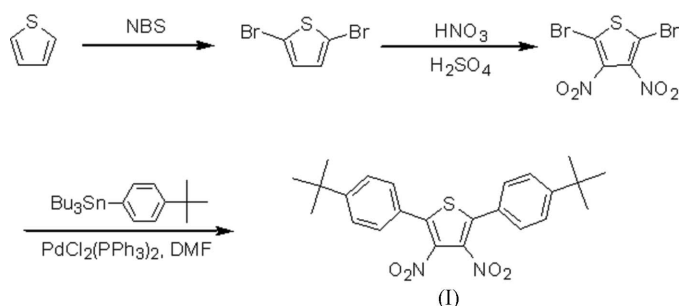


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Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.030
 wR factor = 0.077
Data-to-parameter ratio = 13.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.2,5-Bis(4-*tert*-butylphenyl)-3,4-dinitrothiopheneThe 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)°, and that between the two benzene rings is 35.7 (1)°.Received 2 August 2005
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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)°, respectively, and that between the two benzene rings is 35.7 (1)°.

Experimental

Compound (I) was prepared *via* several synthetic steps, such as bromination, nitration and a palladium-catalysed aromatic Stillb-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)

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 $1.5U_{\text{eq}}(\text{C}_{\text{Me}})$.

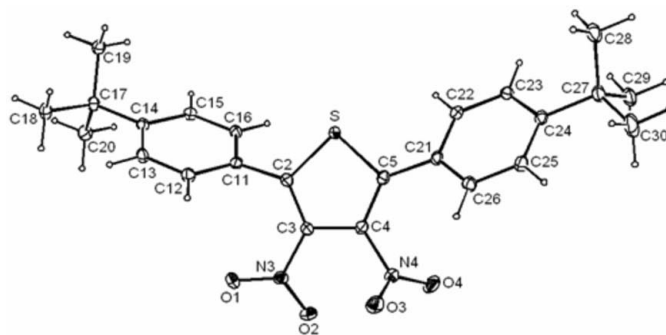


Figure 1

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).

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