

Crystal structures of two thiazolidinone derivatives bearing a trichloromethyl substituent at the 2-position

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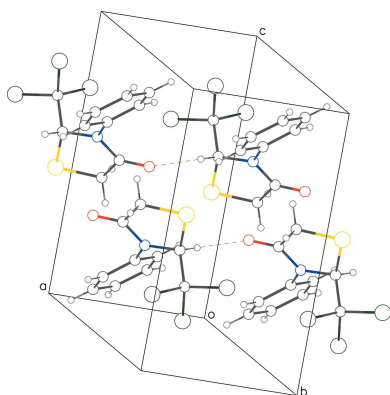
Keywords: crystal structure; 1,3-thiazolidin-4-ones; trichloromethyl.**CCDC references:** 1868299; 1868298**Supporting information:** this article has supporting information at journals.iucr.org/e

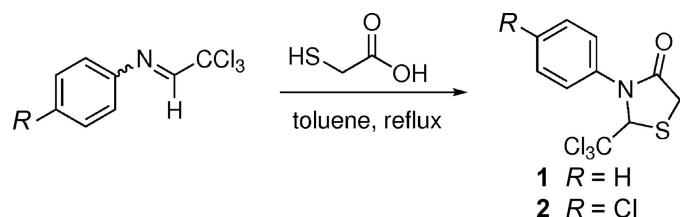
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The title compounds 2-trichloromethyl-3-phenyl-1,3-thiazolidin-4-one (C₁₀H₈Cl₃NOS), **1** and 3-(4-chlorophenyl)-2-trichloromethyl-1,3-thiazolidin-4-one (C₁₀H₇Cl₄NOS) **2**, are structurally related with one atom substitution difference in the *para* position of the benzene ring. In both structures, the thiazolidinone ring adopts an envelope conformation with the S atom as the flap. The dihedral angles between the rings [48.72 (11) in **1** and 48.42 (9)° in **2**] are very similar and the molecules are almost superimposable. In both crystal structures, C—H···O ‘head-to-tail’ interactions between the chiral carbon atoms and the thiazolidinone oxygen atoms result in infinite monochiral chains along the direction of the shortest unit-cell parameter, namely *a* in **1** and *b* in **2**. C—H···π interactions between the thiazolidinone carbon atom at the 4-position and the phenyl ring of the neighboring enantiomer also help to stabilize the packing in each case, although the crystals are not isostructural.

1. Chemical context

The title compounds **1** and **2** are unique structures containing a trichloromethyl substituent at the 2-position of the thiazolidinone ring. Their synthesis was first reported as two of only three known 2-alkyl thiazolidin-4-one compounds (Tierney, 1989; Issac *et al.*, 1996). Substituted thiazolidin-4-one compounds are synthesized by reacting an *in situ* generated imine (Schiff base) with thioglycolic acid and with a mechanism to remove the water byproduct (Surrey, 1947; Erlenmeyer & Oberlin, 1947). Therefore, when chloral is reacted with arylamines, the corresponding imine is formed, which, upon reacting with thioglycolic acid, produces the desired 2-trichloromethyl-3-aryl-thiazolidin-4-one (Issac *et al.*, 1996). It is interesting to note, however, that the reaction of chloral with some alkyl amines results in an *N*-alkylformamide product when the initially formed aminol loses chloroform instead of water (Mascavage *et al.*, 2010). The loss of chloroform appears to be more facile in electron-rich *N*-alkylamines that can stabilize the transition state and lower the energy of activation of the elimination step better than the less electron-rich *N*-arylamines. On the other hand, imine formation is favored with arylamines because of the lower p*K_a* of the proton on the nitrogen in the aminol, which facilitates the removal of water to give an imine. As part of our ongoing studies in this area, we now describe the crystal structures of **1** and **2**.





2. Structural commentary

Compounds **1** and **2** are structurally related with one atom substitution difference in the *para* position of the benzene ring; a hydrogen atom is substituted for a chlorine atom (Figs. 1 and 2). Both contain a stereogenic centre at C1, which is arbitrarily assigned as having an *R* configuration in the asymmetric units of the centrosymmetric unit cells. In both structures, the thiazolidinone ring adopts an envelope conformation with the S atom as the flap. The sulfur atom is displaced from the thiazolidinone ring plane by 0.35 (2) Å in both structures. The dihedral angles between the thiazolidinone and phenyl rings are 48.72 (11)° in **1** and 48.42 (9)° in **2**. The C1–N1 and C1–S1 bond lengths are 1.445 (2) Å and 1.816 (2) Å, respectively, for structure **1** and 1.4471 (18) Å and 1.8181 (16) Å, respectively, for structure **2**. The N–C–S bond angle is found to be 106.52 (12)° in structure **1** and 106.23 (10)° in structure **2**. Overall, the molecular structures of both are almost exactly superimposable (Fig. 3). Bond length and angle values in the thiazolidinone ring in both structures appear to be typical and match currently available data (Yennawar *et al.*, 2015).

