IUCrData

ISSN 2414-3146

(2*E*)-3-Anilino-1-(2-chlorophenyl)-3-(methylsulfanyl)prop-2-en-1-one

A. J. Ravi,[a] A. C. Vinayaka,[b] Shamantha Kumar,[c] M. P. Sadashiva[b] and H. C. Devarajegowda[a]*

[a]Department of Physics, Yuvaraja's College (Constituent College), University of Mysore, Mysore 570 005, Karnataka, India, [b]Department of Studies in Chemistry, Manasagangotri, University of Mysore, Mysore 570 006, India, and [c]Department of Physics, SJB Institute of Technology, Kengeri,Bangalore 560 060, India. *Correspondence e-mail: devarajegowda@yahoo.com

Received 15 November 2016
Accepted 15 December 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

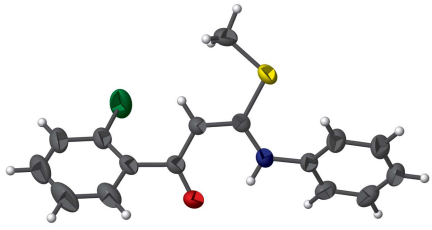
Keywords: crystal structure; enaminones; hydrogen bonding.

CCDC reference: 1522913

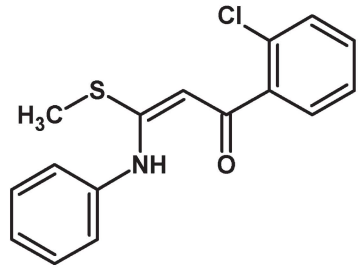
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_{14}ClNOS$, the dihedral angle between the aromatic rings is 86.34 (9)° and an intramolecular N—H···O hydrogen bond closes an *S*(6) ring. The methylsulfanyl group and Cl atom lie to the same side of the molecule. In the crystal, C—H···O hydrogen bonds link the molecules into (010) double sheets.

3D view



Chemical scheme



Structure description

β-Enaminones are compounds containing the conjugated system —N—C═C—C═O and can also be defined as monoenamines of 1,3-dicarbonyl compounds or vinylogous amides. *β*-Enaminones have been used in the synthesis of many heterocycles: for instance, biologically important isoxazoles (Dou *et al.*, 2013), pyrroles (Yan *et al.*, 2010) and pyrazoles (Neumann *et al.*, 2010) were synthesized from suitably substituted beta-enaminones. As part of our studies in this area, the title compound was synthesized and its crystal structure is reported here.

In the title compound (Fig. 1), the mean plane of the aniline unit makes a dihedral angle of 86.34 (9)° with the chlorobenzene moiety. The enaminone group is present in a *syn-clinal* (C5—C6—C7—O8) conformation with respect to the chlorobenzene moiety, as indicated by the torsion angle value of 44.8 (3)°. This conformation is supported by an intramolecular N—H···O hydrogen bond, which closes an *S*(6) ring. In the crystal, the molecules are linked by C—H···O hydrogen bonds (Table 1), generating (010) double sheets.

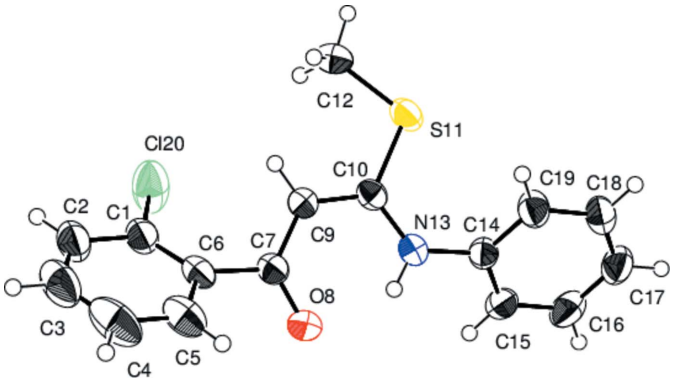


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

Synthesis and crystallization

A mixture of 1-(2-chlorophenyl)-3,3-bis(methylsulfanyl)prop-2-en-1-one, 1 (2.0 mmol, 1 equiv.) and aniline, 2 (2.6 mmol, 1.6 equiv.) was adsorbed on acidic silica and anhydrous $AlCl_3$ (0.03 equiv.) was added. The reaction mixture was stirred vigorously at 60°C for 4 h. After completion of reaction (monitored by TLC), the crude compound was purified by silica gel column chromatography. Colourless prisms were obtained from chloroform solution on slow evaporation at room temperature.

