

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

## 3,4-Dibromo-2,5-dimethyl-1-phenyl-sulfonyl-1H-pyrrole

J. Kanchanadevi,<sup>a</sup> G. Anbalagan,<sup>b</sup> R. Sureshbabu,<sup>c</sup>  
A. K. Mohanakrishnan<sup>c</sup> and V. Manivannan<sup>d\*</sup>

<sup>a</sup>Department of Physics, Velammal Institute of Technology, Panchetty, Chennai 601 204, India, <sup>b</sup>Department of Physics, Presidency College (Autonomous), Chennai 600 005, India, <sup>c</sup>Department of Organic Chemistry, University of Madras, Guindy campus, Chennai 600 025, India, and <sup>d</sup>Department of Research and Development, PRIST University, Vallam, Thanjavur 613 403, Tamil Nadu, India  
Correspondence e-mail: crystallography2010@gmail.com, phdguna@gmail.com

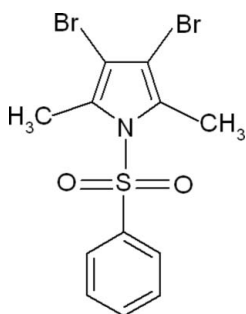
Received 14 July 2011; accepted 28 July 2011

Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.034;  $wR$  factor = 0.075; data-to-parameter ratio = 21.2.

In the title compound,  $\text{C}_{12}\text{H}_{11}\text{Br}_2\text{NO}_2\text{S}$ , the dihedral angle between the two rings is  $78.79$  ( $12$ )°. The crystal packing features  $\text{C}-\text{H}\cdots\pi$  interactions.

### Related literature

For the biological activity of heterocyclic compounds, see: Ali *et al.* (1989); Amal Raj *et al.* (2003). For related structures, see: Seshadri *et al.* (2009); Gunasekaran *et al.* (2009).



### Experimental

#### Crystal data

$\text{C}_{12}\text{H}_{11}\text{Br}_2\text{NO}_2\text{S}$   
 $M_r = 393.10$

Orthorhombic,  $P2_12_12_1$   
 $a = 6.6248$  (4) Å

$b = 9.7172$  (6) Å  
 $c = 21.2083$  (11) Å  
 $V = 1365.27$  (14) Å<sup>3</sup>  
 $Z = 4$

Mo  $K\alpha$  radiation  
 $\mu = 6.08$  mm<sup>-1</sup>  
 $T = 295$  K  
 $0.35 \times 0.25 \times 0.20$  mm

#### Data collection

Bruker Kappa APEXII CCD diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.945$ ,  $T_{\max} = 0.955$

9255 measured reflections  
3491 independent reflections  
2735 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.075$   
 $S = 1.02$   
3491 reflections  
165 parameters  
H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.47$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.92$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983),  
1460 Friedel pairs  
Flack parameter: 0.010 (10)

**Table 1**

Hydrogen-bond geometry (Å, °).

$\text{Cg}2$  is the centroid of the  $\text{C}5-\text{C}10$  ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}12-\text{H}12A\cdots\text{Cg}2^i$	0.96	2.85	3.545 (7)	130

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

Data collection: APEX2 (Bruker, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5579).

### References

- Ali, R., Misra, B. & Nizamuddin, M. (1989). *Indian J. Chem. Sect. B*, **28**, 526–528.  
Amal Raj, A., Raghunathan, R., Sridevikumari, M. R. & Raman, N. (2003). *Bioorg. Med. Chem.* **11**, 407–419.  
Bruker (2003). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.  
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
Gunasekaran, B., Sureshbabu, R., Mohanakrishnan, A. K., Chakkaravarthi, G. & Manivannan, V. (2009). *Acta Cryst.* **E65**, o2069.  
Seshadri, P. R., Balakrishnan, B., Ilangovan, K., Sureshbabu, R. & Mohanakrishnan, A. K. (2009). *Acta Cryst.* **E65**, o531.  
Sheldrick, G. M. (1996). SADABS, University of Göttingen, Germany.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

## supporting information

*Acta Cryst.* (2011). E67, o2224 [doi:10.1107/S1600536811030443]

## 3,4-Dibromo-2,5-dimethyl-1-phenylsulfonyl-1H-pyrrole

J. Kanchanadevi, G. Anbalagan, R. Sureshbabu, A. K. Mohanakrishnan and V. Manivannan

### S1. Comment

Heterocycles, especially five-membered rings, are involved in a wide range of biologically important chemical reactions in living organisms, and therefore they form one of the most important and well investigated classes of organic compounds. They have exhibit antifungal (Amal Raj *et al.*, 2003) and fungicidal (Ali *et al.*, 1989) activity.

