = 296 (2) K
Prism, orange
0.75 × 0.61 × 0.42 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe, 2002)
T_{min} = 0.206, T_{max} = 0.351
39907 measured reflections
5479 independent reflections
4315 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.070
S = 1.06
5479 reflections
352 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0211P)^2 + 1.3801P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00058 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.87 (3)	1.90 (3)	2.585 (3)	135 (3)
N11—H1*A*···O12	0.85 (4)	1.92 (4)	2.609 (3)	137 (4)
C23—H23···O1^{i}	0.93	2.54	3.311 (3)	141
C31—H31*A*···O11^{ii}	0.97	2.58	3.345 (4)	136

Symmetry codes: (i) $-x+\frac{5}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y, -z+1$.

The H atoms bonded to N1 and N11 were refined freely. All other H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *X-AREA* (Stoe, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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