Refinement

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

Acknowledgements

The authors thank the Universities Sophisticated Instrumental Centre, Karnatak University, Dharwad for the CCD X-ray facilities.

References

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dou, G., Xu, P., Li, Q., Xi, Y., Huang, Z. & Shi, D. (2013). *Molecules*, **18**, 13645–13653.

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

D—H···A	D—H	H···A	D···A	D—H···A
N13—H13···O8	0.86	1.92	2.627 (2)	139
C12—H12A···O8^{i}	0.96	2.35	3.237 (3)	153
C15—H15···O8^{ii}	0.93	2.55	3.475 (3)	176

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{14}ClNOS$
M_r	303.79
Crystal system, space group	Orthorhombic, *Pccn*
Temperature (K)	293
a, b, c (Å)	15.9745 (6), 25.7276 (10), 7.4153 (3)
V (Å^3)	3047.6 (2)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	3.45
Crystal size (mm)	0.24 × 0.20 × 0.12
Data collection	
Diffractometer	Bruker SMART CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2007)
T_{min}, T_{max}	0.770, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19433, 2521, 2372
R_{int}	0.050
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.586
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.133, 1.06
No. of reflections	2521
No. of parameters	181
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.49, −0.46

Computer programs: *SMART* and *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Neumann, J. J., Suri, M. & Glorius, F. (2010). *Angew. Chem. Int. Ed.* **49**, 7790–7794.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Yan, R. L., Luo, J., Wang, C. X., Ma, C. W., Huang, G. S. & Liang, Y. M. (2010). *J. Org. Chem.* **75**, 5395–5397.

full crystallographic data

IUCrData (2016). **1**, x161994 [https://doi.org/10.1107/S2414314616019945]

(2*E*)-3-Anilino-1-(2-chlorophenyl)-3-(methylsulfanyl)prop-2-en-1-one

A. J. Ravi, A. C. Vinayaka, Shamantha Kumar, M. P. Sadashiva and H. C. Devarajegowda

(2*E*)-3-Anilino-1-(2-chlorophenyl)-3-(methylsulfanyl)prop-2-en-1-one

Crystal data

$C_{16}H_{14}ClNOS$
M_r = 303.79
Orthorhombic, *Pccn*
a = 15.9745 (6) Å
b = 25.7276 (10) Å
c = 7.4153 (3) Å
V = 3047.6 (2) Å^3
Z = 8
F(000) = 1264
D_x = 1.324 Mg m^{-3}
Melting point: 461 K
Cu *Kα* radiation, λ = 1.54178 Å
Cell parameters from 2521 reflections
θ = 5.5–64.5°
μ = 3.45 mm^{-1}
T = 293 K
Prism, colourless
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2007)
T_{min} = 0.770, T_{max} = 1.000
19433 measured reflections
2521 independent reflections
2372 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 64.5°, θ_{min} = 5.5°
h = −17→17
k = −29→29
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.133
S = 1.06
2521 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.079P)^2 + 1.4469P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S11	0.80692 (3)	0.46779 (2)	0.21016 (8)	0.0459 (2)
O8	0.54141 (10)	0.53861 (6)	0.2078 (3)	0.0576 (5)
N13	0.64227 (11)	0.45814 (7)	0.1896 (3)	0.0434 (4)
H13	0.5930	0.4716	0.1870	0.052*
C10	0.70480 (13)	0.49199 (8)	0.2154 (2)	0.0359 (5)
C14	0.64485 (12)	0.40362 (7)	0.1658 (3)	0.0383 (5)
C15	0.59380 (14)	0.38200 (9)	0.0350 (3)	0.0479 (5)
H15	0.5597	0.4032	−0.0352	0.057*
C1	0.63779 (15)	0.66268 (9)	0.2213 (3)	0.0478 (5)
C6	0.59290 (13)	0.62072 (9)	0.2879 (3)	0.0421 (5)
C18	0.69367 (16)	0.31856 (10)	0.2422 (4)	0.0554 (6)
H18	0.7276	0.2971	0.3120	0.066*
C16	0.59351 (17)	0.32902 (10)	0.0088 (4)	0.0567 (6)
H16	0.5589	0.3146	−0.0788	0.068*
C9	0.68755 (13)	0.54435 (8)	0.2402 (3)	0.0389 (5)
H9	0.7321	0.5670	0.2592	0.047*
C12	0.86963 (15)	0.52501 (10)	0.2226 (4)	0.0572 (6)
H12A	0.9278	0.5156	0.2206	0.086*
H12B	0.8575	0.5470	0.1215	0.086*
H12C	0.8574	0.5432	0.3325	0.086*
C19	0.69407 (16)	0.37150 (9)	0.2720 (3)	0.0492 (6)
H19	0.7272	0.3856	0.3629	0.059*
C2	0.62222 (19)	0.71335 (10)	0.2789 (4)	0.0627 (7)
H2	0.6531	0.7409	0.2323	0.075*
C7	0.60564 (13)	0.56482 (8)	0.2381 (3)	0.0400 (5)
C17	0.64396 (15)	0.29716 (9)	0.1111 (4)	0.0553 (6)
H17	0.6443	0.2615	0.0913	0.066*
C5	0.53032 (18)	0.63185 (11)	0.4144 (4)	0.0628 (7)
H5	0.4980	0.6049	0.4607	0.075*
C4	0.5158 (2)	0.68208 (15)	0.4713 (4)	0.0821 (10)
H4	0.4744	0.6886	0.5566	0.099*
C3	0.5617 (2)	0.72210 (12)	0.4031 (4)	0.0741 (9)
H3	0.5514	0.7558	0.4423	0.089*
Cl20	0.71297 (6)	0.65572 (3)	0.05499 (12)	0.0822 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S11	0.0398 (4)	0.0438 (4)	0.0543 (4)	0.0122 (2)	0.0001 (2)	0.0011 (2)
O8	0.0343 (9)	0.0455 (9)	0.0931 (13)	0.0023 (7)	−0.0026 (8)	0.0005 (8)
N13	0.0386 (10)	0.0337 (9)	0.0579 (11)	0.0046 (7)	−0.0030 (8)	0.0017 (8)
C10	0.0368 (11)	0.0376 (11)	0.0335 (10)	0.0042 (8)	−0.0009 (7)	0.0033 (8)
C14	0.0393 (11)	0.0339 (10)	0.0415 (10)	0.0003 (8)	0.0024 (8)	0.0021 (8)
C15	0.0420 (12)	0.0465 (12)	0.0551 (13)	−0.0084 (9)	−0.0074 (10)	0.0094 (10)
C1	0.0497 (13)	0.0409 (12)	0.0528 (13)	0.0074 (10)	−0.0064 (10)	−0.0030 (9)

