

## 2-(3-Methylphenyl)-1,2-benzoselenazol-3(2H)-one

Liyun Wang,<sup>a</sup> Ying Xu,<sup>a</sup> Zhiqiang Guo<sup>b\*</sup> and Xuehong Wei<sup>b</sup><sup>a</sup>School of Chemistry and Chemical Engineering, Shanxi University, Taiyuan 030006, People's Republic of China, and<sup>b</sup>Scientific Instrument Center, Shanxi University, Taiyuan 030006, People's Republic of China. \*Correspondence e-mail: gzq@sxu.edu.cn

Received 23 March 2017

Accepted 10 April 2017

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

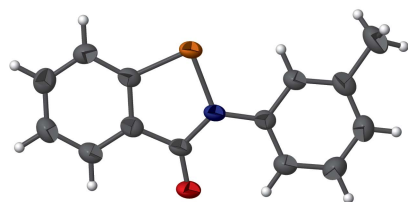
Keywords: crystal structure; ebselen analogue; benzoselenazole; short Se···O contact; C—H···O hydrogen bonds; offset  $\pi$ – $\pi$  interactions.

CCDC reference: 666227

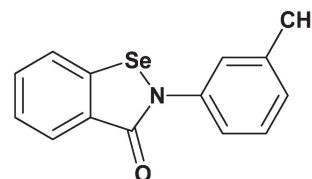
Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

In the title ebselen derivative, C<sub>14</sub>H<sub>11</sub>NOSe, the nine-membered benzoselenazolyl ring system is approximately planar (r.m.s. deviation = 0.021 Å). The dihedral angle between its mean plane and that of the 3-methylphenyl ring is 5.37 (11)°. The five-membered isoselenazolyl ring is severely strained at the Se atom: Se–N = 1.889 (2) Å, Se–C<sub>ar</sub> = 1.882 (3) Å and N–Se–C<sub>ar</sub> = 83.30 (10)°. In the crystal, molecules are linked by C–H···O hydrogen bonds and short intermolecular Se···O contacts of 2.6917 (19) Å, forming chains along the *c*-axis direction. Neighbouring molecules are linked by offset  $\pi$ – $\pi$  interactions [intercentroid distance = 3.535 (2) Å]. The chains are also linked by C–H··· $\pi$  interactions, forming a three-dimensional structure.

## 3D view



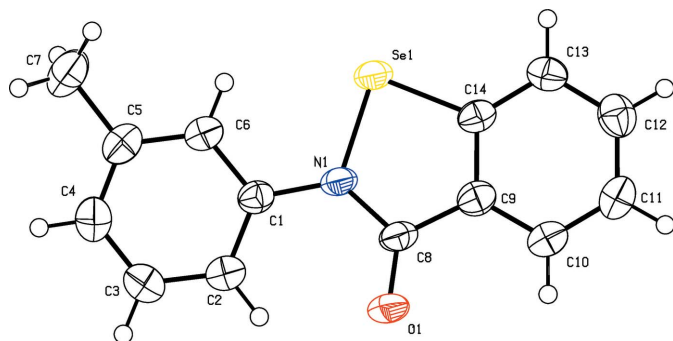
## Chemical scheme



## Structure description

Ebselen (systematic name: 2-phenyl-1,2-benzoselenazol-3-one) is a synthetic organoselenium drug which has been reported to exhibit anti-inflammatory, anti-oxidant and cytoprotective activity (Mugesh & Singh, 2000; Mugesh *et al.*, 2001*a,b*). We report herein on the synthesis and crystal structure of the 3-methylphenyl derivative of ebselen.

The molecular structure of the title compound is shown in Fig. 1. The nine-membered benzoselenazolyl group is roughly planar (r.m.s. deviation = 0.021 Å). The dihedral angle between its mean plane and the 3-methylphenyl ring is 5.37 (11)°. This value is much smaller than that reported for *N*-(2-methylphenyl)-1,2-benzoselenazol-3(2H)-one (Zhu *et al.*, 2013) in which the 2-methylphenyl ring is inclined to the benzoselenazolyl ring system by 78.15 (11)°. In two polymorphs of ebselen, the corresponding dihedral angle is 24.24 (8)° for the monoclinic *P*2<sub>1</sub>/*c* polymorph and 33.36 (12)° for the orthorhombic *Pbca* polymorph (Thomas *et al.*, 2015). In the orthorhombic *Pna*2<sub>1</sub> polymorph of *m*-ebselenol (Thomas *et al.*, 2015), the corresponding dihedral angle is 35.17 (12)°. The five-membered isoselenazolyl ring is severely strained at the Se atom: Se1–N1 =

