

Stewart, Davidson & Simpson (1965). The positional parameters and the equivalent isotropic thermal parameters of the non-H atoms are listed in Table 1; * bond lengths and angles are given in Table 2. The thermal ellipsoid drawing (*SHELXTL-Plus*; Sheldrick, 1987) of the molecule with the atom-labelling scheme is given in Fig. 1. The ring puckering parameters (Cremer & Pople, 1985; Boeyens, 1978; Boessenkool & Boeyens, 1980) were calculated using program *PLATON* (Spek, 1990).

Related literature. Abboud, Simonsen, Tyllick & Roberts (1988, 1991); Cioranescu, Banciu, Jelescu, Rentzea, Elian & Nenitzescu (1969*a,b*); Cristol & Noreen (1976).

The authors acknowledge the support of the Robert A. Welch Foundation [Grants F-017 (SHS) and F-1055 (RMR)].

* Tables of the crystallographic data, anisotropic thermal parameters, H-atom positional parameters and bond lengths and angles, a packing diagram and the structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53541 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1991). **C47**, 674–676

2-Amino-4-methyl-5-phenylthiophene-3-carboxamide

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(Received 16 May 1990; accepted 31 July 1990)

Abstract. C₁₂H₁₂N₂OS, *M_r* = 232.2, triclinic, *P* $\bar{1}$, *a* = 7.690 (3), *b* = 7.898 (3), *c* = 9.923 (2) Å, α = 67.032 (2), β = 83.92 (2), γ = 78.885 (2)°, *V* = 544.1 (3) Å³, *Z* = 2, *D_x* = 1.418 g cm⁻³, $\lambda(\text{Cu K}\alpha)$ =

1.5418 Å, μ = 23.81 cm⁻¹, *F*(000) = 244, *T* = 295 K, *R* = 0.05 for 2002 observed reflections [*I* > 3σ(*I*)]. Molecular geometry is within the normal limits. The thiophene ring is planar [max. deviation: C(2), 0.003 (2) Å] as is the phenyl ring [max. deviation: C(56), 0.010 (2) Å]. The amino, carboxamido, methyl and phenyl groups are attached preferentially to

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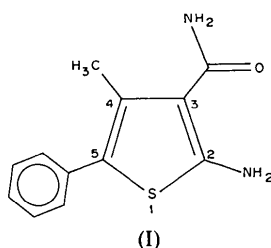
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positions 2, 3, 4 and 5 of the thiophene ring. The C—S distances are 1.745 (2) and 1.722 (2) Å. The C—S—C bond angle is 92.0 (1)°. The distances C(31)—O(1) = 1.234 (2) and C(31)—N(2) = 1.336 (3) Å of the carboxamido group are typical of the amide C—N and carbonyl C=O bond lengths, respectively. The steric repulsion between the phenyl ring and the substituent group (CH₃) at the adjacent C atom C(4) of the thiophene ring is minimized by (i) the rotation of the phenyl ring [dihedral angle 54.9 (1)° between the phenyl and thiophene rings] and (ii) the widening of the external angle at C(5) by 10.3 (3)° [difference between the angles C(4)—C(5)—C(51) and S(1)—C(5)—C(51)]. The molecule exhibits both intra- and intermolecular N—H...O type H bonding. The intramolecular hydrogen bonding, N(1)—H(1)...O(1), has a 'pulling effect' on the C(4)—C(3)—C(31) bond angle which is evident from the widening of this bond angle by 7.4 (3)° [difference between the angles C(4)—C(3)—C(31) and C(2)—C(3)—C(31)].

Experimental. The title compound (I) was prepared by the following procedure: benzyl methyl ketone was allowed to undergo a Cope reaction (Cope, Hofmann, Wyckoff & Hardenberg, 1941) with cyanoacetamide, followed by a Gewald reaction (Gewald, 1962, 1965; Gewald, Schinke & Bottcher, 1966) in the presence of sulfur to form 2-amino-4-methyl-5-phenylthiophene-3-carboxamide as a crystalline solid. This was filtered, washed and recrystallized from ethanol; light yellow needles were obtained.