C6	0.0400 (11)	0.0429 (12)	0.0432 (11)	0.0123 (9)	−0.0043 (9)	−0.0025 (9)
C18	0.0613 (16)	0.0370 (12)	0.0678 (15)	0.0046 (10)	−0.0090 (12)	0.0058 (11)
C16	0.0594 (15)	0.0526 (13)	0.0581 (13)	−0.0193 (11)	−0.0075 (12)	−0.0027 (11)
C9	0.0345 (11)	0.0350 (11)	0.0471 (11)	0.0019 (8)	0.0003 (8)	−0.0010 (9)
C12	0.0347 (12)	0.0616 (15)	0.0752 (17)	0.0035 (11)	−0.0053 (11)	0.0037 (12)
C19	0.0609 (15)	0.0388 (12)	0.0480 (12)	0.0009 (10)	−0.0136 (10)	0.0010 (9)
C2	0.0687 (17)	0.0427 (13)	0.0767 (17)	0.0126 (12)	−0.0213 (14)	−0.0101 (12)
C7	0.0358 (11)	0.0388 (11)	0.0454 (11)	0.0034 (9)	0.0013 (8)	0.0025 (8)
C17	0.0603 (15)	0.0360 (11)	0.0696 (15)	−0.0063 (10)	0.0040 (12)	−0.0062 (11)
C5	0.0584 (15)	0.0699 (16)	0.0601 (14)	0.0232 (13)	0.0038 (12)	−0.0044 (13)
C4	0.080 (2)	0.102 (3)	0.0648 (17)	0.047 (2)	0.0060 (15)	−0.0224 (17)
C3	0.085 (2)	0.0623 (17)	0.0748 (18)	0.0291 (16)	−0.0190 (16)	−0.0257 (15)
Cl20	0.0963 (6)	0.0550 (4)	0.0954 (6)	0.0047 (3)	0.0439 (4)	0.0124 (3)

Geometric parameters (Å, º)

