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cis-2-(*tert*-Butyl)-4-(*p*-chlorophenylthio)-3-phenylacetyl-1,3-oxazolidin-5-one

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Abstract

The *tert*-butyl and *p*-chlorophenylthio groups occupy *cis* positions with respect to the oxazolidinone ring. The ring adopts a shallow envelope conformation with the N atom displaced 0.160 (9) Å from the least-squares plane through the other atoms of the ring. The degree of pyramidalization at the N atom is small (the angles at the N atom sum to 353.6°).

Comment

This structure determination continues a study on compounds prepared during investigations on diastereoselectivities of free-radical reactions (Beckwith & Chai, 1990; Beckwith, Chai & Tozer, 1992). We have previously reported the structure of *trans*-3-benzoyl-2-*tert*-butyl-4-isobutyl-1,3-oxazolidin-5-one (Willis, Beckwith & Tozer, 1991) in which the substituents at the 2 and 4 positions lie on opposite sides of the ring. There, the conformation of the ring is a shallow envelope with C5 lying 0.170 (4) Å from the plane of the other four atoms of the ring. In the present compound the substituents at the 2 and 4 positions are both situated on the same side of the oxazolidinone ring. The ring is again in a shallow envelope conformation, but now with N4 exoplanar to the other atoms of the ring. The angle between the least-squares plane through C5, O1, C2 and C3 and the plane of C3, N4 and C5 is 11.3 (5)°. Bond lengths and angles are comparable with corresponding dimensions in the previous compound, except for small variations about C3 which presumably arise from the more electron-donating nature of the *p*-chlorophenylthio group. Diagrams and most calculations were performed with the *Xtal*3.0 package (Hall & Stewart, 1990), as was the generation of the Crystallographic Information File used for the submission of this paper.

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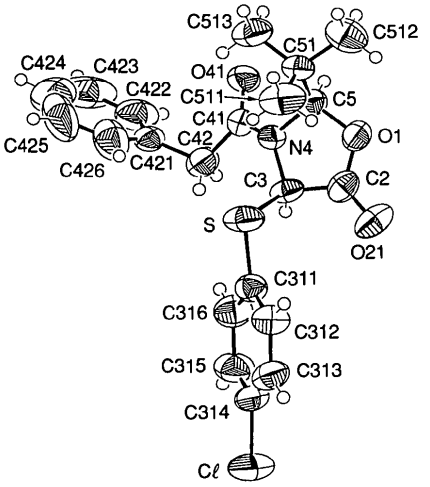

Fig. 1. View of $C_{21}H_{22}ClNO_3S$ showing the labelling of the non-H atoms. Thermal ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radius.

Experimental

Crystal data

$C_{21}H_{22}ClNO_3S$
M_r = 403.92
Monoclinic
$P2_1/c$
a = 11.419 (5) Å
b = 20.749 (8) Å
c = 8.946 (3) Å
β = 101.85 (3)°
Cell parameters from 25 reflections
θ = 10–14°
V = 2074 (1) Å^3
Z = 4
D_x = 1.293 Mg m^{-3}
Mo $K\alpha$
λ = 0.71069 Å
μ = 0.3 mm^{-1}
T = 293 K
Needle
0.12 × 0.12 × 0.35 mm
Colourless

Data collection

Philips PW 1100/20 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2703 measured reflections
2703 independent reflections
1235 observed reflections
Criterion: $I_{net} \geq 3\sigma(I_{net})$
θ_{max} = 22.48°
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 9$
3 standard reflections monitored every 90 reflections
intensity variation: 0%

Refinement

Refinement on F
Final R = 0.044
wR = 0.046
S = 1.371
1235 reflections
244 parameters
H atoms not refined
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{max}$ = 0.056
$\Delta\rho_{max}$ = 0.149 e Å^{-3}
$\Delta\rho_{min}$ = −0.175 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

0108-2701/92/030593-02$03.00

Data collection: Philips PW 1100/20 software 1976. Cell refinement: Philips PW 1100/20 software 1976. Data reduction: *PWREDU* (McLaughlin, 1983), *Xtal ADDREF, SORTRF*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *Xtal CRYLSQ*. Molecular graphics: *Xtal*. Software used to prepare material for publication: *Xtal BONDLA, CIFIO*.

