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N-Benzoyl-*N*′-(2-{2-[2-(*N*-benzoylthioureido)-phenoxy]ethoxy}phenyl)thiourea

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.069
wR factor = 0.188
Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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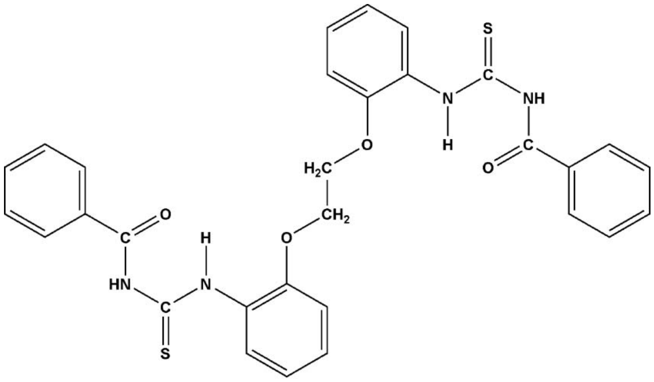
The molecule of the title compound, $C_{30}H_{26}N_4O_4S_2$, has an inversion centre. The molecule is planar, with a mean devation of 0.179 Å from the least-squares plane defined by the 40 constituent atoms (excluding H atoms). The crystal structure has inversion-related intermolecular N—H···S hydrogen bonds and intramolecular N—H···O hydrogen bonds.

Comment

In the last few decades, *N*-alkyl- and *N*,*N*-dialkyl-*N*′-acyl (aroyl) thiourea compounds and their coordination chemistry have been studied for their potential use in extraction, separation, medicine, agriculture and analytical chemistry (Schroeder, 1955; Antholine & Taketa, 1982). The crystal structures of such compounds have revealed many significant structural phenomena, including inter- and intramolecular hydrogen bonds and π–π interactions between aromatic rings, so they are also of current interest in supramolecular chemistry and host–guest chemistry (Ramnathan *et al.*, 1995; Wamhoff *et al.*, 1994; Dannecker *et al.*, 1979; Koch *et al.*, 1999, 2001; Bourne *et al.*, 2005). In the light of these findings, and as part of our work on the synthesis, biological activity, coordination behaviour and supramolecular chemistry of thiourea derivatives (Zhang *et al.*, 2001, 2003, 2006), we report here the synthesis and crystal structure of the title compound, (I) (Fig. 1).



(I)

In the centrosymmetric compound (I), bond lengths and angles are as expected for this type of compound (Zhang *et al.*, 2001, 2003, 2006). The dihedral angle between the C1–C6 phenyl ring and the C7–C12 benzene ring is 10.0 (2)°, while the centrosymmetrically related benzene rings are parallel to each other. The benzene rings have different orientations with respect to the acylthiourea unit [–C(O)NHC(S)NH–], with torsion angles C13—N1—C14—O1 = 0.2 (5)°, C13—N1—

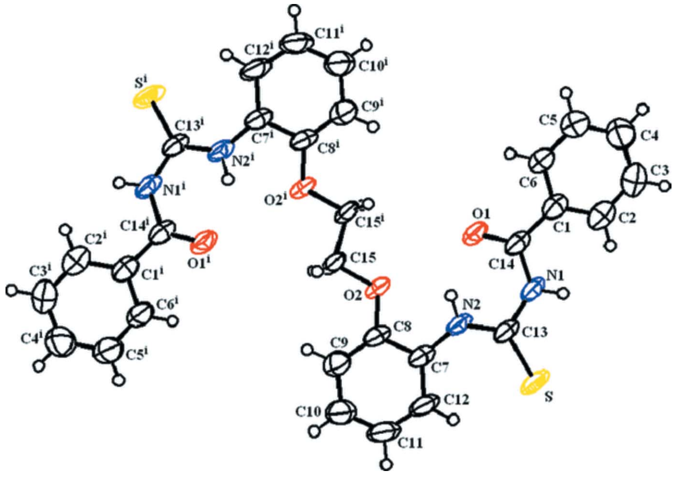


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $-x$, $1 - y$, $-z$.]

