



Crystal structures of 2,3-bis(4-chlorophenyl)-1,3-thiazolidin-4-one and *trans*-2,3-bis(4-chlorophenyl)-1,3-thiazolidin-4-one 1-oxide

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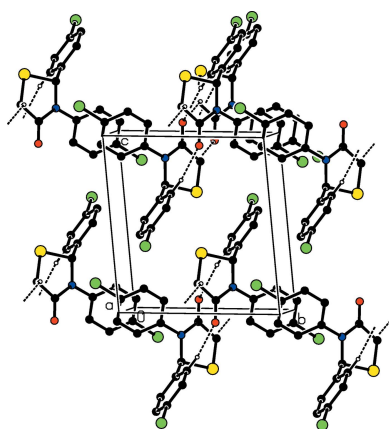
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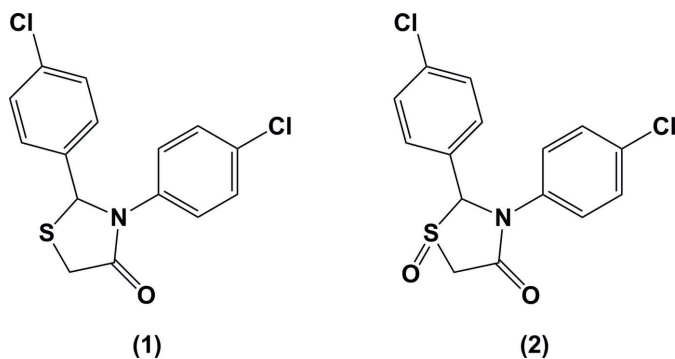
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In the crystal structures of the title compounds, C₁₅H₁₁Cl₂NOS, (1), and C₁₅H₁₁Cl₂NO₂S, (2), wherein (2) is the oxidized form of (1), the thiazolidine ring is attached to two chlorophenyl rings. The chlorophenyl ring on the 2-carbon atom position points in the same direction as that of the S atom in (1), while in (2), the S atom points in the opposite direction. The O atom on the chiral S atom in (2) is *trans* to the chlorophenyl ring on the 2-carbon. The chlorophenyl ring planes in each structure are close to orthogonal, making dihedral angles of 78.61 (6) and 87.46 (8)° in (1) and (2), respectively. The thiazolidine ring has a twisted conformation on the S—C_{methine} bond in (1), and an envelope conformation with the S atom 0.715 (3) Å out of the plane of other four atoms in (2). In the crystal of (1), molecules are linked by C—H...O hydrogen bonds, as well as by slipped parallel π - π interactions [inter-centroid distance = 3.840 (3) Å] between inversion-related phenyl rings, forming sheets parallel to (001). In the crystal of (2), molecules are linked *via* C—H...O and C—H...Cl hydrogen bonds, forming slabs parallel to (001).

1. Chemical context

1,3-Thiazolidin-4-ones, also known as 4-thiazolidinones, are known to have a wide range of biological activities (Jain *et al.*, 2012; Abhinit *et al.*, 2009; Hamama *et al.*, 2008; Singh *et al.*, 1981; Brown, 1961; Tripathi *et al.*, 2014; Prabhakar *et al.*, 2006). The *S*-oxides have been observed to show enhanced activity, for example, it was shown that on converting a 4-thiazolidinone to its sulfoxide and sulfone, the oxide showed greater activity against some cancer cell lines than the sulfide (Gududuru *et al.*, 2004). Oxidation from sulfide to sulfoxide makes the sulfur a chiral center, and produces *cis* and *trans* diastereomers with regard to the relationship of the oxygen atom attached to the S atom and the substituent at the 2-position (Rozwadowska *et al.*, 2002; Colombo *et al.*, 2008). The stereocenters may however be configurationally unstable in solution or even in the solid state (Rozwadowska *et al.*, 2002). We have previously reported on the preparation and NMR studies of a series of 2,3-diaryl-1,3-thiazolidin-4-ones in which the two aryl groups had the same substitution pattern (Tierney *et al.*, 2005). In this study, we report on the *S*-oxidation of one of these compounds, 2,3-bis(4-chlorophenyl)-1,3-thiazolidin-4-one (1), with Oxone (Trost & Curran, 1981; Yu *et al.*, 2012; Webb, 1994), which gave compound (2), and on their crystal structures.





2. Structural commentary

The molecular structures of compounds (1) and (2), Figs. 1 and 2, respectively, show a slight dissimilarity in the thiazolidine ring conformation. In (1), the ring pucker is twisted on the S1–C1 bond, while in (2) the ring has an envelope conformation with atom S1 as the flap. The structures also differ in the disposition of the chlorophenyl ring at atom C1. In (1), this ring points in the same direction as the S atom with respect to the thiazolidine ring plane, while in (2), the S atom points in the opposite direction. The *trans* relationship between the oxygen atom on the S atom and the aromatic ring on C1 is favoured due to steric hindrance which would occur in the *cis* isomer. The chlorophenyl rings are almost orthogonal to each other, making a dihedral angle of 78.61 (6)° in (1) and 87.46 (8)° in (2).

Comparison of the two structures shows that the oxygen–sulfur bond in (2) formed on the less hindered side of compound (1), away from the aryl group on C1, leading to a *trans* stereoisomer. Steric strain was further relieved by twisting so that both the aryl ring on C1 and the oxygen on S1 became pseudo-axial.

