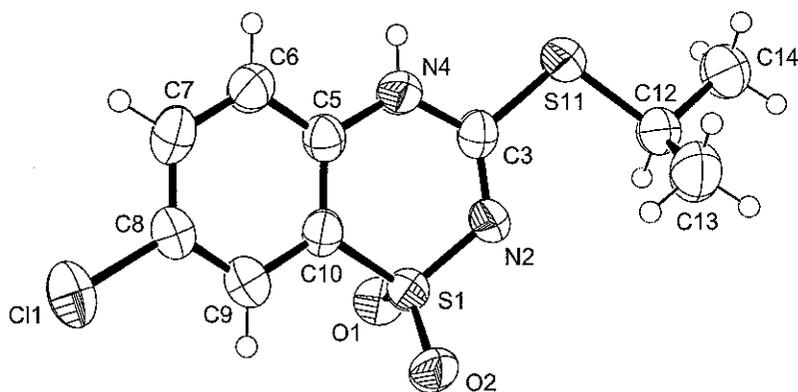


# Crystal structure of 7-chloro-3-isopropylsulfanyl-4*H*-1,2,4-benzothiadiazine 1,1-dioxide, C<sub>10</sub>H<sub>11</sub>ClN<sub>2</sub>O<sub>2</sub>S<sub>2</sub>

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## Abstract

C<sub>10</sub>H<sub>11</sub>ClN<sub>2</sub>O<sub>2</sub>S<sub>2</sub>, orthorhombic, *Pcab* (no 61),  
 $a = 8.5908(5) \text{ \AA}$ ,  $b = 12.0056(8) \text{ \AA}$ ,  $c = 24.213(1) \text{ \AA}$ ,  
 $V = 2497.3 \text{ \AA}^3$ ,  $Z = 8$ ,  $R_{\text{gt}}(F) = 0.058$ ,  $wR_{\text{ref}}(F^2) = 0.169$ ,  
 $T = 293 \text{ K}$ .

## Source of material

The compound was synthesized by alkylation of 7-chloro-3-thio-oxo-3,4-dihydro-4*H*-1,2,4-benzothiadiazine 1,1-dioxide, a well known synthetic intermediate [1] with isopropyl iodide. Crystals were obtained by slow evaporation of a methanol solution.

## Discussion

7-Chloro-3-isopropylsulfanyl-4*H*-1,2,4-benzothiadiazine 1,1-dioxide (BPDZ209), is an old compound developed initially as antimicrobial drug [2]. As it is structurally related to BPDZ73 (7-chloro-3-isopropylamino-4*H*-1,2,4-benzothiadiazine 1,1-dioxide), a reference pancreatic ATP sensitive potassium channel opener (K<sub>ATP</sub> PCO) [1,3], it was recently evaluated as putative K<sub>ATP</sub> opener. The isosteric change of the 3-isopropylamino chain of BPDZ73 by a 3-isopropylsulfanyl side chain led to an important change in the pharmacological profile of the drug. The X-ray analysis of BPDZ209 demonstrated that the N4—C3 distance [1.360(5) Å] corresponds to a single bond type whereas the double bond character of N2—C3 [1.310(5) Å] is well defined. The 4*H*- rather than the 2*H*-tautomeric form is so exhibited in the crystalline state of BPDZ209 like in BPDZ73 and also in many benzothiadiazine 1,1-dioxides previously studied. The presence of an N4—H · O hydrogen bond and the absence of a meaningful peak near N2 in the Fourier difference map confirm this scheme. The geometry of the hydrogen bond N4—H4 · O1<sup>i</sup> [symmetry code (i) ½+x, ½-y, z], is characterized by the N4 · O2<sup>i</sup> distance of 2.826(4) Å and the N4—H4—O2<sup>i</sup> angle of 174(4)°. The

C3—S11—C12—C13 torsion angle is equal to  $-72.3(4)^\circ$ . The corresponding value in BPDZ73 is  $-70.7(5)^\circ$ . The values of the torsion angles S1—N2—C3—N4 ( $8.5(6)^\circ$ ) and C10—S1—N2—C3 ( $-24.1(4)^\circ$ ) show that the conformation of the heterocycle ring is less planar in BPDZ209 than in BPDZ73 where the relevant angles values are  $4.8(5)^\circ$  and  $-12.3(3)^\circ$ , respectively.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.11 × 0.46 × 0.51 mm
Wavelength:	Cu K $\alpha$ radiation (1.54180 Å)
$\mu$ :	57.79 cm <sup>-1</sup>
Diffractometer, scan mode:	Stoe-Siemens AED, $\omega$
$2\theta_{\text{max}}$ :	136.1°
$N(hkl)_{\text{measured}}$ , $N(hkl)_{\text{unique}}$ :	2242, 2242
Criterion for $I_{\text{obs}}$ , $N(hkl)_{\text{gt}}$ :	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$ , 1463
$N(\text{param})_{\text{refined}}$ :	157
Programs:	SHELXS-97 [4] SHELXL-97 [5], ORTEP-III [6]

Table 2. Atomic coordinates and displacement parameters (in Å<sup>2</sup>).

