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4-(5-*tert*-Butyl-1,3-dithian-2-yl)-5-chloro-2-phenyl-1,3-oxazole

Yongliang Cui, Lei Liu, Ming Liu, Yousheng Duan and Shangzhong Liu*

Department of Applied Chemistry, China Agriculture University, 100094 Beijing, People's Republic of China

Correspondence e-mail: shangzho@cau.edu.cn

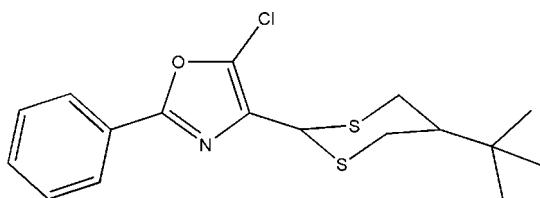
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.050; wR factor = 0.131; data-to-parameter ratio = 15.8.

In the title molecule, $\text{C}_{17}\text{H}_{20}\text{ClNOS}_2$, the phenyl and oxazole rings are nearly coplanar with an average deviation of 0.022 Å from the mean plane (M). The 1,3-dithiane ring adopts a chair conformation and is twisted in such a way that the $\text{C}-\text{C}_{\text{Bu}}$ fragment lies in M (deviations are 0.031 and 0.010 Å, respectively, for the two C atoms).

Related literature

For details of the pharmacological properties of the GABA [GABA = γ -aminobutyric acid] receptor, see: Wachter *et al.* (1992). For the related structural series of the GABA receptor, see: Jeffrey (2003); Naratashi *et al.* (2007).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{20}\text{ClNOS}_2$	$V = 1792.7 (6) \text{ \AA}^3$
$M_r = 353.91$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 7.4543 (15) \text{ \AA}$	$\mu = 0.45 \text{ mm}^{-1}$
$b = 26.222 (5) \text{ \AA}$	$T = 296 (2) \text{ K}$
$c = 9.4772 (19) \text{ \AA}$	$0.34 \times 0.31 \times 0.18 \text{ mm}$
$\beta = 104.59 (3)^\circ$	

Data collection

Rigaku R-Axis RAPID IP area-detector diffractometer	5683 measured reflections
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	3135 independent reflections
$T_{\text{min}} = 0.863$, $T_{\text{max}} = 0.924$	2788 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$	59 restraints
$wR(F^2) = 0.130$	H-atom parameters constrained
$S = 1.10$	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
3135 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
199 parameters	

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in Siemens *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2381).

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4-(5-*tert*-Butyl-1,3-dithian-2-yl)-5-chloro-2-phenyl-1,3-oxazole

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S1. Comment

γ -Aminobutyric acid (GABA) receptor of insect exists in their nerve cell and intramuscular cell, and a combinative site of many insecticide and active compounds (Wacher *et al.*, 1992). A large number of related structural series (*e.g.*, trioxabicyclooctanes, thiazines, arylpyrimidines, oxathianes, and dithianes) was synthesized and assayed on GABA receptor to discover novel insecticides (Jeffrey, 2003). Until now, only synthetic compound Fipronil is broadly used to control certain species of insects that have become resistant to most insecticides (Naratashi *et al.*, 2007). In order to further optimize 1,3-dithiane derivative, the title compound, (I), was synthesized. Herewith we present its crystal structure.

