

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Editors: **W. Clegg** and **D. G. Watson**

2,5-Bis(4-*tert*-butylphenyl)-3,4-dinitrothiophene

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2,5-Bis(4-*tert*-butylphenyl)-3,4-dinitrothiophene

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Key indicators

Single-crystal X-ray study

$T = 100\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

R factor = 0.030

wR factor = 0.077

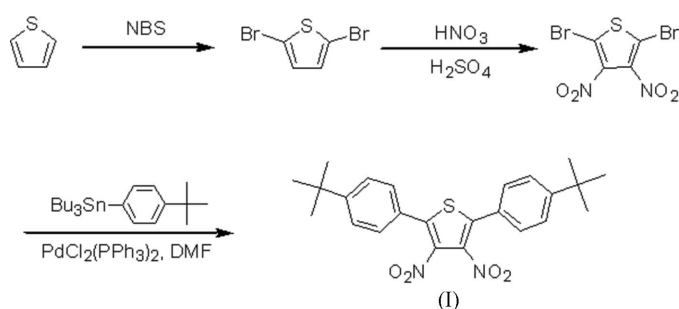
Data-to-parameter ratio = 13.9

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

The title compound, $\text{C}_{24}\text{H}_{26}\text{N}_2\text{O}_4\text{S}$, is a precursor for the production of low band gap conjugated polymers. The dihedral angles between the thiophene and benzene rings are $31.5(1)$ and $52.2(1)^\circ$, and that between the two benzene rings is $35.7(1)^\circ$.

Comment

The title compound, (I), has been shown to be an excellent precursor for the production of low band gap conjugated polymers and organic light-emitting devices, *etc.* As indicated in the scheme, standard procedures were used to synthesize (I) in high yield.



The molecular structure is shown in Fig. 1. The double bonds and C—C single bond of the substituted thiophene ring are slightly shorter than those of the parent thiophene, while the S—C single bond is slightly elongated (Bak *et al.*, 1961). The dihedral angles between the thiophene (S/C2—C5) and benzene rings (C11—C16 and C21—C26) are $52.2(1)$ and $31.5(1)^\circ$, respectively, and that between the two benzene rings is $35.7(1)^\circ$.

Experimental

Compound (I) was prepared *via* several synthetic steps, such as bromination, nitration and a palladium-catalysed aromatic Stillbenzene coupling reaction (see scheme). The compound is soluble in appropriate organic solvents and was considerably purified by column chromatography (Thomas *et al.*, 2002). The compound was synthesized by the following procedure. A two-necked round-bottomed flask was charged with $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (140 mg), tributyl(4-*tert*-butylphenyl)stannane (9.3 g, 22 mmol), 2,5-dibromo-3,4-dinitrothiophene (3.32 g, 10 mmol) and DMF (20 ml), and the reaction mixture stirred under nitrogen and heated at 343 K for 16 h. After cooling, the mixture was diluted with diethyl ether and the organic phase was washed with water and brine. After drying over anhydrous MgSO_4 and removing the volatiles, the residue was purified by column chromatography using $\text{CH}_2\text{Cl}_2/n$ -hexane as eluent, followed by recrystallization from CH_2Cl_2 and MeOH to yield 2.4 g (60%) of (I)

Received 2 August 2005

Accepted 16 August 2005

Online 14 September 2005

as a pale-yellow solid. Crystals suitable for X-ray diffraction were grown from a CH_2Cl_2 solution layered with hexane at room temperature.

Crystal data

$\text{C}_{24}\text{H}_{26}\text{N}_2\text{O}_4\text{S}$
 $M_r = 438.53$
 Monoclinic, $P2_1/c$
 $a = 14.026$ (3) Å
 $b = 11.120$ (2) Å
 $c = 14.491$ (3) Å
 $\beta = 99.349$ (9)°
 $V = 2230.2$ (8) Å³
 $Z = 4$

$D_x = 1.306$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 7514 reflections
 $\theta = 2.9\text{--}31.2^\circ$
 $\mu = 0.18$ mm⁻¹
 $T = 100$ (2) K
 Prism, yellow
 $0.32 \times 0.24 \times 0.2$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 32280 measured reflections
 3915 independent reflections

3530 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -12 \rightarrow 16$
 $k = -13 \rightarrow 13$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.077$
 $S = 1.04$
 3915 reflections
 281 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 1.2009P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0031 (5)

Table 1

Selected geometric parameters (Å, °).

S—C2	1.7224 (14)	C3—C4	1.4198 (19)
S—C5	1.7327 (14)	C4—C5	1.361 (2)
C2—C3	1.3727 (19)		
C2—S—C5	94.37 (7)	C2—C3—C4	113.62 (12)
C3—C2—S	108.90 (10)	C5—C4—C3	114.49 (12)

H atoms were positioned geometrically and refined as riding, with C—H = 0.95–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{Me}})$.

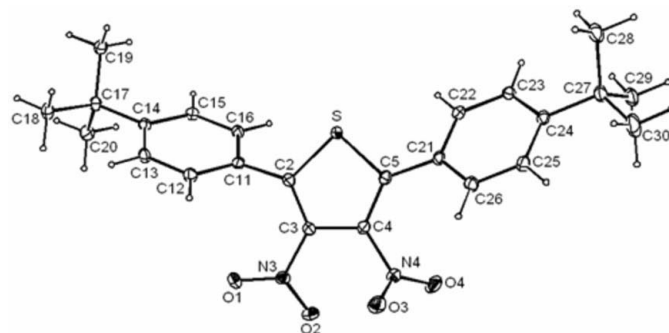


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work is partially supported by the Institute of Chemistry, Academia Sinica, the Department of Chemistry, National Taiwan Normal University, and Kang-Ning Junior College of Medical Care and Management.

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