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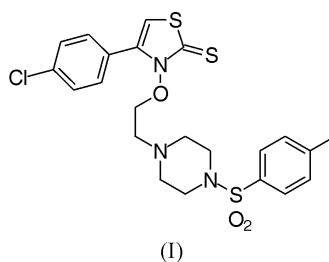
Key indicators

Single-crystal X-ray study
T = 299 K
Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
R factor = 0.062
wR factor = 0.135
Data-to-parameter ratio = 18.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The N—O bond as an element of chirality:
(*P*)-4-(*p*-chlorophenyl)-3-{2-[4-(*p*-toluene-
sulfonyl)piperazino]ethoxy}thiazole-2(3*H*)-
thione

The title compound, $\text{C}_{22}\text{H}_{24}\text{ClN}_3\text{O}_3\text{S}_3$, an *N*-alkoxy-substituted 4-(*p*-chlorophenyl)thiazole-2(3*H*)-thione, exhibits a (*P*) configuration at the N—O bond. The *p*-chlorophenyl-substituent is twisted by $-40.3(6)^\circ$ out of the thiazole-2(3*H*)-thione plane. The piperazino spacer in the *N*-alkoxy side chain adopts a chair conformation, with its substituents at the N atoms located in equatorial positions. The 1,2-ethanamine bridge, which connects the thiazolethione and the piperazine moieties, exhibits an orthogonal arrangement.

Comment

The title compound, (I), was obtained from the reaction of the *N*-(hydroxy)-4-(*p*-chlorophenyl)thiazole-2(3*H*)-thione tetraethylammonium salt and 4-(2-chloroethyl)piperazino-1-*p*-toluenesulfonamide in DMF (Hartung *et al.*, 1999). The latter material is a minor contaminant of alkyl tosylates if they are prepared from *p*-toluenesulfonic acid chloride, 1,4-diazabicyclo[2.2.2]octane, and a sterically demanding alcohol (Hartung *et al.*, 1997). The identity of heterocyclic compound (I) was established by X-ray crystallography and its structure is reported here.



Compound (I) crystallizes in the non-centrosymmetric space group $P2_12_12_1$ (orthorhombic). The C2—S2 [1.655 (5) Å] and N3—O3 [1.394 (4) Å] distances were interpreted as C=S and N—O bonds. All bond lengths are in agreement with corresponding distances in structurally related derivatives that have been documented in the literature (Rochester *et al.*, 1987; Ugozzoli & Andreotti, 1987; Shin & Lim, 1995; Hartung *et al.*, 1999).

The absolute (*P*) configuration at the N—O bond of (I) [C2—N3—O1—C12 = $94.1(4)^\circ$] (Hartung *et al.*, 2001) was established with the aid of the Flack (1983) parameter [$-0.05(10)$]. An almost orthogonal arrangement is found for the 1,2-diacceptor-substituted C_2 fragment [O3—C12—C13—N1 = $89.4(5)^\circ$]. The *p*-chlorophenyl group in (I) is twisted out of the thiazole-2(3*H*)-thione plane [C7—C6—C4—N3 = $-40.3(6)^\circ$]. The piperazino subunit of the *N*-alkoxy side chain adopts a chair conformation, with the substituents at the N

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atoms located in equatorial positions [C16–C17–N1–C13 = –176.6 (4)° and C17–C16–N2–S3 = –164.8 (3)°].

Experimental

The title compound gradually crystallized from a mixture of petroleum ether/CH₂Cl₂ (Hartung & Špehar, 2003).

Crystal data

C₂₂H₂₄ClN₃O₃S₃

M_r = 510.07

Orthorhombic, *P*2₁2₁2₁

a = 8.227 (1) Å

b = 14.098 (1) Å

c = 22.178 (1) Å

V = 2572.3 (4) Å³

Z = 4

D_x = 1.317 Mg m^{–3}

Mo *K*α radiation

Cell parameters from 1846 reflections

θ = 3.2–16.9°

μ = 0.42 mm^{–1}

T = 299 (2) K

Needle, pale yellow

0.30 × 0.12 × 0.06 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer

ω scans

Absorption correction: none

17 136 measured reflections

5188 independent reflections

3163 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.055

θ_{\max} = 26.4°

h = –10 → 5

k = –17 → 17

l = –27 → 27

Refinement

Refinement on *F*²

R [*F*² > 2σ(*F*²)] = 0.062

wR (*F*²) = 0.135

S = 0.97

5188 reflections

289 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2]$

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

(Δ/σ)_{max} = 0.002

$\Delta\rho_{\max} = 0.30 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.21 \text{ e \AA}^{-3}$

Absolute structure: Flack (1983),

2192 Friedel pairs

Flack parameter = –0.05 (10)

Table 1

Selected geometric parameters (Å, °).

C2–N3	1.356 (5)	C4–N3	1.405 (5)
C2–S2	1.655 (5)	C4–C6	1.478 (6)
C2–S1	1.729 (5)	C5–S1	1.721 (4)
C4–C5	1.330 (6)	N3–O3	1.394 (4)
N3–C2–S2	127.4 (4)	C2–N3–O3	120.1 (4)
N3–C2–S1	106.5 (3)	C2–N3–C4	118.2 (4)
S2–C2–S1	126.1 (3)	O3–N3–C4	121.7 (3)
C5–C4–N3	109.9 (4)	N3–O3–C12	110.1 (3)
C5–C4–C6	128.2 (4)	C5–S1–C2	92.8 (2)
C4–C5–S1	112.5 (3)		
N3–C4–C5–S1	1.1 (5)	C16–C17–N1–C14	60.1 (5)
C6–C4–C5–S1	–176.5 (4)	C15–C14–N1–C17	–58.3 (5)
O3–C12–C13–N1	89.4 (5)	C14–C15–N2–C16	–57.3 (5)
N1–C14–C15–N2	57.0 (5)	C17–C16–N2–C15	57.8 (5)
N2–C16–C17–N1	–59.7 (5)	C17–C16–N2–S3	–164.8 (3)
C16–C17–N1–C13	–176.6 (4)	C2–N3–O3–C12	94.1 (4)

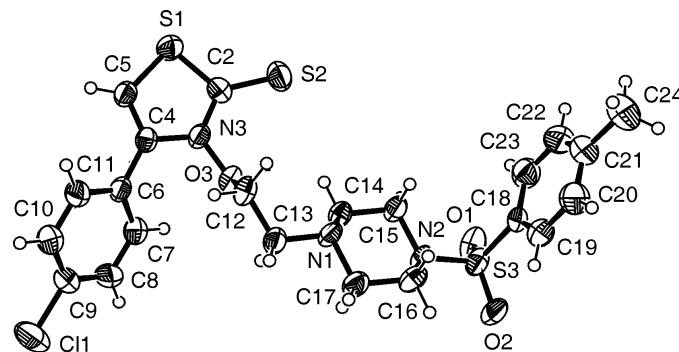


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2002); software used to prepare material for publication: *SHELXL97*.

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