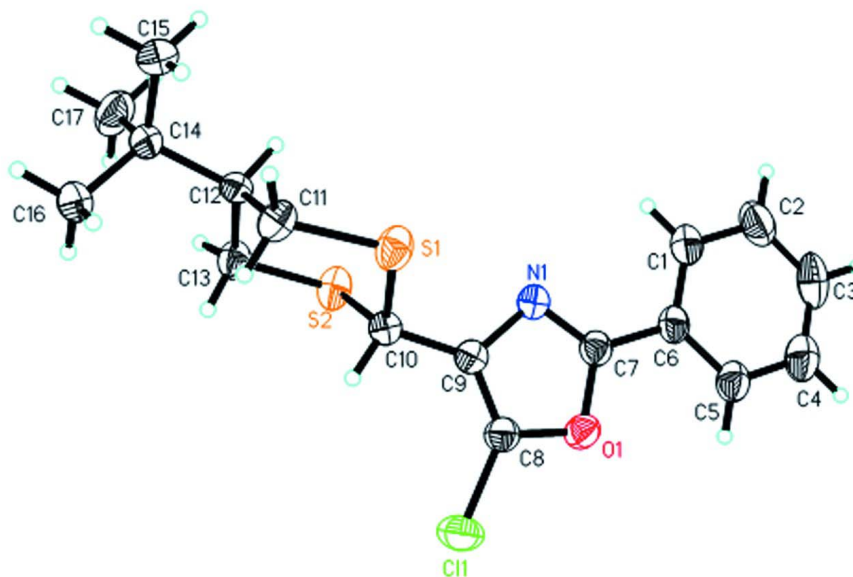
In the title molecule, the phenyl and oxazole rings are nearly coplanar with the average deviation of 0.022 Å from the mean plane (*M*). The 1,3-dithiane ring adopts a chair conformation being twisted in such a way, that two-atomic fragment C12—C14 actually lie in *M* with deviations of 0.031 and 0.010 Å, respectively. The crystal structure exhibits no classical hydrogen bonds.

S2. Experimental

Compound (I) was prepared by the 4 h reaction of 0.8 g (3.85 mmol) of 5-chloro-2-phenyloxazole-4-carbaldehyde and 0.8 g (4.88 mmol) of 2-*tert*-butylpropane-1,3-dithiol in the presence of two drops formic acid used as a catalyst at room temperature with stirring. The resulting mixture was dissolved in chloroform (60 ml), washed with aqueous 10% NaOH (3×20 ml) and H₂O (3×20 ml), and then dried with anhydrous sodium sulfate. After concentration, the residue was purified by re-crystallization in a mixed solvent of ethyl acetate and petroleum ether. Single crystals suitable for X-ray data collection were obtained by re-crystallization of the crude product from a mixed solvent ethyl acetate and petroleum ether (*v/v*, 1/20) as a light yellow crystalline solid (60%), m.p. 453 K.

S3. Refinement

The H atoms were positioned with idealized geometry (C—H 0.93–0.98 Å), and refined using a riding model with $U_{\text{iso}}(\text{H})=1.2$ or $1.5U_{\text{eq}}(\text{C})$.

**Figure 1**

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

4-(5-*tert*-Butyl-1,3-dithian-2-yl)-5-chloro-2-phenyl-1,3-oxazole ?

Crystal data

$C_{17}H_{20}ClNOS_2$

$M_r = 353.91$

Monoclinic, $P2_1/c$

$a = 7.4543$ (15) Å

$b = 26.222$ (5) Å

$c = 9.4772$ (19) Å

$\beta = 104.59$ (3)°

$V = 1792.7$ (6) Å³

$Z = 4$

$F(000) = 744$

$D_x = 1.311$ Mg m⁻³

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

Cell parameters from 13320 reflections

$\theta = 2.2$ – 27.5 °

$\mu = 0.45$ mm⁻¹

$T = 296$ K

Plate, colourless

$0.34 \times 0.31 \times 0.18$ mm

Data collection

Rigaku R-AXIS RAPID IP area-detector
diffractometer

Radiation source: rotating anode

Graphite monochromator

ω scans at fixed $\chi = 45$ °

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.863$, $T_{\max} = 0.924$

5683 measured reflections

3135 independent reflections

2788 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 25.0$ °, $\theta_{\min} = 2.7$ °