The geometric parameters of the title molecule (Fig. 1) agree well with reported similar structure (Seshadri *et al.*, 2009). The phenyl and pyrrole rings inclined at an angle of 78.79 (12) °. The sum of bond angles around N1 [359.6 (3) °] indicates the  $sp^2$  hybridization state of atom N1 in the molecule.

The angular disposition of the bonds about the 'S' atom show significant deviation from that a regular tetrahedron, with the largest deviation in O—S—O angle. The widening of angle O1—S1—O2 = 120.09 (18) ° from the ideal tetrahedral value is the result of the repulsive interactions between the short S=O bonds similar to that observed in other structures (Gunasekaran *et al.*, 2009).

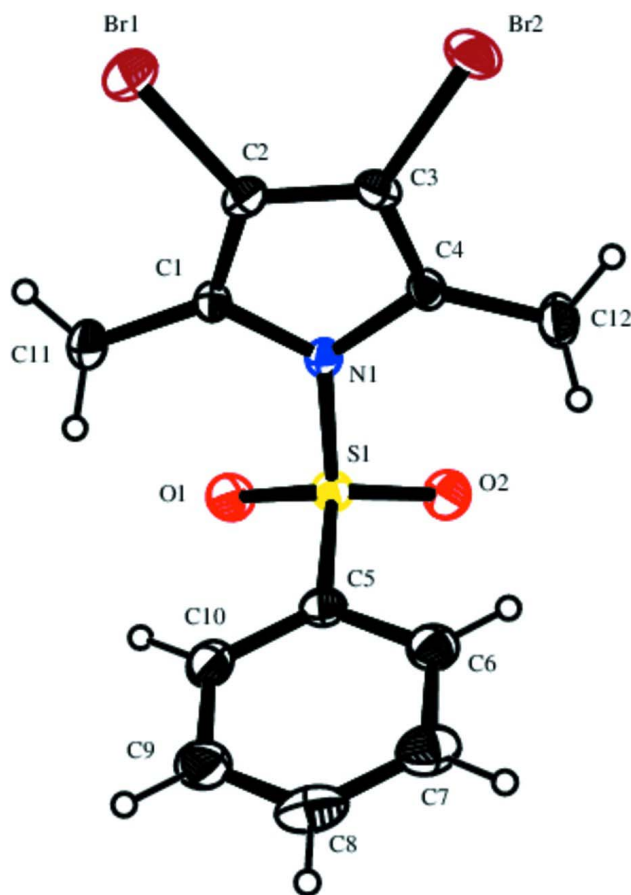
The molecular structure is stabilized by weak intramolecular C—H...O and C—H...Br interactions. The crystal packing is controlled by C—H... $\pi$  [C12—H12A...Cg2(1 - x, -1/2 + y, 1/2 - z) distance of 3.545 (7) Å (Cg2 is the centroid of the ring defined by the atoms C5—C10)] interaction.

### S2. Experimental

To a solution of tertiary butoxide (1.33 g, 11.85 mmol) and 18-crown -6 (0.15 g, 0.59 mmol) in dry tetrahydrofuran (30 ml), 3,4-dibromo-2,5-dimethyl-1H-pyrrole (1.5 g, 5.92 mmol) was added. It was then stirred at room temperature for 30 minutes under nitrogen atmosphere. Then, phenylsulfonyl chloride (0.9 ml, 7.11 mmol) was added through syringe and stirred at the same temperature for 4 h. The reaction mixture was poured to water (100 ml) and extracted with ethyl-acetate (2 x 30 ml). The solvent was removed under reduced pressure. The solid obtained was recrystallized from chloroform to give pure product as a pale brown solid. The yield of the product is 65% and melting point is 515 K.