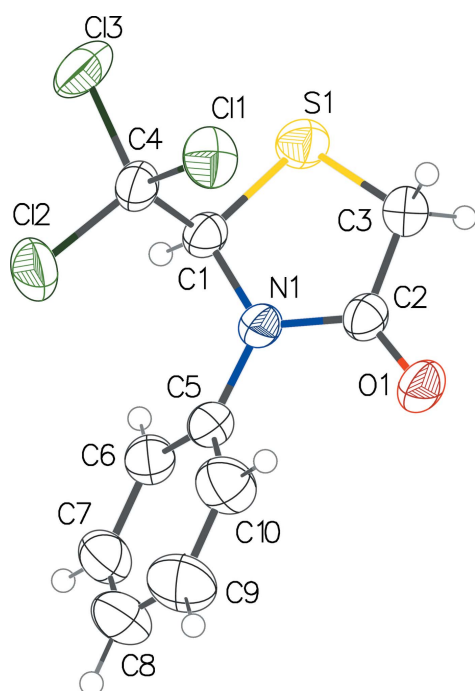


Figure 1
The molecular structure of compound **1** with displacement ellipsoids drawn at the 50% probability level.

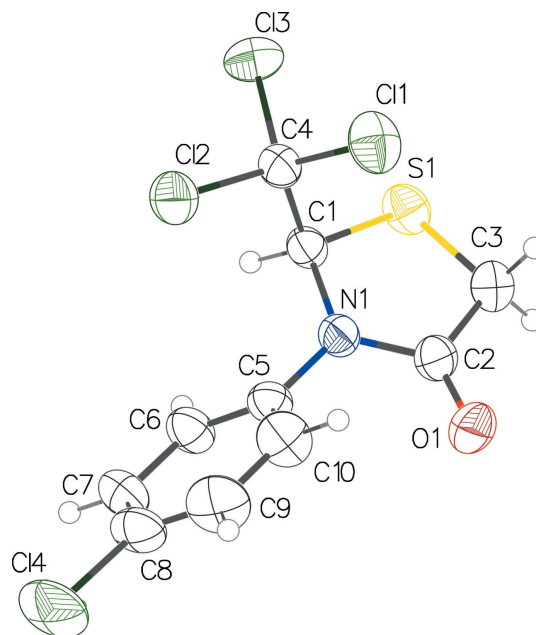


Figure 2
The molecular structure of compound **2** with displacement ellipsoids drawn at the 50% probability level.

3. Supramolecular features

Both extended structures exhibit C–H···O ‘head-to-tail’ intermolecular interactions between the chiral carbon atom C1 and the thiazolidinone oxygen atom (Tables 1 and 2; Figs. 4 and 5) that result in infinite monochiral chains propagating

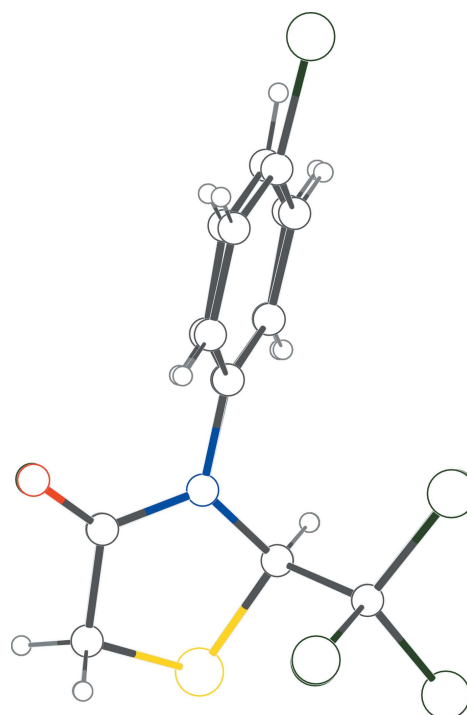


Figure 3
Superposition image for structures **1** and **2** showing similarity of conformation.

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

$Cg2$ is the centroid of the C5–C10 ring

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C1-H1\cdots O1^i$	0.98	2.30	3.251 (2)	164
$C3-H3A\cdots Cg2^{ii}$	0.97	2.79	3.748 (2)	170

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

along the shortest unit-cell dimension, namely a in **1** and b in **2**; in both cases adjacent molecules are related only by translational symmetry. The short $H\cdots O$ distances of 2.30 Å suggest that these interactions are relatively strong. Weak $C-H\cdots\pi$ interactions between the tetrahedral, non-chiral carbon atom (C3) of the thiazolidinone ring and the phenyl ring of the symmetry-related enantiomer are also observed in both structures (Tables 1 and 2). Despite the similar molecular conformations and intermolecular interactions, the crystals are not isostructural (**1** is triclinic and **2** is monoclinic).

4. Database survey

To date, there have been no reported X-ray structures of substituted 2-trichloromethyl-3-phenyl-1,3-thiazolidin-4-ones or the unsubstituted parent compound. However, there are a number of studies for structures containing aromatic moieties at the 2- and 3-positions of the thiazolidin-4-one ring (Kumar *et al.*, 2016; Yennawar *et al.*, 2014). In addition, there is a structural and conformational study of 3-cyclohexyl-2-phenyl-1,3-thiazolidin-4-one (Cannon *et al.*, 2013).