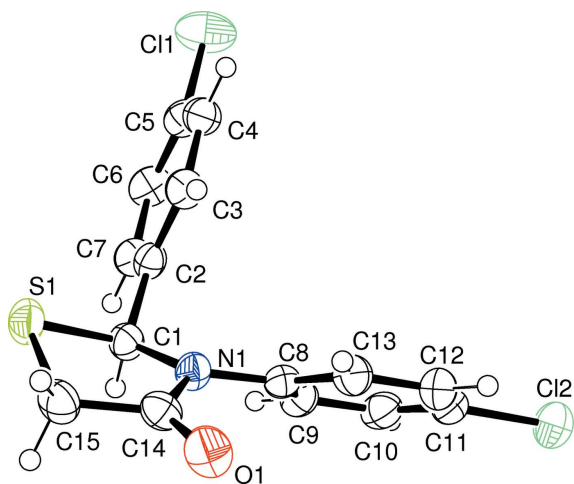


Figure 1
A view of the molecular structure of compound (1), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

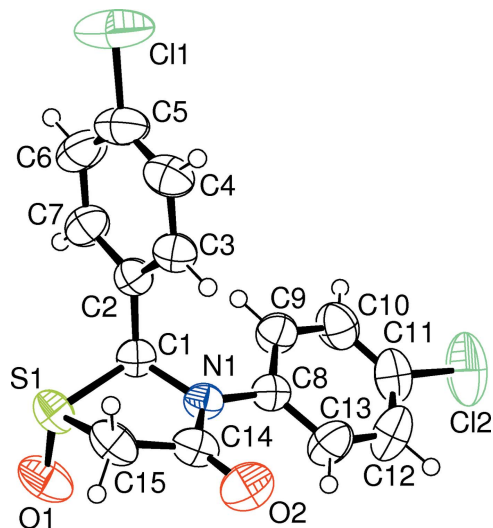


Figure 2
A view of the molecular structure of compound (2), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

3. Supramolecular features

In the crystal of (1), molecules are linked *via* C–H...O hydrogen bonds, forming chains along [100]; see Table 1 and Fig. 3. The chains are linked *via* slipped parallel π – π interactions involving inversion-related chlorophenyl rings, leading to the formation of sheets parallel to (001) [$Cg3 \cdots Cg3^i = 3.840$ (3) Å; $Cg3$ is the centroid of the C8–C13 ring; inter-

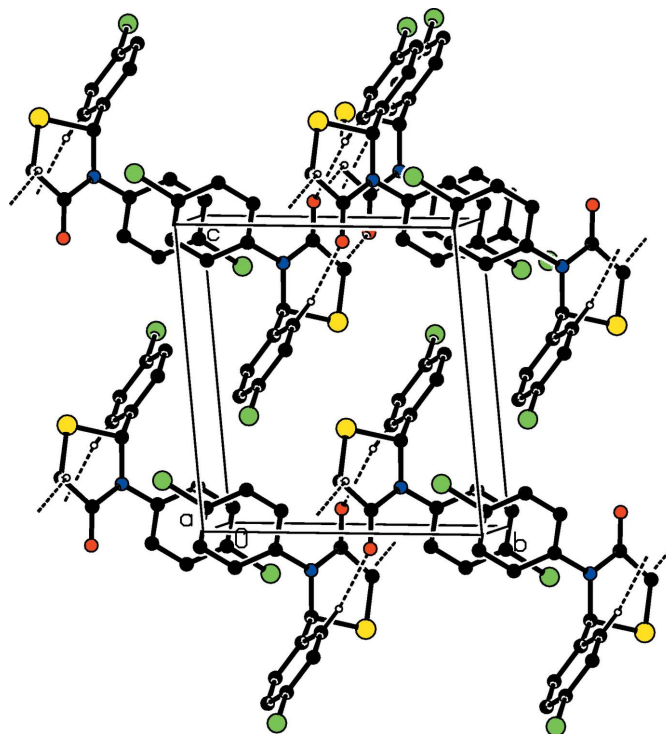


Figure 3
Crystal packing of compound (1) viewed along the *a* axis, showing the hydrogen bonds as dashed lines (see Table 1 for details; H atoms not involved in these interactions have been omitted for clarity).

Table 1

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

<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>
C3—H3...O1 ⁱ	0.93	2.48	3.326 (3)	151
C15—H15B...O1 ⁱⁱ	0.97	2.46	3.221 (3)	135

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

Table 2

Hydrogen-bond geometry (Å, °) for (2).

<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>
C1—H1...O1 ⁱ	0.98	2.19	3.154 (3)	167
C6—H6...Cl2 ⁱⁱ	0.93	2.83	3.676 (3)	152

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

planar distance = 3.3364 (7) Å; slippage = 1.901 Å; symmetry code: (i) $-x + 2, -y, -z + 2$].

In the crystal of (2), molecules are linked *via* by C—H...O and C—H...Cl hydrogen bonds, forming slabs parallel to (001); see Table 2 and Fig. 4.