### S3. Refinement

H atoms were positioned geometrically and refined using riding model with C—H = 0.93 Å and  $U_{iso}(H) = 1.2U_{eq}(C)$  for aromatic C—H, C—H = 0.96 Å and  $U_{iso}(H) = 1.5U_{eq}(C)$  for methyl groups.

**Figure 1**

The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

### 3,4-Dibromo-2,5-dimethyl-1-phenylsulfonyl-1*H*-pyrrole

#### Crystal data

$C_{12}H_{11}Br_2NO_2S$

$M_r = 393.10$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 6.6248$  (4) Å

$b = 9.7172$  (6) Å

$c = 21.2083$  (11) Å

$V = 1365.27$  (14) Å<sup>3</sup>

$Z = 4$

$F(000) = 768$

$D_x = 1.912$  Mg m<sup>-3</sup>

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

Cell parameters from 7415 reflections

$\theta = 2.0$ – $28.7^\circ$

$\mu = 6.08$  mm<sup>-1</sup>

$T = 295$  K

Block, pale brown

$0.35 \times 0.25 \times 0.20$  mm

#### Data collection

Bruker Kappa APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 0 pixels mm<sup>-1</sup>

$\omega$  and  $\phi$  scans

Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.945$ ,  $T_{\max} = 0.955$

9255 measured reflections

3491 independent reflections

2735 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 28.6^\circ$ ,  $\theta_{\min} = 1.9^\circ$   
 $h = -8 \rightarrow 8$

$k = -13 \rightarrow 10$   
 $l = -17 \rightarrow 28$

### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.075$   
 $S = 1.02$   
 3491 reflections  
 165 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  
 $w = 1/[\sigma^2(F_o^2) + (0.029P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.47 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.92 \text{ e } \text{\AA}^{-3}$   
 Absolute structure: Flack (1983), 1460 Friedel  
 pairs  
 Absolute structure parameter: 0.010 (10)