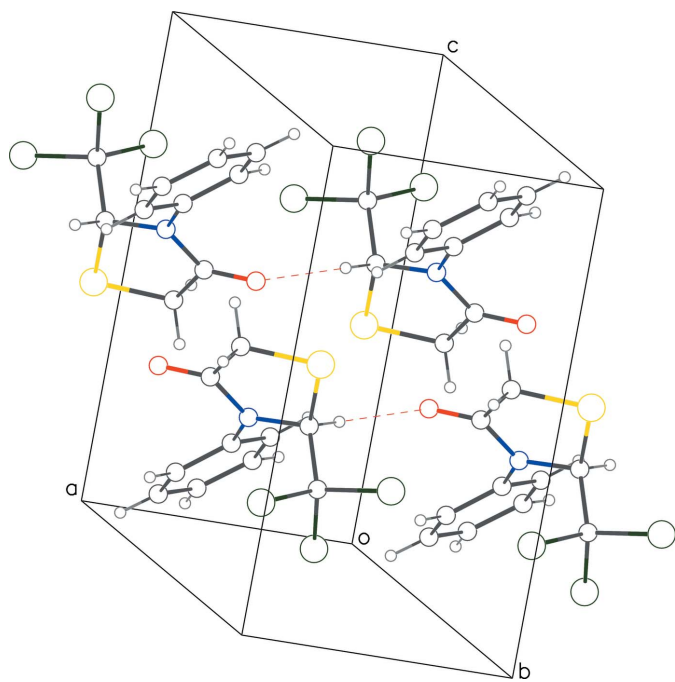


Figure 4
Crystal packing diagram for **1** with red dotted lines for $C-H\cdots O$ contacts.

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

$Cg2$ is the centroid of the C5–C10 ring

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C1-H1\cdots O1^i$	0.98	2.30	3.2643 (19)	168
$C3-H3A\cdots Cg2^{ii}$	0.97	2.85	3.797 (2)	166

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

5. Synthesis and crystallization

The two compounds were synthesized using previously reported procedures (Tierney, 1989; Issac *et al.*, 1996).

2-Trichloromethyl-3-phenyl-1,3-thiazolidin-4-one (**1**): Yield 43%; m.p. 447–448 K; IR: 1687 cm^{-1} ; $^1\text{H NMR}$: δ 7.1–7.5 (m , 5H, aromatics), 5.72 (s , $J = 1.6\text{ Hz}$, 1H), 3.77–3.96 (dd , $J = 1.6, 14.1\text{ Hz}$, 2H); $^{13}\text{C NMR}$: δ 171.65 (C=O), 138.45 (N–Ar),

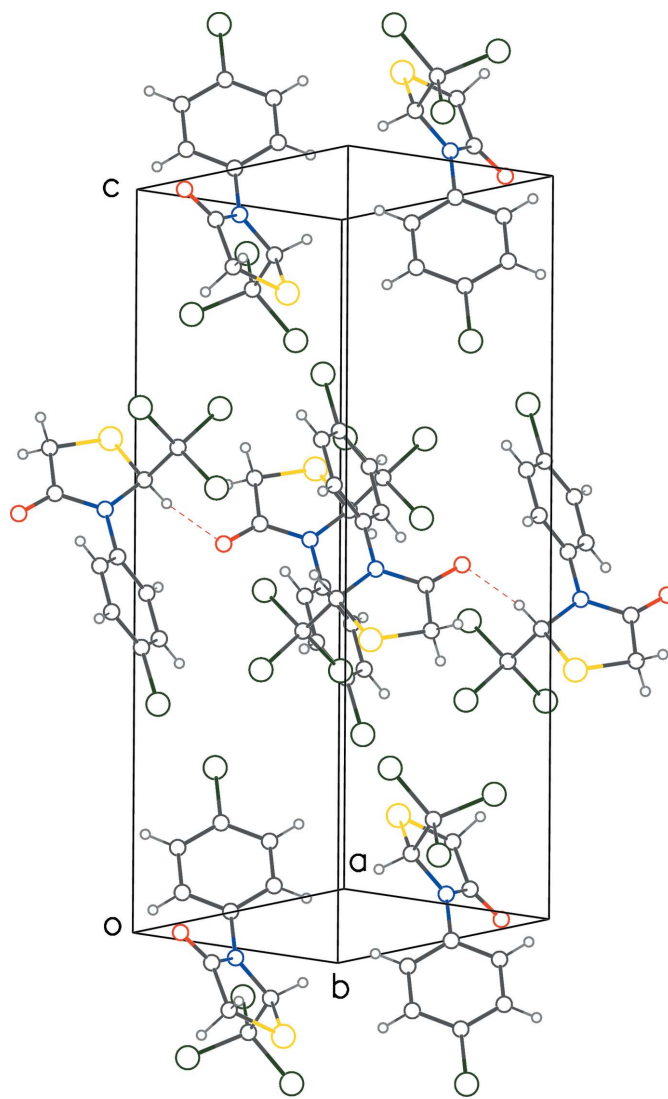


Figure 5
Crystal packing diagram for **2** with red dotted lines for $C-H\cdots O$ contacts.