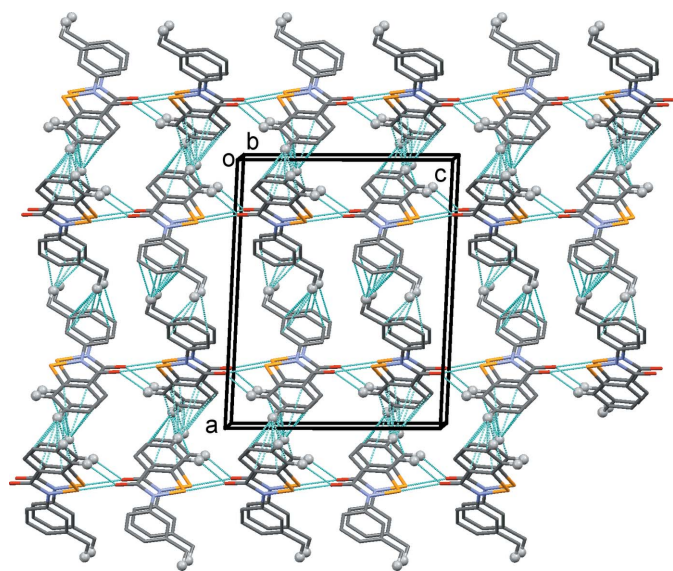


**Figure 1**  
The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 25% probability level.

1.889 (2) Å, Se1—C14 = 1.882 (3) Å, N1—Se1—C14 = 83.30 (10)°. These values are similar to those reported for the above mentioned compounds.

In the crystal, molecules are linked by C—H···O hydrogen bonds, and short Se1···O<sup>i</sup> contacts of 2.6917 (19) Å [symmetry code: (i)  $x, -y + \frac{1}{2}, z - \frac{1}{2}$ ], forming chains along the *c*-axis direction (Table 1 and Fig. 2). Neighbouring molecules are linked by offset  $\pi$ – $\pi$  interactions [ $Cg2 \cdots Cg4^i = 3.535$  (2) Å,  $Cg2$  and  $Cg4$  are the centroids of rings C1–C6 and Se1/N1/C8–C14, respectively,  $\alpha = 5.37$  (11)°, interplanar distances = 3.499 (1) and 3.466 (1) Å, slippage = 0.693 Å; symmetry code: (i)  $x, y - 1, z$ ]. The chains are also linked by C—H··· $\pi$  interactions, forming a three-dimensional structure (Table 1 and Fig. 2).

The Se···O distance in *N*-(2-methylphenyl)-1,2-benzoseleazol-3(2*H*)-one (Zhu *et al.*, 2013) is 2.733 (3) Å. In the two polymorphs of ebselen mentioned above the Se···O contacts are slightly shorter at 2.533 (2) and 2.522 (2) Å.



**Figure 2**  
A view along the *b* axis of the crystal packing of the title compound. The C—H···O hydrogen bonds, Se···O contacts and C—H··· $\pi$  interactions are shown as dashed lines (see Table 1).

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

$Cg2$  and  $Cg3$  are the centroids of rings C1–C6 and C9–C14, respectively.

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C13—H13···O1 <sup>i</sup>	0.93	2.38	3.057 (3)	129
C7—H7C···Cg2 <sup>ii</sup>	0.96	2.97	3.926 (4)	176
C12—H12···Cg3 <sup>iii</sup>	0.93	2.74	3.535 (3)	144

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

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	C <sub>14</sub> H <sub>11</sub> NOSe
<i>M<sub>r</sub></i>	288.20
Crystal system, space group	Monoclinic, <i>P</i> <sub>2</sub> /c
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	15.561 (4), 5.9415 (16), 12.471 (3)
$\beta$ (°)	92.799 (3)
<i>V</i> (Å <sup>3</sup> )	1151.6 (5)
<i>Z</i>	4
Radiation type	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	3.24
Crystal size (mm)	0.20 × 0.10 × 0.10
Data collection	
Diffractometer	Bruker SMART CCD area-detector
Absorption correction	Multi-scan (SADABS; Bruker, 2001)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.563, 0.737
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	4482, 2015, 1728
<i>R<sub>int</sub></i>	0.021
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , <i>S</i>	0.027, 0.073, 1.05
No. of reflections	2015
No. of parameters	155
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$ , $\Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.42, -0.21

Computer programs: SMART and SAINT (Bruker, 2001), SHELXS2014 (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015), PLATON (Spek, 2009), Mercury (Macrae *et al.*, 2008) and publCIF (Westrip, 2010).