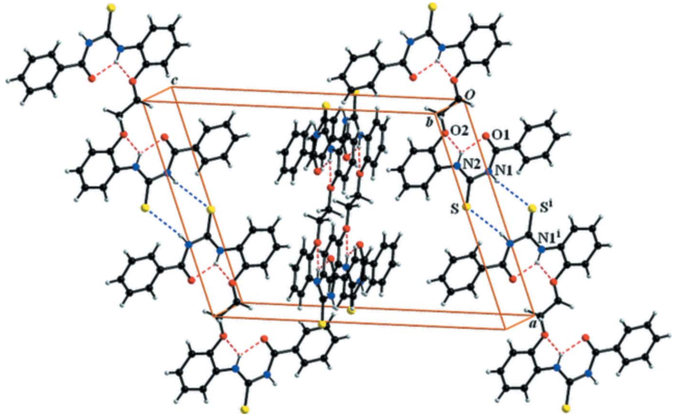


Figure 2
Hydrogen-bond interactions (dashed lines) in (I). [Symmetry code: (i) $1 - x$, $-y$, $-z$.]

C14—C1 = 177.6 (2)°, C7—N2—C13—N1 = 176.6 (2)°, C7—N2—C13—S = 2.1 (5)°, C14—N1—C13—S1 = 175.9 (2)° and C14—N1—C13—N2 = 2.9 (4)°.

Aromatic π–π stacking interactions are not observed. Inter- and intramolecular hydrogen bonds in the structure of (I) are shown in Fig. 2 and Table 1.

Experimental

The title compound was prepared according to a one-pot method in two steps, based on a procedure described in the literature (Douglass & Dains, 1934) with some improvements. A solution of benzoyl chloride (5 mmol) in CH_3COCH_3 (5 ml) was added slowly to a solution of NH_4SCN (6 mmol) in CH_3COCH_3 (5 ml) in a dried flask and the reaction mixture was stirred at room temperature for 1 h. 2,2′-[Ethane-1,2-diylbis(oxy)]dianiline (2.5 mmol) was then added slowly to the reaction mixture, after which it was stirred at room temperature for a further 1 h. When the reaction was complete, the mixture was poured on to a water–ice mixture and stirred for more than 10 min. It was then filtered and an off-white precipitate was obtained. The pure product was obtained by washing three times with ethanol, followed by recrystallization from dimethylformamide. Single crystals suitable for X-ray structure determination were obtained by slow diffusion of ethanol into a solution of (I) in dimethylformamide.

Crystal data

$C_{30}H_{26}N_4O_4S_2$
$M_r = 570.67$
Monoclinic, $P2_1/n$
$a = 14.1248$ (7) Å
$b = 5.5175$ (3) Å
$c = 19.179$ (1) Å
$\beta = 108.408$ (2)°

$V = 1418.21$ (13) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.23$ mm^{-1}
$T = 294$ (2) K
0.39 × 0.23 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
6813 measured reflections

2485 independent reflections
1689 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.170$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.188$
$S = 0.95$
2485 reflections

181 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.40$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.40$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N1···S$^{\rm i}$	0.86	2.81	3.549 (2)	145
N2—H2N2···O1	0.86	1.91	2.634 (3)	141
N2—H2N2···O2	0.86	2.13	2.547 (3)	109

Symmetry code: (i) $-x + 1$, $-y$, $-z$.

All H atoms were placed in idealized positions and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms and 0.96 Å for methyl H atoms, and N—H = 0.86 Å, and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for aromatic H atoms, $1.5U_{\rm eq}$(C) for methyl H atoms and $1.2U_{\rm eq}$(N) for amine H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXTL*.

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