4. Database survey

Compound (1) differs from the previously reported 2,3-diphenyl-1, 3-thiazolidin-4-one (Yennawar *et al.*, 2014) only in the presence of *p*-chlorine atoms on both phenyl rings, and the compound does not have a twist in the thiazine ring. Compound (2) is related to 2-aryl-1,3-thiazolidin-4-one 1-oxides, *viz.* 3-butyl-2-phenyl-1,3-thiazolidine-1,4-dione (Wang *et al.*, 2010), (1*b*, 2*a*, 5*a*)-3, 5-dimethyl-1-oxo-2-phenyl-4-thiazolidinone (Johnson *et al.*, 1983), 2-(2, 6-dichlorophenyl)-3-(4, 5, 6-trimethylpyrimidin-2-yl)-1, 3-thiazolidin-4-one 1-oxide (Chen *et al.*, 2011) and *trans*-3-benzyl-2-(4-methoxyphenyl)thiazolidin-4-one 1-oxide (Colombo *et al.*, 2008). All five compounds have a *trans* relationship between the O atom attached to the S atom and the 2-aryl ring.

5. Synthesis and crystallization

Compound (1): prepared as previously reported (Tierney *et al.*, 2005). Colourless block-like crystals were obtained by slow evaporation of a solution in ethanol.

Compound (2): 2,3-bis (4-chlorophenyl)-1,3-thiazolidin-4-one (1) (0.326 g, 1 mmol) was added to a 25 ml round-bottom flask. Methanol (4 ml) was added and the mixture was stirred at room temperature before cooling to 273–278 K. A solution of Oxone (0.456 g, 3.0 mmol calculated as KHSO₅, 152.2 g mol⁻¹) in distilled water (4 ml) was prepared. This solution (2.67 ml, 2 equivalents) was slowly added to the reaction mixture with stirring at 273–278 K. The reaction was followed by TLC. An additional aliquot of Oxone solution (0.67 ml) was added to convert the remaining starting material to sulfoxide. The mixture was extracted three times with methylene chloride. The organic layers were combined and washed with water and saturated NaCl, then dried over

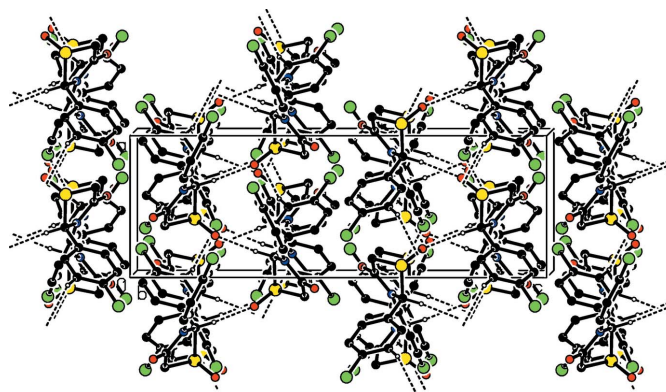


Figure 4

Crystal packing of compound (2) viewed along the *b* axis, showing the hydrogen bonds as dashed lines (see Table 2 for details; H atoms not involved in these interactions have been omitted for clarity).

sodium sulfate. The solution was concentrated under vacuum to give compound (2) as a crude solid. The solid was recrystallized from a mixture of methylene chloride and hexane, and then dried (yield: 0.2413 g; 70.5%; m.p.: 406–409 K). Colourless plate-like crystals were obtained by slow evaporation of a solution in ethanol.

6. Refinement details

Crystal data, data collection and structure refinement details for structures (1) and (2) are summarized in Table 3. H atoms were positioned geometrically with C—H = 0.93–0.97 Å, and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Acknowledgements

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Table 3
Experimental details.

	(1)	(2)
Crystal data		
Chemical formula	C ₁₅ H ₁₁ Cl ₂ NOS	C ₁₅ H ₁₁ Cl ₂ NO ₂ S
<i>M_r</i>	324.21	340.21
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$	Orthorhombic, <i>Pbca</i>
Temperature (K)	298	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.019 (6), 9.562 (8), 9.984 (8)	7.1094 (17), 20.940 (5), 20.940
α , β , γ (°)	88.937 (13), 76.254 (12), 71.586 (13)	90, 90, 90
<i>V</i> (Å ³)	704.3 (10)	3117.4 (11)
<i>Z</i>	2	8
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.60	0.55
Crystal size (mm)	0.22 × 0.20 × 0.16	0.19 × 0.17 × 0.05
Data collection		
Diffractometer	Bruker <i>SMART</i> CCD area detector	Bruker <i>SMART</i> CCD area detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2001)	Multi-scan (<i>SADABS</i> ; Bruker, 2001)
<i>T_{min}</i> , <i>T_{max}</i>	0.879, 0.910	0.902, 0.973
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	6575, 3406, 3070	26788, 3862, 2543
<i>R_{int}</i>	0.016	0.038
(<i>sin</i> θ/ <i>λ</i>) _{max} (Å ⁻¹)	0.666	0.666
Refinement		
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.036, 0.099, 1.05	0.051, 0.138, 1.07
No. of reflections	3406	3862
No. of parameters	181	190
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.24, -0.42	0.33, -0.31

Computer programs: *SMART* and *SAINT* (Bruker, 2001), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