## Synthesis and crystallization

The title compound was prepared following a modified literature procedure (Zhu *et al.*, 2013). A solution of 2-(chloroseleno)benzoyl chloride (0.76 g, 3 mmol) in dry acetonitrile (20 ml) was added dropwise to a solution of 3-methylaniline (0.322 g, 3 mmol) and triethylamine in dry acetonitrile (20 ml) at room temperature. The reaction mixture was stirred at room temperature for about 5 h and the solvent was evaporated *in vacuo*. The precipitate was recrystallized to obtain colourless block-like crystals (yield 55%, m.p. 426–427 K).

<sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  2.38 (s, 3 H, CH<sub>3</sub>), 7.08–7.10 (d, <sup>3</sup>*J* = 7.3 Hz, 1 H, H—C11), 7.27–7.32 (t, <sup>3</sup>*J* = 8.3 Hz, 1 H, H—C10), 7.38–7.45 (m, 3 H, H—C2, C9, C13), 7.59–7.70 (m, 2 H, H—C3, C4), 8.10–8.12 (d, <sup>3</sup>*J* = 7.8 Hz, 1 H, H—C5); <sup>13</sup>C NMR:  $\delta$  24.1 (CH<sub>3</sub>), 125.3 (C9), 126.6 (C13), 128.8 (C11), 129.2 (C10), 130.4 (C2), 131.6 (C4), 131.8 (C5), 132.0 (C3), 135.1 (C1), 140.6

(C12), 141.6 (C6), 142.0 (C8), 168.5 (C=O);  $^{77}\text{Se}$  NMR:  $\delta$  961. Analysis calculated for  $\text{C}_{14}\text{H}_{11}\text{NOSe}$ : C, 58.34; H, 3.85; N, 4.86%. Found: C, 58.20; H, 3.90; N, 4.77%.

### Refinement

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

### Funding information

Funding for this research was provided by: Special Fund for Agro-scientific Research in the Public Interest (award No. 201303106).

### References

- Bruker (2001). *SMART, SAINT and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Mugesh, G., du Mont, W. W. & Sies, H. (2001a). *Chem. Rev.* **101**, 2125–2179.
- Mugesh, G., Panda, A., Singh, H. B., Punekar, N. S. & Butcher, R. J. (2001b). *J. Am. Chem. Soc.* **123**, 839–850.
- Mugesh, G. & Singh, H. B. (2000). *Chem. Soc. Rev.* **29**, 347–357.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst. C* **71**, 3–8.
- Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.
- Thomas, S. P., Satheeshkumar, K., Mugesh, G. & Guru Row, T. N. (2015). *Chem. Eur. J.* **21**, 6793–6800.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Zhu, X., Xu, Y., Han, H., Guo, Z. & Wei, X. (2013). *Acta Cryst. E* **69**, o1538.

## full crystallographic data

*IUCrData* (2017). **2**, x170532 [<https://doi.org/10.1107/S2414314617005326>]

## 2-(3-Methylphenyl)-1,2-benzoselenazol-3(2H)-one

Liyun Wang, Ying Xu, Zhiqiang Guo and Xuehong Wei

## 2-(3-Methylphenyl)-1,2-benzoselenazol-3(2H)-one

*Crystal data*

$C_{14}H_{11}NOSe$

$M_r = 288.20$

Monoclinic,  $P2_1/c$

$a = 15.561$  (4) Å

$b = 5.9415$  (16) Å

$c = 12.471$  (3) Å

$\beta = 92.799$  (3)°

$V = 1151.6$  (5) Å<sup>3</sup>

$Z = 4$

$F(000) = 576$

$D_x = 1.662$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2393 reflections

$\theta = 2.6$ – $27.1$ °

$\mu = 3.24$  mm<sup>-1</sup>

$T = 293$  K

Block, colorless

$0.20 \times 0.10 \times 0.10$  mm

*Data collection*

Bruker SMART CCD area-detector  
diffractometer

phi and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Bruker, 2001)