$h = -8 \rightarrow 8$

$k = -30 \rightarrow 31$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.130$
 $S = 1.10$
 3135 reflections
 199 parameters
 59 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
 $w = 1/[\sigma^2(F_o^2) + (0.0651P)^2 + 0.55P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.37 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.21531 (9)	0.68715 (2)	0.75838 (9)	0.0672 (2)
S2	0.56576 (9)	0.74615 (2)	0.89160 (8)	0.0656 (2)
C11	0.74022 (11)	0.62257 (3)	0.61190 (8)	0.0840 (3)
O1	0.7196 (2)	0.57469 (6)	0.84876 (18)	0.0576 (4)
N1	0.5427 (3)	0.62247 (7)	0.9549 (2)	0.0541 (5)
C1	0.6072 (4)	0.54919 (11)	1.1975 (3)	0.0732 (7)
H1B	0.5353	0.5776	1.2042	0.088*
C2	0.6441 (5)	0.51328 (13)	1.3080 (4)	0.0923 (10)
H2A	0.5966	0.5174	1.3891	0.111*
C3	0.7516 (6)	0.47136 (13)	1.2970 (4)	0.0978 (11)
H3A	0.7789	0.4476	1.3723	0.117*
C4	0.8183 (5)	0.46438 (12)	1.1775 (4)	0.0869 (9)
H4A	0.8881	0.4355	1.1703	0.104*
C5	0.7828 (4)	0.49976 (10)	1.0678 (3)	0.0685 (7)
H5A	0.8293	0.4950	0.9865	0.082*
C6	0.6776 (3)	0.54266 (9)	1.0775 (3)	0.0557 (6)
C7	0.6401 (3)	0.58120 (9)	0.9633 (3)	0.0530 (5)
C8	0.6628 (3)	0.61672 (10)	0.7651 (3)	0.0567 (6)
C9	0.5551 (3)	0.64573 (9)	0.8256 (2)	0.0523 (5)
C10	0.4602 (3)	0.69455 (9)	0.7721 (3)	0.0523 (5)
H10A	0.4767	0.7014	0.6744	0.063*
C11	0.1332 (4)	0.75009 (9)	0.6948 (3)	0.0619 (6)
H11A	0.1585	0.7558	0.6006	0.074*
H11B	-0.0002	0.7511	0.6810	0.074*
C12	0.2190 (3)	0.79347 (9)	0.7963 (2)	0.0490 (5)

H12A	0.2133	0.7835	0.8948	0.059*
C13	0.4239 (3)	0.79820 (9)	0.7989 (3)	0.0598 (6)
H13A	0.4711	0.8300	0.8465	0.072*
H13B	0.4354	0.7999	0.6993	0.072*
C14	0.1074 (4)	0.84396 (9)	0.7598 (3)	0.0573 (6)
C15	-0.0878 (4)	0.83563 (13)	0.7799 (4)	0.0839 (9)
H15A	-0.1582	0.8665	0.7568	0.126*
H15B	-0.0799	0.8263	0.8793	0.126*
H15C	-0.1478	0.8088	0.7163	0.126*
C16	0.0952 (4)	0.86200 (12)	0.6043 (3)	0.0718 (8)
H16A	0.0248	0.8930	0.5861	0.108*
H16B	0.0353	0.8363	0.5365	0.108*
H16C	0.2177	0.8680	0.5927	0.108*
C17	0.2000 (5)	0.88590 (11)	0.8662 (4)	0.0872 (9)
H17A	0.1309	0.9170	0.8433	0.131*
H17B	0.3244	0.8912	0.8580	0.131*
H17C	0.2026	0.8758	0.9641	0.131*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0550 (4)	0.0493 (4)	0.0934 (5)	-0.0030 (3)	0.0113 (3)	-0.0006 (3)
S2	0.0556 (4)	0.0521 (4)	0.0797 (5)	-0.0014 (3)	-0.0003 (3)	0.0031 (3)
C11	0.0849 (5)	0.1090 (6)	0.0667 (4)	0.0281 (4)	0.0352 (4)	0.0140 (4)
O1	0.0560 (9)	0.0558 (10)	0.0616 (9)	0.0092 (7)	0.0160 (8)	-0.0001 (8)
N1	0.0581 (11)	0.0478 (11)	0.0585 (11)	0.0033 (9)	0.0184 (9)	0.0039 (9)
C1	0.089 (2)	0.0544 (15)	0.0798 (18)	-0.0037 (14)	0.0279 (15)	0.0077 (13)
C2	0.127 (3)	0.077 (2)	0.078 (2)	-0.0156 (19)	0.0358 (19)	0.0161 (16)
C3	0.120 (3)	0.066 (2)	0.095 (2)	-0.0088 (19)	0.004 (2)	0.0311 (18)
C4	0.089 (2)	0.0623 (18)	0.103 (2)	0.0064 (16)	0.0135 (18)	0.0195 (17)
C5	0.0613 (15)	0.0566 (15)	0.0855 (18)	0.0035 (12)	0.0146 (13)	0.0100 (13)
C6	0.0536 (13)	0.0455 (12)	0.0659 (14)	-0.0071 (10)	0.0112 (11)	0.0033 (11)
C7	0.0498 (12)	0.0492 (13)	0.0595 (13)	-0.0037 (10)	0.0128 (10)	0.0019 (10)
C8	0.0568 (14)	0.0595 (15)	0.0539 (13)	0.0065 (11)	0.0144 (11)	0.0032 (11)
C9	0.0535 (13)	0.0508 (13)	0.0535 (12)	0.0025 (10)	0.0152 (10)	0.0020 (10)
C10	0.0563 (13)	0.0508 (13)	0.0518 (12)	0.0038 (10)	0.0174 (10)	0.0038 (10)
C11	0.0567 (14)	0.0566 (15)	0.0679 (16)	0.0077 (11)	0.0074 (12)	-0.0025 (12)
C12	0.0570 (13)	0.0518 (12)	0.0415 (11)	0.0058 (10)	0.0185 (9)	0.0037 (9)
C13	0.0595 (14)	0.0468 (13)	0.0719 (16)	-0.0006 (11)	0.0145 (12)	0.0041 (12)
C14	0.0698 (15)	0.0535 (14)	0.0535 (13)	0.0113 (11)	0.0246 (11)	0.0070 (10)
C15	0.0820 (19)	0.082 (2)	0.103 (2)	0.0264 (16)	0.0518 (18)	0.0230 (18)
C16	0.0836 (19)	0.0727 (18)	0.0630 (15)	0.0201 (15)	0.0259 (14)	0.0213 (13)
C17	0.119 (3)	0.0591 (17)	0.0819 (19)	0.0193 (17)	0.0230 (18)	-0.0095 (14)

