

3-Benzyl-1-(3-nitrophenylsulfonyl)-1H-pyrazol-5-amine

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Key indicators

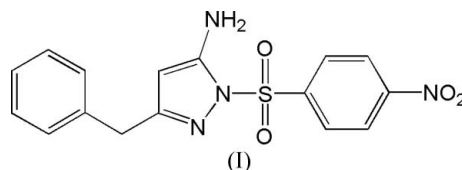
Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
Disorder in main residue
 R factor = 0.061
 wR factor = 0.239
Data-to-parameter ratio = 11.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{16}\text{H}_{14}\text{N}_4\text{O}_4\text{S}$, was synthesized by the reaction of 3-oxo-4-phenylbutanenitrile and 4-nitrobenzenesulfonylhydrazide. The pyrazole ring is almost planar. The two O atoms of the nitro group are disordered in a 0.84 (2):0.16 (2) ratio.

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Comment

Pyrazoles have a widespread occurrence as substructures in a large variety of compounds with important biological activities and pharmacological properties. They can be used in the synthesis of a number of biologically active compounds (Dastrup *et al.*, 2004; Haddad & Baron, 2002). The title compound, (I), was synthesized by the reaction of 3-oxo-4-phenylbutanenitrile and 4-nitrobenzenesulfonylhydrazide. An X-ray structure determination of (I) was carried out and the results are reported here.



The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. In (I), the pyrazole ring is almost

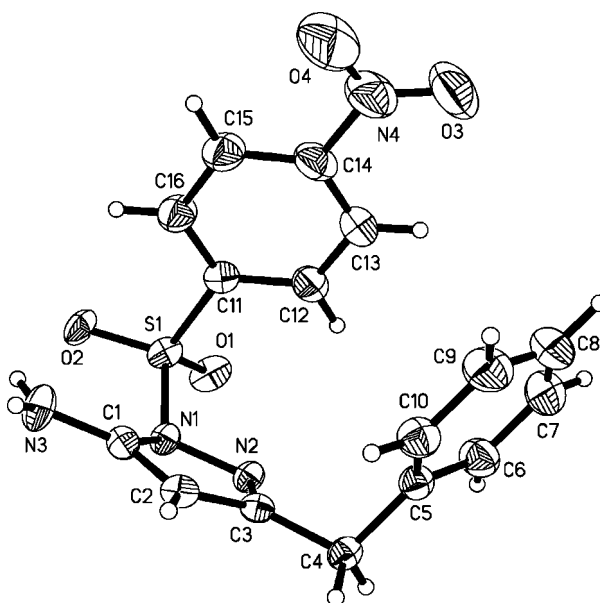


Figure 1
The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

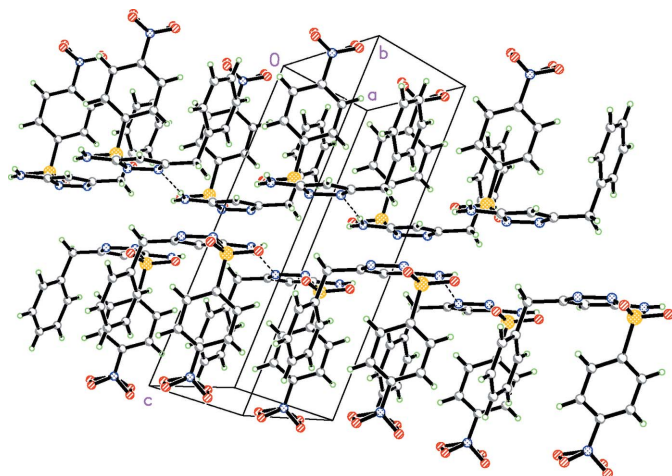


Figure 2
The packing of (I), viewed down the *a* axis.

planar, with an r.m.s. deviation of 0.0159 (3) Å. The dihedral angles between the pyrazole ring and the two benzene rings C5–C10 and C11–C15) are 73.6 (3) and 63.6 (3)°, while the dihedral angle between the two benzene rings is 43.2 (3)°. The S atom has a distorted tetrahedral geometry, with the N1–S1–C11 and O1–S1–C11 angles deviating significantly from the ideal tetrahedral value (Table 1). In the crystal structure, there are intermolecular N–H···N hydrogen bonds between the amino N atom and the N atom of the pyrazole ring, which stabilize the crystal structure (Table 2 and Fig. 2).

Experimental

The title compound was synthesized by the reaction of 3-oxo-4-phenylbutanenitrile (1.25 mmol) and 4-nitrobenzenesulfonylhydrazide (1.25 mmol) in glacial acetic acid (5 ml) was stirred at room temperature for 6 h. The product was obtained (yield 70%) by silica-gel column chromatography. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a dichloromethane solution.

Crystal data

$C_{16}H_{14}N_4O_4S$	$V = 828.7$ (2) Å ³
$M_r = 358.37$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.436$ Mg m ⁻³
$a = 6.1838$ (10) Å	Mo $K\alpha$ radiation
$b = 8.4685$ (14) Å	$\mu = 0.23$ mm ⁻¹
$c = 16.608$ (2) Å	$T = 293$ (2) K
$\alpha = 95.273$ (7)°	Prism, colourless
$\beta = 96.009$ (8)°	$0.24 \times 0.20 \times 0.18$ mm
$\gamma = 105.084$ (14)°	

Data collection

Rigaku Saturn diffractometer	8136 measured reflections
ω scans	2901 independent reflections
Absorption correction: multi-scan (<i>REQAB</i> ; Jacobson, 1998)	1859 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.948$, $T_{\max} = 0.961$	$R_{\text{int}} = 0.042$
	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.239$
 $S = 1.00$
 2901 reflections
 254 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1676P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.79$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.079 (18)

Table 1

Selected bond angles (°).

O1–S1–N1	106.65 (15)	O2–S1–C11	109.17 (16)
O2–S1–N1	106.53 (15)	N1–S1–C11	102.06 (16)
O1–S1–C11	110.16 (17)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N3–H3B···N2 ⁱ	0.85 (5)	2.17 (5)	2.997 (4)	162 (4)

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

H atoms attached to the C atoms were positioned geometrically and refined as riding, with C–H = 0.93–0.97 Å, and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. N-bound H atoms were refined with N3–H3A = 0.93 (6) Å, N3–H3B = 0.85 (5) Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$. The two O atoms of the nitro group are disordered over two sites; the occupancies are 0.84 (2) and 0.16 (2).

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *CrystalStructure* (Rigaku/MS, 2005).

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