Table 3
Experimental details.

	1	2
Crystal data		
Chemical formula	C ₁₀ H ₈ Cl ₃ NOS	C ₁₀ H ₇ Cl ₄ NOS
<i>M_r</i>	296.58	331.03
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$	Monoclinic, <i>P</i> ₂ / <i>n</i>
Temperature (K)	298	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.1968 (13), 9.578 (2), 10.854 (2)	10.907 (2), 6.1686 (12), 19.885 (4)
α , β , γ (°)	103.135 (4), 91.319 (3), 99.239 (3)	90, 96.02 (3), 90
<i>V</i> (Å ³)	618.0 (2)	1330.5 (5)
<i>Z</i>	2	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.89	1.03
Crystal size (mm)	0.3 × 0.05 × 0.03	0.25 × 0.13 × 0.1
Data collection		
Diffractometer	Bruker SMART CCD area detector	Bruker SMART CCD area detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2001)	Multi-scan (<i>SADABS</i> ; BRUKER, 2001)
<i>T</i> _{min} – <i>T</i> _{max}	0.769, 0.9	0.868, 0.9
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	5921, 2977, 2634	12273, 3302, 2883
<i>R</i> _{int}	0.016	0.018
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.667	0.668
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> [<i>F</i> ²], <i>S</i>	0.038, 0.118, 1.01	0.036, 0.107, 1.00
No. of reflections	2977	3302
No. of parameters	145	154
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.53, -0.47	0.47, -0.25

Computer programs: *SMART* and *SAINT* (Bruker, 2001), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXS* and *SHELXL* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

129.17, 127.98, 126.98, 103.18 (CC13), 77.69 (C2), 33.08 (C5). Analysis calculated for C₁₀H₈NOSC1₃: C, 40.40; H, 2.72; N, 4.72; Cl, 35.86. Found: C, 40.60, H, 2.74; N, 4.60; Cl, 35.44.

2-Trichloromethyl-3-(4-chlorophenyl)-1,3-thiazolidin-4-one (**2**): Yield 20%; mp 456–458 K; IR: 1685 cm⁻¹; ¹H NMR: δ 7.11–7.50 (*m*, 4H, aromatics), 6.04 (*s*, *J* = 1.2 Hz, 1H), 3.80–3.92 (*dd*, *J* = 1.2, 15.9 Hz, 2H); ¹³C NMR: δ 171.61 (C=O), 136.96 (N–Ar), 133.78 (C–Cl), 129.46, 127.92, 103.06 (CC13), 77.51 (C2), 32.65 (C5). Analysis calculated for C₁₀H₇NOSC1₄: C, 36.47; H, 2.13; N, 4.25. Found: C, 36.65; H, 2.12; N, 4.04.

Compound **1** was crystallized by vapor diffusion where the sample was dissolved in acetone and placed in a chamber containing hexanes. Compound **2** was crystallized by the same method using methylene chloride as the solvent and a chamber containing hexanes.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The hydrogen atoms were placed in calculated positions with C–H = 0.93–0.98 Å and refined using a riding model with fixed isotropic displacement parameters: *U*_{iso}(H) = 1.5*U*_{eq}(C) for the methyl group and *U*_{iso}(H) = 1.2*U*_{eq}(C) for the remaining H atoms.

Funding information

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supporting information

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Crystal structures of two thiazolidinone derivatives bearing a trichloromethyl substituent at the 2-position

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Computing details

For both structures, data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE* (Bruker, 2001). Program(s) used to solve structure: *olex2.solve* (Bourhis *et al.*, 2015) for (1); *SHELXS* (Sheldrick, 2008) for (2). For both structures, program(s) used to refine structure: *SHELXL* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

2-Trichloromethyl-3-phenyl-1,3-thiazolidin-4-one (1)

Crystal data

$C_{10}H_8Cl_3NOS$

$M_r = 296.58$

Triclinic, $P\bar{1}$

$a = 6.1968$ (13) Å

$b = 9.578$ (2) Å

$c = 10.854$ (2) Å

$\alpha = 103.135$ (4)°

$\beta = 91.319$ (3)°

$\gamma = 99.239$ (3)°

$V = 618.0$ (2) Å³

$Z = 2$

$F(000) = 300$

$D_x = 1.594$ Mg m⁻³

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

Cell parameters from 3362 reflections

$\theta = 2.2$ – 28.3 °

$\mu = 0.89$ mm⁻¹

$T = 298$ K

Needle, colorless

$0.3 \times 0.05 \times 0.03$ mm

Data collection

Bruker SMART CCD area detector
diffractometer

Radiation source: fine-focus sealed tube

Parallel, graphite monochromator

phi and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

$T_{\min} = 0.769$, $T_{\max} = 0.9$

5921 measured reflections

2977 independent reflections

2634 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$

$\theta_{\max} = 28.3$ °, $\theta_{\min} = 1.9$ °

$h = -8 \rightarrow 8$

$k = -12 \rightarrow 12$

$l = -14 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.118$

$S = 1.01$

2977 reflections

145 parameters

0 restraints

Primary atom site location: iterative

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.2774P]$

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

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

$$\Delta\rho_{\max} = 0.53 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.47 \text{ e } \text{\AA}^{-3}$$

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 (20 s exposure) covering -0.300° degrees in ω . The crystal to detector distance was 5.82 cm.