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Crystal structures of 2,3-bis(4-chlorophenyl)-1,3-thiazolidin-4-one and *trans*-2,3-bis(4-chlorophenyl)-1,3-thiazolidin-4-one 1-oxide

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Computing details

For both compounds, data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

(1) 2,3-Bis(4-chlorophenyl)-1,3-thiazolidin-4-one

Crystal data

$C_{15}H_{11}Cl_2NOS$	$Z = 2$
$M_r = 324.21$	$F(000) = 332$
Triclinic, $P1$	$D_x = 1.529 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 8.019 (6) \text{ \AA}$	Cell parameters from 4305 reflections
$b = 9.562 (8) \text{ \AA}$	$\theta = 2.3\text{--}28.2^\circ$
$c = 9.984 (8) \text{ \AA}$	$\mu = 0.60 \text{ mm}^{-1}$
$\alpha = 88.937 (13)^\circ$	$T = 298 \text{ K}$
$\beta = 76.254 (12)^\circ$	Block, colourless
$\gamma = 71.586 (13)^\circ$	$0.22 \times 0.20 \times 0.16 \text{ mm}$
$V = 704.3 (10) \text{ \AA}^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	6575 measured reflections
Radiation source: fine-focus sealed tube	3406 independent reflections
Graphite monochromator	3070 reflections with $I > 2\sigma(I)$
Detector resolution: $8.34 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.016$
phi and ω scans	$\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.1^\circ$
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2001)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.879$, $T_{\text{max}} = 0.910$	$k = -12 \rightarrow 12$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.036$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.099$	H-atom parameters constrained
$S = 1.05$	
3406 reflections	
181 parameters	
0 restraints	

$$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.2029P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.42 \text{ e } \text{\AA}^{-3}$$

Special details

Experimental. The data collection nominally covered a full sphere of reciprocal space by a combination of 4 sets of ω scans each set at different φ and/or 2θ angles and each scan (10 s exposure) covering -0.300° degrees in ω . The crystal to detector distance was 5.82 cm.

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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.48888 (17)	0.30999 (15)	0.71043 (13)	0.0330 (3)
H1	0.4491	0.2300	0.6838	0.040*
C2	0.68202 (17)	0.28648 (14)	0.63246 (13)	0.0310 (3)
C3	0.78163 (19)	0.36815 (16)	0.66932 (14)	0.0356 (3)
H3	0.7306	0.4355	0.7455	0.043*
C4	0.95613 (19)	0.34994 (16)	0.59348 (14)	0.0375 (3)
H4	1.0233	0.4040	0.6184	0.045*
C5	1.02943 (19)	0.24998 (16)	0.47982 (15)	0.0391 (3)
C6	0.9333 (2)	0.16883 (17)	0.44035 (15)	0.0418 (3)
H6	0.9841	0.1029	0.3631	0.050*
C7	0.7584 (2)	0.18715 (16)	0.51810 (14)	0.0377 (3)
H7	0.6920	0.1323	0.4932	0.045*
C8	0.56811 (17)	0.21103 (15)	0.93089 (13)	0.0317 (3)
C9	0.64973 (19)	0.06770 (15)	0.87310 (14)	0.0366 (3)
H9	0.6364	0.0441	0.7872	0.044*
C10	0.7510 (2)	-0.04056 (17)	0.94240 (16)	0.0422 (3)
H10	0.8060	-0.1366	0.9034	0.051*
C11	0.7694 (2)	-0.00403 (18)	1.07003 (17)	0.0434 (3)
C12	0.6911 (2)	0.13821 (19)	1.12805 (16)	0.0457 (3)
H12	0.7053	0.1614	1.2139	0.055*
C13	0.5915 (2)	0.24636 (17)	1.05824 (15)	0.0395 (3)
H13	0.5401	0.3429	1.0964	0.047*
C14	0.29560 (18)	0.41916 (16)	0.93219 (15)	0.0364 (3)
C15	0.19141 (19)	0.51206 (17)	0.83648 (16)	0.0429 (3)
H15A	0.1473	0.6153	0.8689	0.051*
H15B	0.0884	0.4811	0.8329	0.051*
Cl1	1.25015 (6)	0.22688 (6)	0.38612 (5)	0.06617 (16)
Cl2	0.89079 (7)	-0.14012 (6)	1.16121 (6)	0.06647 (16)
N1	0.45860 (15)	0.32152 (13)	0.86178 (11)	0.0322 (2)