$T_{\min} = 0.563$ ,  $T_{\max} = 0.737$

4482 measured reflections

2015 independent reflections

1728 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 25.0$ °,  $\theta_{\min} = 2.6$ °

$h = -18 \rightarrow 16$

$k = -6 \rightarrow 7$

$l = -9 \rightarrow 14$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.073$

$S = 1.05$

2015 reflections

155 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.1339P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.42$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.21$  e Å<sup>-3</sup>

*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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Se1	0.77312 (2)	0.16715 (4)	0.66672 (2)	0.03852 (12)
N1	0.75687 (14)	0.0480 (3)	0.80472 (15)	0.0362 (5)
O1	0.79688 (14)	0.1110 (3)	0.98233 (14)	0.0527 (5)
C1	0.70239 (17)	-0.1434 (4)	0.8143 (2)	0.0350 (6)
C2	0.69338 (19)	-0.2532 (5)	0.9106 (2)	0.0488 (7)
H2	0.7217	-0.2012	0.9731	0.059*
C3	0.6413 (2)	-0.4427 (5)	0.9123 (3)	0.0610 (9)
H3	0.6353	-0.5184	0.9768	0.073*
C4	0.5983 (2)	-0.5212 (5)	0.8206 (3)	0.0554 (8)
H4	0.5637	-0.6484	0.8240	0.066*
C5	0.60614 (19)	-0.4142 (5)	0.7252 (2)	0.0473 (7)
C6	0.65794 (18)	-0.2238 (5)	0.7229 (2)	0.0437 (7)
H6	0.6630	-0.1480	0.6583	0.052*
C7	0.5604 (2)	-0.4993 (6)	0.6237 (2)	0.0652 (9)
H7A	0.5975	-0.6005	0.5876	0.098*
H7B	0.5457	-0.3745	0.5775	0.098*
H7C	0.5090	-0.5772	0.6414	0.098*
C8	0.79923 (18)	0.1616 (4)	0.8870 (2)	0.0378 (6)
C9	0.84756 (17)	0.3536 (4)	0.8470 (2)	0.0393 (6)
C10	0.89553 (18)	0.5007 (5)	0.9122 (2)	0.0458 (7)
H10	0.9003	0.4780	0.9861	0.055*
C11	0.93588 (19)	0.6799 (4)	0.8671 (2)	0.0491 (7)
H11	0.9684	0.7785	0.9105	0.059*
C12	0.92841 (18)	0.7147 (5)	0.7574 (2)	0.0480 (7)
H12	0.9556	0.8380	0.7280	0.058*
C13	0.88175 (18)	0.5709 (5)	0.6909 (2)	0.0432 (6)
H13	0.8772	0.5948	0.6171	0.052*
C14	0.84146 (16)	0.3887 (4)	0.7370 (2)	0.0359 (6)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Se1	0.0507 (2)	0.03956 (18)	0.02478 (17)	0.00204 (11)	-0.00299 (12)	-0.00034 (10)
N1	0.0476 (12)	0.0378 (12)	0.0228 (11)	0.0000 (10)	-0.0027 (9)	-0.0011 (9)
O1	0.0817 (15)	0.0527 (11)	0.0234 (10)	-0.0107 (11)	-0.0015 (9)	-0.0023 (8)
C1	0.0394 (14)	0.0344 (14)	0.0312 (13)	0.0040 (11)	0.0013 (11)	-0.0033 (10)
C2	0.0575 (19)	0.0522 (16)	0.0360 (16)	-0.0063 (15)	-0.0042 (13)	0.0012 (14)
C3	0.075 (2)	0.0583 (19)	0.0489 (19)	-0.0152 (17)	-0.0015 (16)	0.0136 (15)
C4	0.0557 (19)	0.0503 (17)	0.060 (2)	-0.0135 (15)	-0.0011 (15)	0.0002 (15)
C5	0.0476 (17)	0.0471 (15)	0.0464 (18)	0.0019 (13)	-0.0051 (13)	-0.0098 (13)
C6	0.0498 (17)	0.0475 (15)	0.0334 (15)	-0.0029 (13)	-0.0025 (12)	-0.0023 (12)
C7	0.064 (2)	0.069 (2)	0.061 (2)	-0.0125 (17)	-0.0090 (16)	-0.0188 (17)
C8	0.0503 (16)	0.0372 (14)	0.0255 (13)	0.0043 (11)	-0.0038 (11)	-0.0027 (11)
C9	0.0407 (15)	0.0417 (15)	0.0355 (15)	0.0045 (12)	0.0009 (11)	-0.0040 (11)
C10	0.0508 (16)	0.0510 (16)	0.0352 (15)	-0.0011 (14)	-0.0023 (12)	-0.0053 (13)