(SADABS; Bruker, 2001) was used for absorption correction. R(int) was 0.0816 before and 0.0197 after correction. The Ratio of minimum to maximum transmission is 0.7686. The $\lambda/2$ correction factor is 0.0015.

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.

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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.06476 (9)	0.12635 (6)	0.75819 (5)	0.05390 (17)
Cl3	0.50432 (10)	0.09734 (7)	0.68826 (7)	0.0654 (2)
Cl2	0.42957 (13)	0.34274 (7)	0.87998 (6)	0.0692 (2)
S1	0.22368 (9)	0.18205 (6)	0.47724 (5)	0.04902 (16)
O1	-0.1907 (2)	0.43408 (17)	0.58501 (16)	0.0491 (4)
N1	0.1467 (2)	0.40320 (17)	0.65276 (15)	0.0367 (3)
C3	-0.0461 (3)	0.2263 (2)	0.4763 (2)	0.0469 (5)
H3A	-0.0878	0.2397	0.3937	0.056*
H3B	-0.1511	0.1483	0.4950	0.056*
C2	-0.0430 (3)	0.3648 (2)	0.57597 (18)	0.0377 (4)
C1	0.3059 (3)	0.3068 (2)	0.62984 (18)	0.0369 (4)
H1	0.4498	0.3643	0.6244	0.044*
C5	0.1956 (3)	0.5430 (2)	0.73953 (18)	0.0399 (4)
C10	0.0444 (4)	0.5876 (3)	0.8246 (2)	0.0599 (6)
H10	-0.0856	0.5257	0.8292	0.072*
C9	0.0893 (6)	0.7262 (4)	0.9031 (3)	0.0798 (9)
H9	-0.0133	0.7584	0.9592	0.096*
C8	0.2836 (6)	0.8162 (3)	0.8989 (3)	0.0743 (8)
H8	0.3127	0.9086	0.9526	0.089*
C7	0.4339 (5)	0.7705 (3)	0.8162 (3)	0.0609 (6)
H7	0.5660	0.8316	0.8145	0.073*
C6	0.3914 (4)	0.6338 (2)	0.7347 (2)	0.0467 (4)
H6	0.4932	0.6033	0.6774	0.056*
C4	0.3226 (3)	0.2231 (2)	0.7350 (2)	0.0420 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0461 (3)	0.0603 (3)	0.0560 (3)	-0.0005 (2)	0.0048 (2)	0.0215 (2)

C13	0.0508 (3)	0.0625 (4)	0.0953 (5)	0.0280 (3)	0.0029 (3)	0.0312 (3)
C12	0.0806 (4)	0.0620 (4)	0.0589 (4)	0.0015 (3)	-0.0314 (3)	0.0126 (3)
S1	0.0514 (3)	0.0515 (3)	0.0447 (3)	0.0196 (2)	0.0057 (2)	0.0045 (2)
O1	0.0336 (7)	0.0512 (8)	0.0644 (9)	0.0133 (6)	-0.0008 (6)	0.0138 (7)
N1	0.0307 (7)	0.0353 (8)	0.0443 (8)	0.0086 (6)	0.0001 (6)	0.0076 (6)
C3	0.0442 (10)	0.0450 (11)	0.0498 (11)	0.0091 (8)	-0.0080 (8)	0.0072 (9)
C2	0.0317 (8)	0.0383 (9)	0.0448 (10)	0.0058 (7)	0.0017 (7)	0.0133 (7)
C1	0.0302 (8)	0.0369 (9)	0.0450 (9)	0.0079 (7)	0.0021 (7)	0.0108 (7)
C5	0.0437 (9)	0.0370 (9)	0.0399 (9)	0.0102 (8)	-0.0011 (7)	0.0091 (7)
C10	0.0561 (13)	0.0633 (14)	0.0547 (13)	0.0111 (11)	0.0109 (10)	0.0010 (11)
C9	0.090 (2)	0.080 (2)	0.0598 (16)	0.0294 (17)	0.0101 (15)	-0.0135 (14)
C8	0.098 (2)	0.0517 (14)	0.0621 (16)	0.0133 (15)	-0.0183 (15)	-0.0089 (12)
C7	0.0715 (15)	0.0427 (12)	0.0623 (14)	-0.0014 (11)	-0.0179 (12)	0.0095 (10)
C6	0.0486 (11)	0.0407 (10)	0.0512 (11)	0.0051 (8)	-0.0028 (9)	0.0136 (9)
C4	0.0339 (8)	0.0432 (10)	0.0502 (11)	0.0063 (7)	-0.0044 (7)	0.0143 (8)

Geometric parameters (Å, °)