O1	0.23825 (15)	0.42928 (15)	1.05662 (11)	0.0510 (3)
S1	0.34401 (5)	0.48672 (5)	0.66875 (4)	0.04576 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0324 (6)	0.0375 (7)	0.0286 (6)	-0.0104 (5)	-0.0075 (5)	0.0019 (5)
C2	0.0327 (6)	0.0329 (6)	0.0252 (5)	-0.0080 (5)	-0.0064 (5)	0.0040 (5)
C3	0.0379 (7)	0.0386 (7)	0.0279 (6)	-0.0109 (5)	-0.0051 (5)	-0.0032 (5)
C4	0.0380 (7)	0.0413 (7)	0.0344 (7)	-0.0147 (6)	-0.0084 (5)	0.0018 (6)
C5	0.0354 (7)	0.0393 (7)	0.0352 (7)	-0.0084 (6)	0.0004 (5)	0.0025 (6)
C6	0.0465 (8)	0.0391 (7)	0.0328 (7)	-0.0108 (6)	0.0004 (6)	-0.0052 (6)
C7	0.0439 (7)	0.0372 (7)	0.0322 (6)	-0.0145 (6)	-0.0072 (6)	-0.0016 (5)
C8	0.0288 (6)	0.0361 (6)	0.0297 (6)	-0.0121 (5)	-0.0044 (5)	0.0056 (5)
C9	0.0371 (7)	0.0375 (7)	0.0333 (6)	-0.0120 (6)	-0.0053 (5)	0.0035 (5)
C10	0.0378 (7)	0.0370 (7)	0.0476 (8)	-0.0105 (6)	-0.0053 (6)	0.0093 (6)
C11	0.0367 (7)	0.0487 (8)	0.0499 (8)	-0.0184 (6)	-0.0153 (6)	0.0224 (7)
C12	0.0485 (8)	0.0583 (9)	0.0392 (7)	-0.0240 (7)	-0.0185 (6)	0.0127 (7)
C13	0.0413 (7)	0.0427 (7)	0.0368 (7)	-0.0155 (6)	-0.0110 (6)	0.0022 (6)
C14	0.0303 (6)	0.0402 (7)	0.0372 (7)	-0.0107 (5)	-0.0056 (5)	-0.0027 (5)
C15	0.0308 (6)	0.0451 (8)	0.0473 (8)	-0.0053 (6)	-0.0085 (6)	0.0014 (6)
Cl1	0.0465 (2)	0.0702 (3)	0.0689 (3)	-0.0232 (2)	0.0179 (2)	-0.0170 (2)
Cl2	0.0627 (3)	0.0667 (3)	0.0827 (3)	-0.0258 (2)	-0.0377 (3)	0.0424 (3)
N1	0.0307 (5)	0.0357 (5)	0.0269 (5)	-0.0076 (4)	-0.0050 (4)	0.0013 (4)
O1	0.0377 (5)	0.0675 (8)	0.0360 (5)	-0.0062 (5)	-0.0002 (4)	-0.0081 (5)
S1	0.0374 (2)	0.0520 (2)	0.0426 (2)	-0.00607 (16)	-0.01206 (16)	0.01364 (17)

Geometric parameters (Å, °)

C1—N1	1.473 (2)	C8—N1	1.4277 (18)
C1—C2	1.506 (2)	C9—C10	1.386 (2)
C1—S1	1.8282 (17)	C9—H9	0.9300
C1—H1	0.9800	C10—C11	1.380 (3)
C2—C7	1.386 (2)	C10—H10	0.9300
C2—C3	1.388 (2)	C11—C12	1.377 (3)
C3—C4	1.382 (2)	C11—Cl2	1.7455 (17)
C3—H3	0.9300	C12—C13	1.382 (2)
C4—C5	1.384 (2)	C12—H12	0.9300
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.373 (2)	C14—O1	1.212 (2)
C5—Cl1	1.7408 (19)	C14—N1	1.3751 (19)
C6—C7	1.390 (2)	C14—C15	1.510 (2)
C6—H6	0.9300	C15—S1	1.7930 (19)
C7—H7	0.9300	C15—H15A	0.9700
C8—C9	1.387 (2)	C15—H15B	0.9700
C8—C13	1.391 (2)		
N1—C1—C2	114.30 (11)	C10—C9—H9	119.8

