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2,3-Bis(5-bromo-2-thienyl)-6,7-dichloroquinoxaline

Vanessa Stacy,[a] Guy Crundwell,[a]* James B. Updegraff III,[b] Matthias Zeller[b] and Allen D. Hunter[b]

[a]Department of Chemistry, Central Connecticut State University, New Britain, CT 06053, USA, and [b]Department of Chemistry, Youngstown State University, One University Plaza, Youngstown, Ohio 44555-3663, USA

Correspondence e-mail: crundwellg@mail.ccsu.edu

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.031
wR factor = 0.067
Data-to-parameter ratio = 24.7

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

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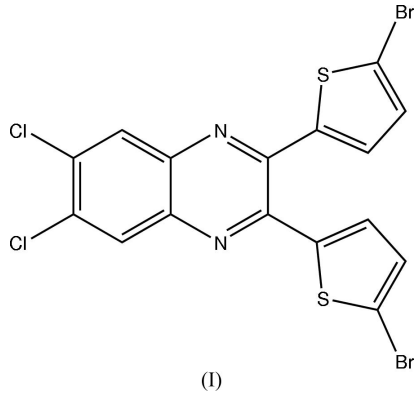
The title compound, $C_{16}H_6Br_2Cl_2N_2S_2$, was synthesized from the reaction of equimolar amounts of 1,2-diamino-4,5-dichlorobenzene and 1,2-bis(5-bromothien-2yl)ethanedione. The crystal structure was determined at 100 K.

Comment

During our investigations of the metal-binding properties of dithienylquinoxalines, we have published crystal structures of 2,3-dithien-2-ylquinoxaline (Crundwell *et al.*, 2003), its protonated perchlorate salt (Foss *et al.*, 2004) and 2,3-bis(5-bromothien-2-yl)quinoxaline (Crundwell *et al.*, 2004). We report here the crystal structure of the title compound, (I) (Fig. 1).



(I)

All bond lengths and angles fall within established ranges; however, in accordance with past publications where we have been interested in the planarity of the aryl substituents in order to assess potential metal-binding through thienyl ring S atoms, the angles between thienyl rings and quinoxaline are as follows (Crundwell *et al.*, 2004). The angle between ring 1 (C9–C12/S1) and the quinoxaline moiety is 18.82 (9)°; whereas the angle between ring 2 and the quinoxaline subunit is 36.66 (5)°.

Experimental

To a 50 ml round-bottomed flask equipped with a reflux condenser were added 1,2-bis(5-bromothien-2yl)ethanedione (0.38 g, 1 mmol) and 4,5-dichloro-1,2-phenylenediamine (0.177 g, 1 mmol) (which were crushed together in a mortar and pestle), and ethanol (about 20 ml). The mixture was heated with refluxing for 5.5 h. After this time, the resulting green–yellow solution was placed in an ice bath. The yellow precipitate was vacuum-filtered and washed with cold water and ethanol. The product was purified by recrystallization from ethanol and yielded (I) as a yellow solid (0.2 g, yield 52.6%). Crystals of (I) melted at 448 K.

Crystal data

$C_{16}H_6Br_2Cl_2N_2S_2$
$M_r = 521.07$
Tetragonal, $P\bar{4}2_1c$
$a = 20.2456\ (6)$ Å
$c = 8.5209\ (5)$ Å
$V = 3492.6\ (3)$ Å^3
$Z = 8$
$D_x = 1.982$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6538 reflections
$\theta = 2.6$–$30.3°$
$\mu = 5.19$ mm^{-1}
$T = 100\ (2)$ K
Needle, yellow
$0.19 \times 0.05 \times 0.05$ mm

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.667$, $T_{max} = 0.77$
41333 measured reflections
5351 independent reflections
4694 reflections with $I > 2\sigma(I)$
$R_{int} = 0.064$
$\theta_{max} = 30.6°$
$h = -28 \rightarrow 28$
$k = -28 \rightarrow 28$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.067$
$S = 1.01$
5351 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.012$
$\Delta\rho_{max} = 0.65$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}
Extinction correction: none
Absolute structure: Flack (1983), 2370 Friedel pairs
Flack parameter: 0.001 (6)

H atoms were included in calculated positions, with a C—H distance of 0.95 Å, and were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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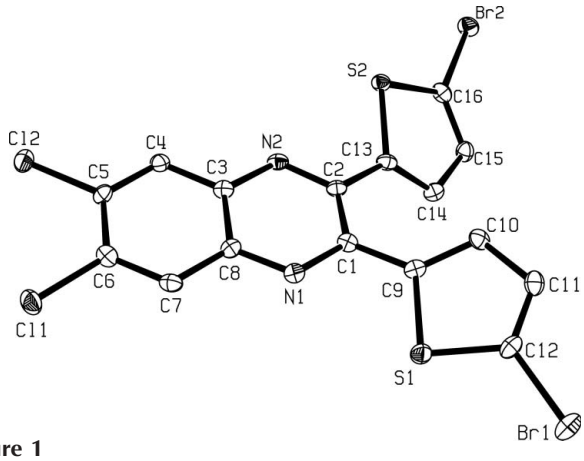

Figure 1
A view of (I) (Farrugia, 1997). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

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