C11	0.0471 (17)	0.0473 (16)	0.0524 (19)	-0.0062 (13)	-0.0032 (14)	-0.0103 (13)
C12	0.0440 (16)	0.0411 (15)	0.059 (2)	0.0015 (13)	0.0015 (14)	0.0054 (13)
C13	0.0459 (16)	0.0468 (15)	0.0367 (15)	0.0030 (13)	0.0007 (12)	0.0068 (12)
C14	0.0383 (14)	0.0378 (13)	0.0313 (14)	0.0072 (11)	-0.0028 (11)	-0.0027 (11)

*Geometric parameters (Å, °)*

Se1—C14	1.882 (3)	C6—H6	0.9300
Se1—N1	1.889 (2)	C7—H7A	0.9600
N1—C8	1.370 (3)	C7—H7B	0.9600
N1—C1	1.427 (3)	C7—H7C	0.9600
O1—C8	1.229 (3)	C8—C9	1.467 (4)
C1—C2	1.379 (4)	C9—C14	1.386 (4)
C1—C6	1.389 (4)	C9—C10	1.387 (4)
C2—C3	1.388 (4)	C10—C11	1.370 (4)
C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.377 (4)	C11—C12	1.383 (4)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.360 (4)	C12—C13	1.373 (4)
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.390 (4)	C13—C14	1.390 (4)
C5—C7	1.508 (4)	C13—H13	0.9300
C14—Se1—N1	86.30 (10)	C5—C7—H7C	109.5
C8—N1—C1	126.6 (2)	H7A—C7—H7C	109.5
C8—N1—Se1	114.61 (17)	H7B—C7—H7C	109.5
C1—N1—Se1	118.78 (15)	O1—C8—N1	124.6 (2)
C2—C1—C6	118.9 (3)	O1—C8—C9	123.9 (2)
C2—C1—N1	122.4 (2)	N1—C8—C9	111.4 (2)
C6—C1—N1	118.7 (2)	C14—C9—C10	119.6 (2)
C1—C2—C3	118.8 (3)	C14—C9—C8	116.2 (2)
C1—C2—H2	120.6	C10—C9—C8	124.2 (2)
C3—C2—H2	120.6	C11—C10—C9	119.5 (3)
C4—C3—C2	121.5 (3)	C11—C10—H10	120.2
C4—C3—H3	119.3	C9—C10—H10	120.2
C2—C3—H3	119.3	C10—C11—C12	120.3 (3)
C5—C4—C3	120.5 (3)	C10—C11—H11	119.9
C5—C4—H4	119.7	C12—C11—H11	119.9
C3—C4—H4	119.7	C13—C12—C11	121.4 (3)
C4—C5—C6	118.3 (3)	C13—C12—H12	119.3
C4—C5—C7	121.3 (3)	C11—C12—H12	119.3
C6—C5—C7	120.4 (3)	C12—C13—C14	118.1 (3)
C1—C6—C5	122.0 (3)	C12—C13—H13	121.0
C1—C6—H6	119.0	C14—C13—H13	121.0
C5—C6—H6	119.0	C9—C14—C13	121.1 (2)
C5—C7—H7A	109.5	C9—C14—Se1	111.45 (19)
C5—C7—H7B	109.5	C13—C14—Se1	127.5 (2)
H7A—C7—H7B	109.5		

*Hydrogen-bond geometry (Å, °)*

Cg2 and Cg3 are the centroids of rings C1–C6 and C9–C14, respectively.

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C13—H13 $\cdots$ O1 <sup>i</sup>	0.93	2.38	3.057 (3)	129
C7—H7C $\cdots$ Cg2 <sup>ii</sup>	0.96	2.97	3.926 (4)	176
C12—H12 $\cdots$ Cg3 <sup>iii</sup>	0.93	2.74	3.535 (3)	144

Symmetry codes: (i)  $x, -y+1/2, z-1/2$ ; (ii)  $-x+1, y-1/2, -z+3/2$ ; (iii)  $-x+2, y+1/2, -z+3/2$ .