Geometric parameters (Å, °)

S1—C10	1.808 (3)	C9—C10	1.489 (3)
S1—C11	1.810 (3)	C10—H10A	0.9800

S2—C10	1.812 (2)	C11—C12	1.523 (3)
S2—C13	1.813 (3)	C11—H11A	0.9700
C11—C8	1.699 (3)	C11—H11B	0.9700
O1—C8	1.362 (3)	C12—C13	1.526 (3)
O1—C7	1.372 (3)	C12—C14	1.556 (3)
N1—C7	1.294 (3)	C12—H12A	0.9800
N1—C9	1.393 (3)	C13—H13A	0.9700
C1—C6	1.378 (4)	C13—H13B	0.9700
C1—C2	1.383 (4)	C14—C16	1.528 (3)
C1—H1B	0.9300	C14—C15	1.530 (4)
C2—C3	1.380 (5)	C14—C17	1.534 (4)
C2—H2A	0.9300	C15—H15A	0.9600
C3—C4	1.359 (5)	C15—H15B	0.9600
C3—H3A	0.9300	C15—H15C	0.9600
C4—C5	1.369 (4)	C16—H16A	0.9600
C4—H4A	0.9300	C16—H16B	0.9600
C5—C6	1.387 (4)	C16—H16C	0.9600
C5—H5A	0.9300	C17—H17A	0.9600
C6—C7	1.455 (3)	C17—H17B	0.9600
C8—C9	1.335 (3)	C17—H17C	0.9600
C10—S1—C11	100.18 (12)	C12—C11—H11B	108.6
C10—S2—C13	98.67 (12)	S1—C11—H11B	108.6
C8—O1—C7	103.14 (18)	H11A—C11—H11B	107.6
C7—N1—C9	105.1 (2)	C11—C12—C13	109.28 (19)
C6—C1—C2	119.7 (3)	C11—C12—C14	112.3 (2)
C6—C1—H1B	120.1	C13—C12—C14	114.3 (2)
C2—C1—H1B	120.1	C11—C12—H12A	106.9
C3—C2—C1	119.6 (3)	C13—C12—H12A	106.9
C3—C2—H2A	120.2	C14—C12—H12A	106.9
C1—C2—H2A	120.2	C12—C13—S2	113.93 (17)
C4—C3—C2	120.8 (3)	C12—C13—H13A	108.8
C4—C3—H3A	119.6	S2—C13—H13A	108.8
C2—C3—H3A	119.6	C12—C13—H13B	108.8
C3—C4—C5	120.0 (3)	S2—C13—H13B	108.8
C3—C4—H4A	120.0	H13A—C13—H13B	107.7
C5—C4—H4A	120.0	C16—C14—C15	109.7 (2)
C4—C5—C6	120.2 (3)	C16—C14—C17	108.7 (2)
C4—C5—H5A	119.9	C15—C14—C17	107.7 (2)
C6—C5—H5A	119.9	C16—C14—C12	112.15 (19)
C1—C6—C5	119.6 (3)	C15—C14—C12	108.8 (2)
C1—C6—C7	119.0 (2)	C17—C14—C12	109.7 (2)
C5—C6—C7	121.3 (2)	C14—C15—H15A	109.5
N1—C7—O1	113.7 (2)	C14—C15—H15B	109.5
N1—C7—C6	128.8 (2)	H15A—C15—H15B	109.5
O1—C7—C6	117.5 (2)	C14—C15—H15C	109.5
C9—C8—O1	110.1 (2)	H15A—C15—H15C	109.5
C9—C8—C11	133.3 (2)	H15B—C15—H15C	109.5