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1597 (5)	0.2073 (3)	0.41513 (14)	0.0293 (7)
C2	0.1708 (5)	0.0859 (3)	0.44645 (14)	0.0327 (8)
C3	0.3469 (6)	0.0151 (3)	0.42718 (15)	0.0329 (7)
C4	0.4461 (5)	0.0895 (3)	0.38337 (14)	0.0298 (7)
C5	0.2313 (6)	0.2831 (3)	0.25488 (15)	0.0327 (8)
C6	0.3161 (7)	0.1922 (4)	0.21200 (17)	0.0469 (10)
H6	0.4434	0.1548	0.2190	0.056*
C7	0.2079 (8)	0.1584 (4)	0.15883 (18)	0.0549 (11)
H7	0.2633	0.0990	0.1291	0.066*
C8	0.0186 (8)	0.2121 (4)	0.14939 (18)	0.0596 (13)
H8	-0.0538	0.1877	0.1135	0.072*
C9	-0.0642 (7)	0.3002 (5)	0.19171 (19)	0.0610 (12)
H9	-0.1935	0.3344	0.1850	0.073*
C10	0.0435 (6)	0.3399 (5)	0.24533 (17)	0.0499 (10)
H10	-0.0102	0.4030	0.2738	0.060*
C11	0.0040 (6)	0.3164 (4)	0.42098 (18)	0.0449 (9)
H11A	-0.0988	0.2870	0.4499	0.067*
H11B	-0.0552	0.3336	0.3804	0.067*
H11C	0.0655	0.3993	0.4364	0.067*
C12	0.6350 (6)	0.0542 (4)	0.34928 (18)	0.0474 (10)
H12A	0.6855	-0.0324	0.3643	0.071*
H12B	0.7340	0.1246	0.3565	0.071*
H12C	0.6077	0.0476	0.3049	0.071*
N1	0.3311 (4)	0.2116 (3)	0.37564 (12)	0.0285 (6)
O1	0.2928 (4)	0.4577 (2)	0.34533 (11)	0.0435 (6)
O2	0.5824 (4)	0.3247 (3)	0.30554 (12)	0.0448 (6)
S1	0.37395 (13)	0.33329 (9)	0.32099 (4)	0.03095 (19)
Br1	-0.00952 (7)	0.02664 (5)	0.50804 (2)	0.05823 (14)
Br2	0.43075 (7)	-0.15500 (4)	0.45930 (2)	0.05195 (13)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0269 (18)	0.0320 (17)	0.0290 (17)	0.0039 (15)	0.0017 (15)	-0.0010 (14)
C2	0.0326 (19)	0.0388 (19)	0.0267 (18)	-0.0042 (16)	0.0028 (15)	-0.0001 (14)
C3	0.0411 (19)	0.0277 (16)	0.0299 (17)	0.0031 (15)	-0.0065 (15)	0.0011 (13)
C4	0.032 (2)	0.0269 (15)	0.0301 (17)	0.0053 (15)	-0.0025 (15)	-0.0039 (13)
C5	0.036 (2)	0.0324 (17)	0.0294 (18)	-0.0045 (16)	-0.0004 (15)	0.0039 (14)
C6	0.063 (3)	0.037 (2)	0.041 (2)	0.006 (2)	0.003 (2)	0.0050 (17)
C7	0.081 (3)	0.042 (2)	0.041 (2)	-0.006 (3)	-0.003 (2)	-0.0039 (19)
C8	0.075 (4)	0.068 (3)	0.036 (2)	-0.025 (3)	-0.008 (2)	0.005 (2)
C9	0.040 (2)	0.100 (4)	0.043 (2)	-0.002 (2)	-0.006 (2)	0.008 (2)
C10	0.039 (2)	0.075 (3)	0.036 (2)	0.004 (2)	0.0026 (17)	-0.0014 (18)
C11	0.046 (2)	0.045 (2)	0.044 (2)	0.014 (2)	0.011 (2)	-0.0015 (16)
C12	0.043 (2)	0.040 (2)	0.059 (2)	0.0122 (19)	0.012 (2)	0.0013 (18)
N1	0.0301 (16)	0.0276 (13)	0.0279 (13)	0.0044 (12)	0.0036 (13)	-0.0001 (12)
O1	0.0573 (17)	0.0265 (12)	0.0466 (15)	0.0032 (13)	0.0010 (13)	-0.0011 (11)
O2	0.0325 (13)	0.0490 (15)	0.0528 (15)	-0.0089 (13)	0.0050 (12)	0.0076 (12)
S1	0.0317 (4)	0.0273 (4)	0.0339 (4)	-0.0018 (4)	0.0008 (4)	0.0021 (3)
Br1	0.0557 (3)	0.0637 (3)	0.0552 (2)	0.0000 (2)	0.0204 (2)	0.0186 (2)
Br2	0.0661 (3)	0.03477 (19)	0.0549 (2)	0.0103 (2)	-0.0033 (2)	0.01168 (17)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C1—C2	1.356 (4)	C7—H7	0.9300
C1—N1	1.412 (4)	C8—C9	1.356 (6)
C1—C11	1.484 (5)	C8—H8	0.9300
C2—C3	1.415 (5)	C9—C10	1.397 (5)
C2—Br1	1.861 (3)	C9—H9	0.9300
C3—C4	1.348 (5)	C10—H10	0.9300
C3—Br2	1.872 (3)	C11—H11A	0.9600
C4—N1	1.420 (4)	C11—H11B	0.9600
C4—C12	1.486 (5)	C11—H11C	0.9600
C5—C10	1.377 (5)	C12—H12A	0.9600
C5—C6	1.386 (5)	C12—H12B	0.9600
C5—S1	1.760 (3)	C12—H12C	0.9600
C6—C7	1.376 (5)	N1—S1	1.680 (3)
C6—H6	0.9300	O1—S1	1.420 (2)
C7—C8	1.373 (7)	O2—S1	1.422 (3)
C2—C1—N1	105.8 (3)	C10—C9—H9	119.8
C2—C1—C11	128.2 (3)	C5—C10—C9	118.1 (4)
N1—C1—C11	126.0 (3)	C5—C10—H10	121.0
C1—C2—C3	109.1 (3)	C9—C10—H10	121.0
C1—C2—Br1	125.3 (3)	C1—C11—H11A	109.5
C3—C2—Br1	125.5 (3)	C1—C11—H11B	109.5
C4—C3—C2	109.9 (3)	H11A—C11—H11B	109.5
C4—C3—Br2	125.4 (3)	C1—C11—H11C	109.5