Atom	Site	x	y	z	$U_{\text{iso}}$
H(4)	8c	0.4165	0.2924	0.4052	0.064
H(6)	8c	0.4850	0.2892	0.4993	0.067
H(7)	8c	0.4789	0.1962	0.5836	0.071
H(9)	8c	0.2331	-0.0512	0.5154	0.061
H(12)	8c	0.2404	0.1347	0.2453	0.070
H(13A)	8c	0.4289	0.0158	0.2086	0.125
H(13B)	8c	0.4372	0.0159	0.2733	0.125
H(13C)	8c	0.5594	0.0837	0.2389	0.125
H(14A)	8c	0.4728	0.2585	0.1885	0.114
H(14B)	8c	0.2975	0.2937	0.1926	0.114
H(14C)	8c	0.3447	0.1882	0.1584	0.114

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**Table 3.** Atomic coordinates and displacement parameters (in Å<sup>2</sup>)

Atom	Site	x	y	z	U <sub>11</sub>	U <sub>22</sub>	U <sub>33</sub>	U <sub>12</sub>	U <sub>13</sub>	U <sub>23</sub>
S(1)	8c	0 1789(1)	0 02418(7)	0 40516(4)	0 0490(5)	0 0423(5)	0 0426(5)	-0 0005(4)	0 0000(4)	0 0019(4)
O(1)	8c	0 0215(3)	0 0626(2)	0 4110(1)	0 048(1)	0 058(2)	0 063(2)	0 003(1)	-0 002(1)	0 002(1)
O(2)	8c	0 1991(4)	-0 0932(2)	0 4006(1)	0 082(2)	0 036(1)	0 057(2)	0 002(1)	-0 001(2)	-0 002(1)
N(2)	8c	0 2550(4)	0 0843(3)	0 3525(1)	0 064(2)	0 055(2)	0 036(1)	-0 014(2)	0 003(2)	0 004(1)
C(3)	8c	0 3292(5)	0 1792(3)	0 3569(2)	0 054(2)	0 052(2)	0 039(2)	0 005(2)	-0 001(2)	-0 000(2)
N(4)	8c	0 3669(4)	0 2299(3)	0 4053(1)	0 072(2)	0 042(2)	0 046(2)	-0 006(2)	-0 002(2)	0 002(1)
C(5)	8c	0 3591(4)	0 1760(3)	0 4567(2)	0 050(2)	0 047(2)	0 044(2)	0 005(2)	0 001(2)	0 002(2)
C(6)	8c	0 4335(6)	0 2214(3)	0 5024(2)	0 069(2)	0 048(2)	0 050(2)	-0 001(2)	-0 002(2)	-0 004(2)
C(7)	8c	0 4310(5)	0 1652(4)	0 5528(2)	0 069(2)	0 064(3)	0 044(2)	0 008(2)	-0 008(2)	-0 008(2)
C(8)	8c	0 3569(5)	0 0633(4)	0 5566(2)	0 050(2)	0 056(2)	0 041(2)	0 005(2)	0 003(2)	0 006(2)
C(9)	8c	0 2831(5)	0 0172(3)	0 5123(2)	0 048(2)	0 057(3)	0 047(2)	0 003(2)	0 004(2)	0 006(2)
C(10)	8c	0 2837(4)	0 0749(3)	0 4615(1)	0 051(2)	0 046(2)	0 040(2)	0 005(2)	-0 000(2)	-0 002(2)
S(11)	8c	0 3948(2)	0 2517(1)	0 29941(4)	0 0840(8)	0 0556(6)	0 0468(5)	-0 0171(6)	0 0042(5)	0 0046(4)
C(12)	8c	0 3489(5)	0 1591(4)	0 2423(2)	0 072(3)	0 058(3)	0 043(2)	-0 008(2)	-0 002(2)	0 001(2)
C(13)	8c	0 4528(8)	0 0598(4)	0 2406(2)	0 117(4)	0 071(3)	0 061(3)	0 001(3)	0 007(3)	-0 001(2)
C(14)	8c	0 3678(7)	0 2317(4)	0 1906(2)	0 105(4)	0 074(3)	0 049(2)	-0 010(3)	-0 003(3)	0 010(2)
Cl(1)	8c	0 3590(2)	-0 0069(1)	0 61909(4)	0 0688(7)	0 0874(9)	0 0445(5)	0 0099(6)	-0 0005(5)	0 0128(5)

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