N1—C1—S1	104.57 (9)	C8—C9—H9	119.8
C2—C1—S1	109.22 (10)	C11—C10—C9	119.14 (15)
N1—C1—H1	109.5	C11—C10—H10	120.4
C2—C1—H1	109.5	C9—C10—H10	120.4
S1—C1—H1	109.5	C12—C11—C10	121.07 (14)
C7—C2—C3	119.49 (13)	C12—C11—C12	119.13 (13)
C7—C2—C1	119.49 (12)	C10—C11—C12	119.80 (13)
C3—C2—C1	120.94 (12)	C11—C12—C13	119.72 (15)
C4—C3—C2	120.40 (13)	C11—C12—H12	120.1
C4—C3—H3	119.8	C13—C12—H12	120.1
C2—C3—H3	119.8	C12—C13—C8	120.07 (15)
C3—C4—C5	119.01 (13)	C12—C13—H13	120.0
C3—C4—H4	120.5	C8—C13—H13	120.0
C5—C4—H4	120.5	O1—C14—N1	124.72 (14)
C6—C5—C4	121.77 (14)	O1—C14—C15	122.94 (13)
C6—C5—C11	119.69 (12)	N1—C14—C15	112.33 (13)
C4—C5—C11	118.54 (12)	C14—C15—S1	107.22 (11)
C5—C6—C7	118.72 (14)	C14—C15—H15A	110.3
C5—C6—H6	120.6	S1—C15—H15A	110.3
C7—C6—H6	120.6	C14—C15—H15B	110.3
C2—C7—C6	120.60 (13)	S1—C15—H15B	110.3
C2—C7—H7	119.7	H15A—C15—H15B	108.5
C6—C7—H7	119.7	C14—N1—C8	121.42 (12)
C9—C8—C13	119.47 (13)	C14—N1—C1	115.85 (11)
C9—C8—N1	120.56 (13)	C8—N1—C1	120.65 (11)
C13—C8—N1	119.96 (13)	C15—S1—C1	91.77 (7)
C10—C9—C8	120.50 (14)		
N1—C1—C2—C7	138.56 (13)	C11—C12—C13—C8	1.0 (2)
S1—C1—C2—C7	-104.71 (14)	C9—C8—C13—C12	-1.8 (2)
N1—C1—C2—C3	-44.66 (17)	N1—C8—C13—C12	177.27 (13)
S1—C1—C2—C3	72.06 (15)	O1—C14—C15—S1	168.76 (13)
C7—C2—C3—C4	-0.5 (2)	N1—C14—C15—S1	-12.48 (15)
C1—C2—C3—C4	-177.31 (13)	O1—C14—N1—C8	6.5 (2)
C2—C3—C4—C5	0.5 (2)	C15—C14—N1—C8	-172.19 (12)
C3—C4—C5—C6	0.1 (2)	O1—C14—N1—C1	170.20 (14)
C3—C4—C5—C11	-179.42 (11)	C15—C14—N1—C1	-8.53 (17)
C4—C5—C6—C7	-0.7 (2)	C9—C8—N1—C14	136.45 (14)
C11—C5—C6—C7	178.88 (12)	C13—C8—N1—C14	-42.65 (18)
C3—C2—C7—C6	0.0 (2)	C9—C8—N1—C1	-26.44 (18)
C1—C2—C7—C6	176.81 (13)	C13—C8—N1—C1	154.46 (13)
C5—C6—C7—C2	0.6 (2)	C2—C1—N1—C14	144.10 (13)
C13—C8—C9—C10	1.3 (2)	S1—C1—N1—C14	24.72 (14)
N1—C8—C9—C10	-177.86 (12)	C2—C1—N1—C8	-52.09 (16)
C8—C9—C10—C11	0.2 (2)	S1—C1—N1—C8	-171.47 (9)
C9—C10—C11—C12	-1.0 (2)	C14—C15—S1—C1	22.62 (11)
C9—C10—C11—C12	178.08 (11)	N1—C1—S1—C15	-26.42 (10)
C10—C11—C12—C13	0.4 (2)	C2—C1—S1—C15	-149.16 (10)

C12—C11—C12—C13 -178.68 (12)

Hydrogen-bond geometry (Å, °)

<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>
C3—H3···O1 ⁱ	0.93	2.48	3.326 (3)	151
C15—H15B···O1 ⁱⁱ	0.97	2.46	3.221 (3)	135

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

(2) 2,3-Bis(4-chlorophenyl)-1,3-thiazolidin-4-one 1-oxide

Crystal data

C₁₅H₁₁Cl₂NO₂S*M_r* = 340.21Orthorhombic, *Pbca*

Hall symbol: -P 2ab 2ac

a = 7.1094 (17) Å*b* = 20.940 (5) Å*c* = 20.940 Å*V* = 3117.4 (11) Å³*Z* = 8*F*(000) = 1392*D_x* = 1.450 Mg m⁻³Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 5771 reflections

θ = 2.2–28.2°

μ = 0.55 mm⁻¹*T* = 298 K

Plate, colourless

0.19 × 0.17 × 0.05 mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.34 pixels mm⁻¹

phi and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

T_{min} = 0.902, *T_{max}* = 0.973

26788 measured reflections

3862 independent reflections

2543 reflections with *I* > 2σ(*I*)*R_{int}* = 0.038θ_{max} = 28.3°, θ_{min} = 2.0°*h* = -9→9*k* = -27→27*l* = -27→27

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.051*wR*(*F*²) = 0.138*S* = 1.07

3862 reflections

190 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0581*P*)² + 1.0427*P*]where *P* = (*F_o*² + 2*F_c*²)/3(Δ/σ)_{max} = 0.003Δρ_{max} = 0.33 e Å⁻³Δρ_{min} = -0.31 e Å⁻³

Special details

Experimental. The data collection nominally covered a full sphere of reciprocal space by a combination of 4 sets of ω scans each set at different φ and/or 2θ angles and each scan (10 s exposure) covering -0.300° degrees in ω. The crystal to detector distance was 5.82 cm.