C11—C4	1.767 (2)	C1—C4	1.547 (3)
C13—C4	1.778 (2)	C5—C10	1.379 (3)
C12—C4	1.766 (2)	C5—C6	1.384 (3)
S1—C3	1.790 (2)	C10—H10	0.9300
S1—C1	1.816 (2)	C10—C9	1.386 (4)
O1—C2	1.208 (2)	C9—H9	0.9300
N1—C2	1.374 (2)	C9—C8	1.372 (5)
N1—C1	1.445 (2)	C8—H8	0.9300
N1—C5	1.434 (2)	C8—C7	1.362 (4)
C3—H3A	0.9700	C7—H7	0.9300
C3—H3B	0.9700	C7—C6	1.386 (3)
C3—C2	1.506 (3)	C6—H6	0.9300
C1—H1	0.9800		
C3—S1—C1	92.98 (9)	C5—C10—H10	120.5
C2—N1—C1	117.34 (16)	C5—C10—C9	119.0 (3)
C2—N1—C5	120.50 (15)	C9—C10—H10	120.5
C5—N1—C1	121.60 (15)	C10—C9—H9	119.7
S1—C3—H3A	110.2	C8—C9—C10	120.6 (3)
S1—C3—H3B	110.2	C8—C9—H9	119.7
H3A—C3—H3B	108.5	C9—C8—H8	119.9
C2—C3—S1	107.58 (14)	C7—C8—C9	120.1 (2)
C2—C3—H3A	110.2	C7—C8—H8	119.9
C2—C3—H3B	110.2	C8—C7—H7	119.8
O1—C2—N1	124.51 (18)	C8—C7—C6	120.5 (3)
O1—C2—C3	123.05 (18)	C6—C7—H7	119.8
N1—C2—C3	112.43 (16)	C5—C6—C7	119.2 (2)
S1—C1—H1	108.8	C5—C6—H6	120.4
N1—C1—S1	106.52 (12)	C7—C6—H6	120.4
N1—C1—H1	108.8	C11—C4—C13	108.87 (11)

N1—C1—C4	112.89 (16)	C12—C4—C11	108.94 (12)
C4—C1—S1	110.96 (14)	C12—C4—C13	108.22 (10)
C4—C1—H1	108.8	C1—C4—C11	111.51 (13)
C10—C5—N1	119.72 (19)	C1—C4—C13	108.20 (14)
C10—C5—C6	120.6 (2)	C1—C4—C12	111.02 (14)
C6—C5—N1	119.70 (18)		
S1—C3—C2—O1	168.28 (16)	C1—S1—C3—C2	15.36 (16)
S1—C3—C2—N1	-10.8 (2)	C1—N1—C2—O1	179.29 (18)
S1—C1—C4—C11	-64.40 (16)	C1—N1—C2—C3	-1.7 (2)
S1—C1—C4—C13	55.31 (15)	C1—N1—C5—C10	-135.2 (2)
S1—C1—C4—C12	173.93 (9)	C1—N1—C5—C6	46.7 (3)
N1—C1—C4—C11	55.1 (2)	C5—N1—C2—O1	-9.2 (3)
N1—C1—C4—C13	174.80 (12)	C5—N1—C2—C3	169.87 (17)
N1—C1—C4—C12	-66.58 (18)	C5—N1—C1—S1	-158.41 (14)
N1—C5—C10—C9	-176.5 (2)	C5—N1—C1—C4	79.6 (2)
N1—C5—C6—C7	177.9 (2)	C5—C10—C9—C8	-1.8 (5)
C3—S1—C1—N1	-16.08 (14)	C10—C5—C6—C7	-0.2 (3)
C3—S1—C1—C4	107.16 (14)	C10—C9—C8—C7	0.7 (5)
C2—N1—C1—S1	13.0 (2)	C9—C8—C7—C6	0.7 (4)
C2—N1—C1—C4	-108.99 (19)	C8—C7—C6—C5	-1.0 (4)
C2—N1—C5—C10	53.6 (3)	C6—C5—C10—C9	1.5 (4)
C2—N1—C5—C6	-124.5 (2)		

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

Cg2 is the centroid of the C5–C10 ring

<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>
C1—H1 \cdots O1 ⁱ	0.98	2.30	3.251 (2)	164
C3—H3A \cdots Cg2 ⁱⁱ	0.97	2.79	3.748 (2)	170

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y+1, -z+1$.**3-(4-Chlorophenyl)-2-trichloromethyl-1,3-thiazolidin-4-one (2)***Crystal data* $\text{C}_{10}\text{H}_7\text{Cl}_4\text{NOS}$ $M_r = 331.03$ Monoclinic, $P2_1/n$ $a = 10.907$ (2) \AA $b = 6.1686$ (12) \AA $c = 19.885$ (4) \AA $\beta = 96.02$ (3) $^\circ$ $V = 1330.5$ (5) \AA^3 $Z = 4$ $F(000) = 664$ $D_x = 1.653$ Mg m^{-3} Mo $K\alpha$ radiation, $\lambda = 0.71073$ \AA

Cell parameters from 5450 reflections

 $\theta = 2.2$ – 28.3 $^\circ$ $\mu = 1.03$ mm^{-1} $T = 298$ K

Block, colorless

 $0.25 \times 0.13 \times 0.1$ mm*Data collection*

Bruker SMART CCD area detector

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ω scans

Absorption correction: multi-scan

(SADABS; BRUKER, 2001)

 $T_{\min} = 0.868$, $T_{\max} = 0.9$

12273 measured reflections
 3302 independent reflections
 2883 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

$\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.0^\circ$
 $h = -12 \rightarrow 14$
 $k = -8 \rightarrow 8$
 $l = -26 \rightarrow 23$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.107$
 $S = 1.00$
 3302 reflections
 154 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
 $w = 1/[\sigma^2(F_o^2) + (0.069P)^2 + 0.2954P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.47 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.25 \text{ e } \text{\AA}^{-3}$

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.