C2—C3—Br2	124.7 (3)	H11A—C11—H11C	109.5
C3—C4—N1	105.4 (3)	H11B—C11—H11C	109.5
C3—C4—C12	128.5 (3)	C4—C12—H12A	109.5
N1—C4—C12	126.1 (3)	C4—C12—H12B	109.5
C10—C5—C6	121.7 (4)	H12A—C12—H12B	109.5
C10—C5—S1	119.4 (3)	C4—C12—H12C	109.5
C6—C5—S1	118.8 (3)	H12A—C12—H12C	109.5
C7—C6—C5	118.6 (4)	H12B—C12—H12C	109.5
C7—C6—H6	120.7	C1—N1—C4	109.8 (3)
C5—C6—H6	120.7	C1—N1—S1	124.5 (2)
C8—C7—C6	120.3 (4)	C4—N1—S1	125.3 (2)
C8—C7—H7	119.8	O1—S1—O2	120.09 (18)
C6—C7—H7	119.8	O1—S1—N1	106.52 (14)
C9—C8—C7	120.9 (4)	O2—S1—N1	106.36 (15)
C9—C8—H8	119.6	O1—S1—C5	108.82 (17)
C7—C8—H8	119.6	O2—S1—C5	108.76 (17)
C8—C9—C10	120.4 (4)	N1—S1—C5	105.30 (15)
C8—C9—H9	119.8		
N1—C1—C2—C3	0.2 (4)	C2—C1—N1—C4	0.4 (3)
C11—C1—C2—C3	-178.7 (3)	C11—C1—N1—C4	179.3 (3)
N1—C1—C2—Br1	177.2 (2)	C2—C1—N1—S1	172.8 (2)
C11—C1—C2—Br1	-1.7 (5)	C11—C1—N1—S1	-8.3 (5)
C1—C2—C3—C4	-0.8 (4)	C3—C4—N1—C1	-0.9 (3)
Br1—C2—C3—C4	-177.8 (2)	C12—C4—N1—C1	178.9 (3)
C1—C2—C3—Br2	178.1 (2)	C3—C4—N1—S1	-173.2 (2)
Br1—C2—C3—Br2	1.1 (4)	C12—C4—N1—S1	6.5 (5)
C2—C3—C4—N1	1.0 (4)	C1—N1—S1—O1	33.9 (3)
Br2—C3—C4—N1	-177.9 (2)	C4—N1—S1—O1	-154.8 (3)
C2—C3—C4—C12	-178.7 (3)	C1—N1—S1—O2	163.1 (3)
Br2—C3—C4—C12	2.4 (5)	C4—N1—S1—O2	-25.6 (3)
C10—C5—C6—C7	-0.2 (6)	C1—N1—S1—C5	-81.6 (3)
S1—C5—C6—C7	-176.9 (3)	C4—N1—S1—C5	89.7 (3)
C5—C6—C7—C8	-1.2 (6)	C10—C5—S1—O1	-16.9 (3)
C6—C7—C8—C9	0.8 (6)	C6—C5—S1—O1	159.8 (3)
C7—C8—C9—C10	1.1 (7)	C10—C5—S1—O2	-149.4 (3)
C6—C5—C10—C9	2.0 (6)	C6—C5—S1—O2	27.4 (3)
S1—C5—C10—C9	178.6 (3)	C10—C5—S1—N1	96.9 (3)
C8—C9—C10—C5	-2.4 (7)	C6—C5—S1—N1	-86.3 (3)

Hydrogen-bond geometry (Å, °)

Cg2 is the centroid of the C5—C10 ring.

<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>
C10—H10...O1	0.93	2.57	2.922 (5)	103
C11—H11A...Br1	0.96	2.88	3.368 (4)	113
C11—H11C...O1	0.96	2.51	2.849 (5)	100
C12—H12A...Br2	0.96	2.88	3.378 (4)	113

## supporting information

---

C12—H12B···O2	0.96	2.44	2.809 (5)	102
C12—H12A···Cg <sup>2i</sup>	0.96	2.85	3.545 (7)	130

---

Symmetry code: (i)  $-x+1, y-1/2, -z+1/2$ .