O1—C8—C11	116.62 (18)	C14—C16—H16A	109.5
C8—C9—N1	108.0 (2)	C14—C16—H16B	109.5
C8—C9—C10	129.0 (2)	H16A—C16—H16B	109.5
N1—C9—C10	123.0 (2)	C14—C16—H16C	109.5
C9—C10—S1	108.47 (16)	H16A—C16—H16C	109.5
C9—C10—S2	109.57 (17)	H16B—C16—H16C	109.5
S1—C10—S2	113.36 (13)	C14—C17—H17A	109.5
C9—C10—H10A	108.4	C14—C17—H17B	109.5
S1—C10—H10A	108.4	H17A—C17—H17B	109.5
S2—C10—H10A	108.4	C14—C17—H17C	109.5
C12—C11—S1	114.70 (17)	H17A—C17—H17C	109.5
C12—C11—H11A	108.6	H17B—C17—H17C	109.5
S1—C11—H11A	108.6		
C6—C1—C2—C3	0.3 (5)	C7—N1—C9—C8	-0.8 (3)
C1—C2—C3—C4	-1.5 (6)	C7—N1—C9—C10	179.2 (2)
C2—C3—C4—C5	1.6 (6)	C8—C9—C10—S1	124.2 (3)
C3—C4—C5—C6	-0.5 (5)	N1—C9—C10—S1	-55.9 (3)
C2—C1—C6—C5	0.8 (4)	C8—C9—C10—S2	-111.6 (3)
C2—C1—C6—C7	-179.0 (3)	N1—C9—C10—S2	68.4 (3)
C4—C5—C6—C1	-0.7 (4)	C11—S1—C10—C9	179.42 (16)
C4—C5—C6—C7	179.1 (3)	C11—S1—C10—S2	57.48 (16)
C9—N1—C7—O1	0.4 (3)	C13—S2—C10—C9	179.80 (17)
C9—N1—C7—C6	178.9 (2)	C13—S2—C10—S1	-58.88 (15)
C8—O1—C7—N1	0.2 (3)	C10—S1—C11—C12	-59.4 (2)
C8—O1—C7—C6	-178.5 (2)	S1—C11—C12—C13	67.9 (2)
C1—C6—C7—N1	-1.5 (4)	S1—C11—C12—C14	-164.19 (16)
C5—C6—C7—N1	178.6 (2)	C11—C12—C13—S2	-70.4 (2)
C1—C6—C7—O1	177.0 (2)	C14—C12—C13—S2	162.84 (16)
C5—C6—C7—O1	-2.9 (3)	C10—S2—C13—C12	63.2 (2)
C7—O1—C8—C9	-0.7 (3)	C11—C12—C14—C16	-59.6 (3)
C7—O1—C8—C11	178.63 (17)	C13—C12—C14—C16	65.6 (3)
O1—C8—C9—N1	1.0 (3)	C11—C12—C14—C15	62.0 (3)
C11—C8—C9—N1	-178.2 (2)	C13—C12—C14—C15	-172.8 (2)
O1—C8—C9—C10	-179.1 (2)	C11—C12—C14—C17	179.6 (2)
C11—C8—C9—C10	1.8 (4)	C13—C12—C14—C17	-55.2 (3)