The θ-scan width was $(0.8 + 0.346\tan\theta)°$, with θ-scan rate $1.2°\,min^{-1}$ and background counts for 8 s on each end of every scan. Large anisotropy of displacement parameters for C423–C425 suggests some disorder of the phenyl group. Distances and angles in this part of the structure are unreliable.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cl	0.3606 (2)	1.0271 (1)	0.1678 (2)	0.127 (2)
S	0.2243 (2)	0.9441 (1)	0.7811 (2)	0.083 (1)
O1	−0.0103 (3)	0.8279 (2)	0.7990 (5)	0.065 (3)
C2	0.0460 (6)	0.8531 (3)	0.6940 (8)	0.062 (4)
C3	0.1764 (6)	0.8592 (3)	0.7594 (6)	0.056 (4)
N4	0.1911 (5)	0.8268 (2)	0.9065 (5)	0.050 (3)
C5	0.0726 (5)	0.8140 (2)	0.9406 (6)	0.052 (4)
O21	−0.0056 (4)	0.8671 (2)	0.5677 (5)	0.091 (3)
C311	0.2565 (6)	0.9616 (3)	0.5986 (6)	0.059 (4)
C312	0.1948 (6)	1.0108 (3)	0.5150 (7)	0.074 (4)
C313	0.2271 (7)	1.0310 (3)	0.3822 (7)	0.082 (5)
C314	0.3189 (7)	1.0014 (4)	0.3342 (7)	0.078 (5)
C315	0.3786 (6)	0.9512 (4)	0.4133 (8)	0.087 (5)
C316	0.3485 (6)	0.9313 (3)	0.5489 (7)	0.079 (5)
C41	0.2856 (6)	0.7878 (3)	0.9682 (7)	0.061 (4)
O41	0.2743 (3)	0.7467 (2)	1.0628 (4)	0.076 (3)
C42	0.4019 (6)	0.7994 (3)	0.9176 (7)	0.082 (4)
C421	0.5054 (5)	0.7990 (4)	1.0494 (7)	0.059 (4)
C422	0.5630 (8)	0.7444 (5)	1.1045 (9)	0.102 (6)
C423	0.658 (1)	0.7431 (9)	1.229 (2)	0.18 (2)
C424	0.686 (2)	0.800 (1)	1.291 (2)	0.21 (2)
C425	0.638 (1)	0.8569 (8)	1.248 (2)	0.18 (1)
C426	0.5434 (7)	0.8553 (4)	1.123 (1)	0.108 (6)
C51	0.0379 (5)	0.8521 (3)	1.0714 (6)	0.060 (4)
C511	0.0379 (6)	0.9243 (3)	1.0422 (7)	0.092 (5)
C512	−0.0869 (6)	0.8310 (3)	1.0853 (8)	0.097 (5)
C513	0.1255 (6)	0.8370 (3)	1.2198 (7)	0.086 (5)

Table 2. *Geometric parameters* (Å, °)

Cl—C314	1.738 (7)	C311—C316	1.374 (10)
S—C3	1.844 (6)	C312—C313	1.380 (10)
S—C311	1.783 (6)	C313—C314	1.359 (11)
O1—C2	1.347 (9)	C314—C315	1.362 (10)
O1—C5	1.446 (6)	C315—C316	1.390 (10)
C2—C3	1.490 (9)	C41—O41	1.226 (8)
C2—O21	1.198 (8)	C41—C42	1.509 (10)
C3—N4	1.457 (7)	C42—C421	1.488 (8)
N4—C5	1.471 (8)	C51—C511	1.520 (9)
N4—C41	1.371 (8)	C51—C512	1.520 (10)
C5—C51	1.530 (8)	C51—C513	1.522 (8)
C311—C312	1.371 (8)		
C3—S—C311	102.1 (3)	Cl—C314—C313	119.8 (5)
C2—O1—C5	111.6 (4)	Cl—C314—C315	119.0 (6)
O1—C2—C3	109.9 (5)	C313—C314—C315	121.2 (7)
O1—C2—O21	122.6 (6)	C314—C315—C316	119.5 (7)
C3—C2—O21	127.4 (7)	C311—C316—C315	119.3 (6)
S—C3—C2	111.9 (4)	N4—C41—O41	120.2 (6)
S—C3—N4	111.7 (3)	N4—C41—C42	116.9 (6)
C2—C3—N4	103.6 (5)	O41—C41—C42	122.8 (6)
C3—N4—C5	109.3 (4)	C41—C42—C421	111.5 (5)
C3—N4—C41	124.9 (5)	C42—C421—C422	122.8 (7)
C5—N4—C41	119.4 (5)	C42—C421—C426	119.9 (7)
O1—C5—N4	104.3 (4)	C422—C421—C426	117.3 (6)
O1—C5—C51	110.0 (4)	C5—C51—C511	111.6 (5)
N4—C5—C51	117.4 (4)	C5—C51—C512	107.9 (5)
S—C311—C312	118.1 (5)	C5—C51—C513	109.4 (5)
S—C311—C316	121.2 (4)	C511—C51—C512	109.4 (5)
C312—C311—C316	120.3 (6)	C511—C51—C513	109.3 (5)
C311—C312—C313	119.9 (6)	C512—C51—C513	109.2 (5)
C312—C313—C314	119.6 (6)		
C5—O1—C2—C3	−0.7 (6)		
O1—C2—C3—N4	7.3 (6)		
C2—C3—N4—C5	−11.1 (5)		
C3—N4—C5—O1	10.9 (5)		
N4—C5—O1—C2	−6.2 (5)		

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54839 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL0011]

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