SADABS V2.05 (BRUKER, 2001) was used for absorption correction. $R(\text{int})$ was 0.0443 before and 0.0167 after correction. The Ratio of minimum to maximum transmission is 0.8678. The $\lambda/2$ correction factor is 0.0015.

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.

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 $F^2 > 2\sigma(F^2)$ 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	0.57569 (4)	0.69974 (7)	0.15220 (2)	0.04596 (13)
C11	0.87252 (5)	0.86052 (9)	0.16992 (3)	0.05996 (16)
C13	0.81425 (5)	0.41241 (9)	0.19023 (3)	0.06369 (16)
C12	0.92398 (5)	0.53959 (10)	0.07182 (3)	0.06652 (18)
C14	0.85656 (7)	0.75038 (16)	-0.22386 (3)	0.0903 (2)
N1	0.67812 (13)	0.8295 (2)	0.04570 (6)	0.0375 (3)
C5	0.72089 (15)	0.8111 (3)	-0.01992 (8)	0.0379 (3)
C4	0.81846 (16)	0.6202 (3)	0.12933 (8)	0.0407 (3)
C1	0.68725 (15)	0.6485 (2)	0.09226 (7)	0.0356 (3)
H1	0.6640	0.5154	0.0672	0.043*
C2	0.61543 (16)	1.0100 (3)	0.06349 (8)	0.0412 (3)
O1	0.60428 (13)	1.17453 (19)	0.03021 (7)	0.0531 (3)
C8	0.80220 (18)	0.7763 (4)	-0.14533 (9)	0.0555 (5)
C6	0.68763 (17)	0.6339 (3)	-0.05979 (8)	0.0446 (4)
H6	0.6373	0.5273	-0.0443	0.053*
C3	0.56058 (19)	0.9806 (3)	0.12923 (10)	0.0507 (4)

H3A	0.4743	1.0222	0.1240	0.061*
H3B	0.6037	1.0704	0.1641	0.061*
C10	0.79363 (19)	0.9722 (3)	-0.04301 (10)	0.0555 (5)
H10	0.8158	1.0918	-0.0160	0.067*
C9	0.8334 (2)	0.9550 (4)	-0.10653 (11)	0.0657 (6)
H9	0.8811	1.0642	-0.1228	0.079*
C7	0.72899 (18)	0.6145 (3)	-0.12273 (9)	0.0518 (4)
H7	0.7079	0.4941	-0.1496	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0473 (3)	0.0496 (2)	0.0431 (2)	-0.00669 (17)	0.01474 (18)	-0.00091 (17)
C11	0.0493 (3)	0.0606 (3)	0.0695 (3)	-0.0120 (2)	0.0035 (2)	-0.0186 (2)
C13	0.0798 (4)	0.0595 (3)	0.0508 (3)	0.0090 (2)	0.0024 (2)	0.0189 (2)
C12	0.0631 (3)	0.0876 (4)	0.0511 (3)	0.0291 (3)	0.0168 (2)	-0.0008 (2)
C14	0.0881 (5)	0.1389 (6)	0.0497 (3)	0.0127 (4)	0.0347 (3)	0.0164 (3)
N1	0.0477 (7)	0.0317 (6)	0.0336 (6)	-0.0006 (5)	0.0070 (5)	0.0000 (5)
C5	0.0417 (8)	0.0386 (7)	0.0338 (7)	-0.0018 (6)	0.0058 (6)	0.0037 (6)
C4	0.0480 (9)	0.0404 (8)	0.0346 (8)	0.0026 (6)	0.0082 (6)	0.0001 (6)
C1	0.0449 (8)	0.0315 (6)	0.0307 (7)	-0.0035 (6)	0.0063 (6)	-0.0016 (5)
C2	0.0457 (8)	0.0349 (7)	0.0419 (8)	-0.0017 (6)	-0.0005 (6)	-0.0047 (6)
O1	0.0675 (9)	0.0331 (6)	0.0581 (8)	0.0029 (5)	0.0030 (6)	0.0019 (5)
C8	0.0502 (10)	0.0796 (13)	0.0390 (9)	0.0082 (9)	0.0155 (8)	0.0131 (9)
C6	0.0533 (10)	0.0455 (8)	0.0360 (8)	-0.0065 (7)	0.0098 (7)	0.0006 (6)
C3	0.0551 (10)	0.0501 (9)	0.0477 (10)	0.0101 (8)	0.0087 (8)	-0.0055 (8)
C10	0.0612 (11)	0.0503 (10)	0.0567 (11)	-0.0161 (8)	0.0139 (9)	0.0015 (8)
C9	0.0635 (13)	0.0725 (13)	0.0650 (13)	-0.0143 (10)	0.0256 (10)	0.0165 (11)
C7	0.0578 (11)	0.0627 (11)	0.0359 (8)	-0.0008 (9)	0.0090 (7)	-0.0040 (8)