S11—C10	1.747 (2)	C18—H18	0.9300
S11—C12	1.783 (3)	C16—C17	1.377 (4)
O8—C7	1.248 (3)	C16—H16	0.9300
N13—C10	1.339 (3)	C9—C7	1.411 (3)
N13—C14	1.414 (3)	C9—H9	0.9300
N13—H13	0.8600	C12—H12A	0.9600
C10—C9	1.387 (3)	C12—H12B	0.9600
C14—C15	1.384 (3)	C12—H12C	0.9600
C14—C19	1.386 (3)	C19—H19	0.9300
C15—C16	1.377 (3)	C2—C3	1.354 (5)
C15—H15	0.9300	C2—H2	0.9300
C1—C6	1.387 (3)	C17—H17	0.9300
C1—C2	1.394 (3)	C5—C4	1.379 (4)
C1—Cl20	1.731 (3)	C5—H5	0.9300
C6—C5	1.400 (3)	C4—C3	1.362 (5)
C6—C7	1.499 (3)	C4—H4	0.9300
C18—C17	1.371 (4)	C3—H3	0.9300
C18—C19	1.380 (3)		
C10—S11—C12	103.25 (11)	S11—C12—H12A	109.5
C10—N13—C14	129.89 (18)	S11—C12—H12B	109.5
C10—N13—H13	115.1	H12A—C12—H12B	109.5
C14—N13—H13	115.1	S11—C12—H12C	109.5
N13—C10—C9	120.16 (19)	H12A—C12—H12C	109.5
N13—C10—S11	117.49 (16)	H12B—C12—H12C	109.5
C9—C10—S11	122.32 (16)	C18—C19—C14	119.7 (2)
C15—C14—C19	119.5 (2)	C18—C19—H19	120.2
C15—C14—N13	117.94 (18)	C14—C19—H19	120.2
C19—C14—N13	122.49 (19)	C3—C2—C1	119.4 (3)
C16—C15—C14	119.9 (2)	C3—C2—H2	120.3
C16—C15—H15	120.0	C1—C2—H2	120.3
C14—C15—H15	120.0	O8—C7—C9	124.24 (19)

C6—C1—C2	121.8 (2)	O8—C7—C6	116.82 (19)
C6—C1—Cl20	122.15 (17)	C9—C7—C6	118.79 (19)
C2—C1—Cl20	116.0 (2)	C18—C17—C16	119.4 (2)
C1—C6—C5	116.7 (2)	C18—C17—H17	120.3
C1—C6—C7	126.07 (19)	C16—C17—H17	120.3
C5—C6—C7	117.3 (2)	C4—C5—C6	121.1 (3)
C17—C18—C19	120.8 (2)	C4—C5—H5	119.4
C17—C18—H18	119.6	C6—C5—H5	119.4
C19—C18—H18	119.6	C3—C4—C5	120.3 (3)
C15—C16—C17	120.7 (2)	C3—C4—H4	119.9
C15—C16—H16	119.7	C5—C4—H4	119.9
C17—C16—H16	119.7	C2—C3—C4	120.8 (3)
C10—C9—C7	123.1 (2)	C2—C3—H3	119.6
C10—C9—H9	118.5	C4—C3—H3	119.6
C7—C9—H9	118.5		

C14—N13—C10—C9	−178.4 (2)	N13—C14—C19—C18	179.9 (2)
C14—N13—C10—S11	3.3 (3)	C6—C1—C2—C3	0.0 (4)
C12—S11—C10—N13	173.40 (17)	Cl20—C1—C2—C3	177.2 (2)
C12—S11—C10—C9	−4.9 (2)	C10—C9—C7—O8	−2.6 (3)
C10—N13—C14—C15	−137.6 (2)	C10—C9—C7—C6	172.84 (19)
C10—N13—C14—C19	44.6 (3)	C1—C6—C7—O8	−135.6 (2)
C19—C14—C15—C16	−1.3 (3)	C5—C6—C7—O8	44.8 (3)
N13—C14—C15—C16	−179.2 (2)	C1—C6—C7—C9	48.6 (3)
C2—C1—C6—C5	0.8 (3)	C5—C6—C7—C9	−131.0 (2)
Cl20—C1—C6—C5	−176.24 (18)	C19—C18—C17—C16	−0.4 (4)
C2—C1—C6—C7	−178.7 (2)	C15—C16—C17—C18	1.2 (4)
Cl20—C1—C6—C7	4.2 (3)	C1—C6—C5—C4	−1.2 (4)
C14—C15—C16—C17	−0.4 (4)	C7—C6—C5—C4	178.4 (2)
N13—C10—C9—C7	−0.7 (3)	C6—C5—C4—C3	0.9 (4)
S11—C10—C9—C7	177.57 (16)	C1—C2—C3—C4	−0.4 (4)
C17—C18—C19—C14	−1.3 (4)	C5—C4—C3—C2	0.0 (5)
C15—C14—C19—C18	2.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N13—H13···O8	0.86	1.92	2.627 (2)	139
C12—H12*A*···O8^{i}	0.96	2.35	3.237 (3)	153
C15—H15···O8^{ii}	0.93	2.55	3.475 (3)	176

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