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1427 (3)	0.56601 (10)	0.84451 (10)	0.0447 (5)
H1	0.1817	0.5615	0.7999	0.054*
C2	0.2832 (3)	0.60780 (10)	0.87867 (10)	0.0446 (5)
C3	0.3302 (3)	0.59815 (12)	0.94158 (11)	0.0508 (6)
H3	0.2783	0.5640	0.9638	0.061*
C4	0.4542 (4)	0.63897 (14)	0.97198 (12)	0.0627 (7)
H4	0.4843	0.6326	1.0147	0.075*
C5	0.5322 (4)	0.68858 (14)	0.93908 (15)	0.0710 (8)
C6	0.4887 (4)	0.69891 (13)	0.87616 (16)	0.0745 (8)
H6	0.5433	0.7327	0.8541	0.089*
C7	0.3629 (4)	0.65874 (12)	0.84583 (12)	0.0594 (6)
H7	0.3316	0.6658	0.8033	0.071*
C8	0.2445 (3)	0.45232 (11)	0.85694 (9)	0.0455 (5)
C9	0.4140 (3)	0.46695 (12)	0.82790 (12)	0.0563 (6)
H9	0.4447	0.5094	0.8199	0.068*
C10	0.5379 (4)	0.41909 (14)	0.81076 (13)	0.0684 (7)
H10	0.6511	0.4293	0.7910	0.082*
C11	0.4939 (5)	0.35700 (14)	0.82285 (13)	0.0716 (8)
C12	0.3310 (5)	0.34158 (14)	0.85240 (14)	0.0808 (9)
H12	0.3038	0.2990	0.8612	0.097*
C13	0.2046 (4)	0.38873 (13)	0.86954 (13)	0.0677 (7)
H13	0.0925	0.3778	0.8896	0.081*
C14	-0.0326 (3)	0.49782 (12)	0.91318 (10)	0.0508 (6)
C15	-0.1372 (3)	0.56016 (13)	0.91777 (10)	0.0593 (7)
H15A	-0.0952	0.5839	0.9549	0.071*
H15B	-0.2711	0.5523	0.9219	0.071*
C11	0.68956 (15)	0.73930 (6)	0.97629 (6)	0.1258 (4)
C12	0.64436 (17)	0.29545 (5)	0.79888 (5)	0.1196 (4)
N1	0.1163 (2)	0.50246 (9)	0.87174 (8)	0.0434 (4)
O1	-0.2071 (3)	0.57407 (11)	0.79699 (8)	0.0779 (6)
O2	-0.0736 (3)	0.45046 (9)	0.94350 (8)	0.0715 (5)
S1	-0.09086 (9)	0.60445 (3)	0.84674 (3)	0.0560 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0457 (12)	0.0505 (13)	0.0378 (10)	-0.0016 (10)	0.0072 (9)	0.0010 (9)
C2	0.0398 (11)	0.0484 (12)	0.0456 (11)	0.0011 (10)	0.0086 (9)	-0.0022 (9)
C3	0.0454 (13)	0.0594 (15)	0.0475 (12)	-0.0024 (11)	0.0063 (10)	-0.0046 (10)
C4	0.0507 (14)	0.0793 (19)	0.0580 (14)	-0.0008 (13)	0.0003 (12)	-0.0162 (13)

C5	0.0479 (15)	0.077 (2)	0.089 (2)	-0.0116 (14)	0.0071 (14)	-0.0282 (16)
C6	0.0671 (18)	0.0580 (17)	0.099 (2)	-0.0187 (14)	0.0212 (16)	-0.0015 (15)
C7	0.0607 (15)	0.0567 (15)	0.0607 (14)	-0.0082 (12)	0.0083 (12)	0.0027 (12)
C8	0.0469 (12)	0.0508 (13)	0.0387 (11)	-0.0038 (10)	0.0008 (9)	-0.0001 (9)
C9	0.0518 (14)	0.0577 (15)	0.0593 (14)	-0.0008 (11)	0.0120 (11)	0.0030 (11)
C10	0.0566 (16)	0.079 (2)	0.0700 (17)	0.0129 (14)	0.0092 (13)	0.0026 (14)
C11	0.080 (2)	0.0716 (19)	0.0636 (16)	0.0273 (16)	0.0024 (15)	0.0056 (14)
C12	0.110 (3)	0.0470 (15)	0.085 (2)	0.0075 (16)	0.0129 (19)	0.0078 (14)
C13	0.0747 (19)	0.0548 (16)	0.0735 (17)	-0.0092 (14)	0.0156 (14)	0.0053 (13)
C14	0.0445 (12)	0.0728 (16)	0.0352 (10)	-0.0074 (11)	0.0039 (9)	0.0008 (11)
C15	0.0434 (13)	0.0903 (19)	0.0442 (12)	0.0087 (13)	0.0042 (10)	-0.0051 (12)
Cl1	0.0974 (7)	0.1391 (9)	0.1409 (9)	-0.0599 (7)	-0.0018 (6)	-0.0500 (7)
Cl2	0.1392 (9)	0.1012 (7)	0.1184 (8)	0.0708 (7)	0.0144 (7)	0.0076 (6)
N1	0.0405 (9)	0.0512 (10)	0.0387 (8)	-0.0037 (8)	0.0068 (7)	0.0014 (8)
O1	0.0607 (12)	0.1197 (16)	0.0534 (10)	-0.0018 (11)	-0.0179 (9)	-0.0016 (10)
O2	0.0764 (13)	0.0803 (13)	0.0579 (10)	-0.0134 (10)	0.0236 (9)	0.0120 (9)
S1	0.0494 (4)	0.0702 (4)	0.0484 (3)	0.0080 (3)	-0.0057 (3)	0.0022 (3)

Geometric parameters (Å, °)