Geometric parameters (Å, °)

S1—C1	1.8181 (16)	C2—O1	1.211 (2)
S1—C3	1.795 (2)	C2—C3	1.505 (3)
C11—C4	1.7595 (17)	C8—C9	1.368 (3)
C13—C4	1.7674 (17)	C8—C7	1.382 (3)
C12—C4	1.7766 (17)	C6—H6	0.9300
C14—C8	1.7349 (19)	C6—C7	1.379 (2)
N1—C5	1.436 (2)	C3—H3A	0.9700
N1—C1	1.4471 (18)	C3—H3B	0.9700
N1—C2	1.372 (2)	C10—H10	0.9300
C5—C6	1.377 (2)	C10—C9	1.382 (3)
C5—C10	1.380 (2)	C9—H9	0.9300
C4—C1	1.549 (2)	C7—H7	0.9300
C1—H1	0.9800		
C3—S1—C1	92.91 (8)	C9—C8—C14	119.59 (16)
C5—N1—C1	120.90 (12)	C9—C8—C7	121.07 (17)

C2—N1—C5	121.10 (13)	C7—C8—C14	119.33 (18)
C2—N1—C1	117.64 (13)	C5—C6—H6	120.0
C6—C5—N1	119.66 (14)	C5—C6—C7	119.99 (17)
C6—C5—C10	120.42 (16)	C7—C6—H6	120.0
C10—C5—N1	119.91 (15)	S1—C3—H3A	110.2
C11—C4—C13	109.17 (9)	S1—C3—H3B	110.2
C11—C4—C12	108.82 (10)	C2—C3—S1	107.67 (12)
C13—C4—C12	107.64 (9)	C2—C3—H3A	110.2
C1—C4—C11	111.83 (11)	C2—C3—H3B	110.2
C1—C4—C13	108.57 (11)	H3A—C3—H3B	108.5
C1—C4—C12	110.71 (11)	C5—C10—H10	120.2
S1—C1—H1	108.9	C5—C10—C9	119.67 (19)
N1—C1—S1	106.23 (10)	C9—C10—H10	120.2
N1—C1—C4	112.92 (13)	C8—C9—C10	119.64 (18)
N1—C1—H1	108.9	C8—C9—H9	120.2
C4—C1—S1	110.85 (10)	C10—C9—H9	120.2
C4—C1—H1	108.9	C8—C7—H7	120.4
N1—C2—C3	112.34 (14)	C6—C7—C8	119.18 (19)
O1—C2—N1	124.29 (16)	C6—C7—H7	120.4
O1—C2—C3	123.36 (16)		
C11—C4—C1—S1	-65.17 (12)	C1—S1—C3—C2	15.17 (13)
C11—C4—C1—N1	53.89 (15)	C1—N1—C5—C6	47.7 (2)
C13—C4—C1—S1	55.34 (12)	C1—N1—C5—C10	-133.18 (17)
C13—C4—C1—N1	174.40 (10)	C1—N1—C2—O1	177.64 (15)
C12—C4—C1—S1	173.30 (8)	C1—N1—C2—C3	-2.8 (2)
C12—C4—C1—N1	-67.64 (14)	C2—N1—C5—C6	-125.20 (17)
C14—C8—C9—C10	-177.79 (18)	C2—N1—C5—C10	54.0 (2)
C14—C8—C7—C6	178.93 (15)	C2—N1—C1—S1	13.99 (17)
N1—C5—C6—C7	-179.61 (16)	C2—N1—C1—C4	-107.72 (16)
N1—C5—C10—C9	-179.26 (18)	O1—C2—C3—S1	169.53 (14)
N1—C2—C3—S1	-10.00 (18)	C6—C5—C10—C9	-0.1 (3)
C5—N1—C1—S1	-159.12 (12)	C3—S1—C1—N1	-16.38 (12)
C5—N1—C1—C4	79.18 (17)	C3—S1—C1—C4	106.63 (12)
C5—N1—C2—O1	-9.3 (3)	C10—C5—C6—C7	1.2 (3)
C5—N1—C2—C3	170.25 (14)	C9—C8—C7—C6	-0.4 (3)
C5—C6—C7—C8	-1.0 (3)	C7—C8—C9—C10	1.5 (3)
C5—C10—C9—C8	-1.3 (3)		

Hydrogen-bond geometry (Å, °)

Cg2 is the centroid of the C5—C10 ring

<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>
C1—H1 \cdots O1 ⁱ	0.98	2.30	3.2643 (19)	168
C3—H3A \cdots Cg2 ⁱⁱ	0.97	2.85	3.797 (2)	166

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