C1—N1	1.460 (3)	C8—N1	1.425 (3)
C1—C2	1.508 (3)	C9—C10	1.382 (4)
C1—S1	1.846 (2)	C9—H9	0.9300
C1—H1	0.9800	C10—C11	1.361 (4)
C2—C3	1.374 (3)	C10—H10	0.9300
C2—C7	1.390 (3)	C11—C12	1.352 (4)
C3—C4	1.383 (3)	C11—Cl2	1.749 (3)
C3—H3	0.9300	C12—C13	1.382 (4)
C4—C5	1.365 (4)	C12—H12	0.9300
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.371 (4)	C14—O2	1.213 (3)
C5—Cl1	1.728 (3)	C14—N1	1.372 (3)
C6—C7	1.382 (4)	C14—C15	1.506 (4)
C6—H6	0.9300	C15—S1	1.784 (2)
C7—H7	0.9300	C15—H15A	0.9700
C8—C9	1.384 (3)	C15—H15B	0.9700
C8—C13	1.387 (3)	O1—S1	1.4742 (19)
N1—C1—C2	115.38 (18)	C8—C9—H9	119.7
N1—C1—S1	105.78 (14)	C11—C10—C9	119.9 (3)
C2—C1—S1	109.30 (15)	C11—C10—H10	120.1
N1—C1—H1	108.7	C9—C10—H10	120.1
C2—C1—H1	108.7	C12—C11—C10	120.7 (3)
S1—C1—H1	108.7	C12—C11—Cl2	118.6 (2)
C3—C2—C7	119.2 (2)	C10—C11—Cl2	120.7 (2)
C3—C2—C1	122.0 (2)	C11—C12—C13	120.3 (3)
C7—C2—C1	118.7 (2)	C11—C12—H12	119.8
C2—C3—C4	120.4 (2)	C13—C12—H12	119.8

C2—C3—H3	119.8	C12—C13—C8	120.2 (3)
C4—C3—H3	119.8	C12—C13—H13	119.9
C5—C4—C3	119.8 (2)	C8—C13—H13	119.9
C5—C4—H4	120.1	O2—C14—N1	125.1 (2)
C3—C4—H4	120.1	O2—C14—C15	123.8 (2)
C4—C5—C6	120.9 (3)	N1—C14—C15	111.1 (2)
C4—C5—C11	120.2 (2)	C14—C15—S1	107.83 (15)
C6—C5—C11	118.9 (2)	C14—C15—H15A	110.1
C5—C6—C7	119.5 (3)	S1—C15—H15A	110.1
C5—C6—H6	120.3	C14—C15—H15B	110.1
C7—C6—H6	120.3	S1—C15—H15B	110.1
C6—C7—C2	120.2 (3)	H15A—C15—H15B	108.5
C6—C7—H7	119.9	C14—N1—C8	125.37 (19)
C2—C7—H7	119.9	C14—N1—C1	114.26 (19)
C9—C8—C13	118.3 (2)	C8—N1—C1	120.31 (17)
C9—C8—N1	119.3 (2)	O1—S1—C15	105.16 (13)
C13—C8—N1	122.4 (2)	O1—S1—C1	107.35 (11)
C10—C9—C8	120.6 (2)	C15—S1—C1	87.74 (10)
C10—C9—H9	119.7		
N1—C1—C2—C3	-23.1 (3)	C9—C8—C13—C12	-1.0 (4)
S1—C1—C2—C3	96.0 (2)	N1—C8—C13—C12	177.9 (2)
N1—C1—C2—C7	158.9 (2)	O2—C14—C15—S1	-158.4 (2)
S1—C1—C2—C7	-82.1 (2)	N1—C14—C15—S1	23.1 (2)
C7—C2—C3—C4	0.5 (3)	O2—C14—N1—C8	1.0 (4)
C1—C2—C3—C4	-177.5 (2)	C15—C14—N1—C8	179.50 (19)
C2—C3—C4—C5	-0.9 (4)	O2—C14—N1—C1	-176.0 (2)
C3—C4—C5—C6	0.4 (4)	C15—C14—N1—C1	2.4 (3)
C3—C4—C5—C11	-179.2 (2)	C9—C8—N1—C14	-163.5 (2)
C4—C5—C6—C7	0.5 (4)	C13—C8—N1—C14	17.7 (3)
C11—C5—C6—C7	-179.9 (2)	C9—C8—N1—C1	13.4 (3)
C5—C6—C7—C2	-0.8 (4)	C13—C8—N1—C1	-165.4 (2)
C3—C2—C7—C6	0.3 (4)	C2—C1—N1—C14	95.3 (2)
C1—C2—C7—C6	178.4 (2)	S1—C1—N1—C14	-25.7 (2)
C13—C8—C9—C10	1.4 (4)	C2—C1—N1—C8	-82.0 (2)
N1—C8—C9—C10	-177.5 (2)	S1—C1—N1—C8	157.09 (15)
C8—C9—C10—C11	-0.4 (4)	C14—C15—S1—O1	76.00 (19)
C9—C10—C11—C12	-1.0 (5)	C14—C15—S1—C1	-31.37 (17)
C9—C10—C11—C12	177.6 (2)	N1—C1—S1—O1	-72.87 (16)
C10—C11—C12—C13	1.4 (5)	C2—C1—S1—O1	162.31 (15)
C12—C11—C12—C13	-177.2 (2)	N1—C1—S1—C15	32.31 (16)
C11—C12—C13—C8	-0.4 (5)	C2—C1—S1—C15	-92.52 (16)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1 \cdots O1 ⁱ	0.98	2.19	3.154 (3)	167

C6—H6···Cl2 ⁱⁱ	0.93	2.83	3.676 (3)	152
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Symmetry codes: (i) $x+1/2, y, -z+3/2$; (ii) $-x+3/2, y+1/2, z$.