A specimen of dimensions 0.3 × 0.2 × 0.1 mm was chosen. Lattice parameters and their e.s.d.'s derived from a least-squares treatment of 17 reflections (21.3 < θ < 32.1°). Intensity data were collected on an Enraf-Nonius CAD-4 single-crystal diffractometer, graphite-crystal-monochromated Cu Kα radiation, ω/2θ scan mode for h -4→9, k -9→9, l -11→12, 2θ_{max} = 140°. Two standard reflections monitored in every 100 measurements showed a random variation of less than 2% in intensity. Of the 2265 independent reflections collected, 2002 were judged significant (unique), I > 3σ(I). Intensities were corrected for Lp effects; absorption corrections were ignored. Structure was solved by Patterson methods using SHELXS86 (Sheldrick, 1986). Positional and ther-

Table 1. Fractional atomic coordinates (× 10⁴) and equivalent isotropic thermal parameters (× 10⁴) for non-H atoms in C₁₂H₁₂N₂OS, with their e.s.d.'s in parentheses

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

| | x | y | z | U _{eq} (Å ²) |
|-------|------------|------------|------------|-----------------------------------|
| S(1) | 1722.9 (6) | 1583.8 (7) | 1272.0 (5) | 376 (3) |
| O(1) | -3330 (2) | -412 (3) | 3537 (2) | 524 (7) |
| N(1) | -402 (3) | -956 (3) | 1849 (2) | 482 (8) |
| N(2) | -3139 (2) | 985 (3) | 5072 (2) | 422 (7) |
| C(2) | -44 (3) | 480 (3) | 2132 (2) | 331 (6) |
| C(3) | -965 (3) | 1257 (3) | 3092 (2) | 315 (6) |
| C(4) | -186 (2) | 2792 (3) | 3095 (2) | 304 (6) |
| C(5) | 1242 (3) | 3120 (3) | 2186 (2) | 312 (6) |
| C(31) | -2558 (3) | 564 (3) | 3905 (2) | 338 (7) |
| C(41) | -939 (3) | 3994 (3) | 3947 (3) | 426 (8) |
| C(51) | 2389 (2) | 4537 (3) | 1896 (2) | 313 (7) |
| C(52) | 2673 (3) | 5800 (3) | 482 (2) | 390 (7) |
| C(53) | 3708 (3) | 7150 (3) | 227 (3) | 449 (8) |
| C(54) | 4460 (3) | 7271 (3) | 1394 (3) | 461 (9) |
| C(55) | 4174 (3) | 6031 (3) | 2799 (3) | 446 (8) |
| C(56) | 3164 (3) | 4645 (3) | 3057 (2) | 406 (7) |

Table 2. Bond distances (Å), bond angles (°) and selected torsion angles (°), with e.s.d.'s in parentheses in C₁₂H₁₂N₂OS

| | | | |
|-----------------------|-----------|-------------------------|-----------|
| S(1)—C(2) | 1.722 (2) | C(4)—C(41) | 1.504 (4) |
| C(2)—C(3) | 1.389 (3) | C(5)—C(51) | 1.479 (3) |
| C(3)—C(4) | 1.453 (3) | C(51)—C(52) | 1.392 (3) |
| C(4)—C(5) | 1.343 (3) | C(52)—C(53) | 1.381 (3) |
| C(5)—S(1) | 1.745 (2) | C(53)—C(54) | 1.391 (4) |
| C(2)—N(1) | 1.351 (3) | C(54)—C(55) | 1.379 (4) |
| C(3)—C(31) | 1.468 (3) | C(55)—C(56) | 1.391 (3) |
| C(31)—N(2) | 1.336 (3) | C(56)—C(51) | 1.389 (3) |
| C(31)—O(1) | 1.234 (3) | | |
| C(5)—S(1)—C(2) | 92.0 (1) | C(3)—C(4)—C(41) | 124.4 (2) |
| S(1)—C(2)—C(3) | 111.5 (2) | C(41)—C(4)—C(5) | 122.2 (2) |
| C(2)—C(3)—C(4) | 111.5 (2) | C(4)—C(5)—C(51) | 129.3 (2) |
| C(3)—C(4)—C(5) | 113.3 (1) | C(51)—C(52)—C(53) | 120.8 (2) |
| C(4)—C(5)—S(1) | 111.7 (2) | C(52)—C(53)—C(54) | 119.9 (2) |
| S(1)—C(2)—N(1) | 121.0 (1) | C(53)—C(54)—C(55) | 119.6 (2) |
| C(3)—C(2)—N(1) | 127.4 (2) | C(54)—C(55)—C(56) | 120.7 (2) |
| C(2)—C(3)—C(31) | 120.5 (2) | C(55)—C(56)—C(51) | 119.8 (2) |
| C(3)—C(31)—O(1) | 120.9 (2) | C(56)—C(51)—C(52) | 119.2 (2) |
| N(2)—C(31)—O(1) | 120.5 (2) | S(1)—C(5)—C(51) | 119.0 (1) |
| C(31)—C(3)—C(4) | 127.9 (1) | C(5)—C(51)—C(52) | 121.2 (1) |
| C(5)—S(1)—C(2)—C(3) | -0.3 (2) | C(41)—C(4)—C(5)—C(51) | 3.9 (3) |
| C(2)—S(1)—C(5)—C(4) | 0.1 (2) | S(1)—C(5)—C(51)—C(52) | 56.2 (2) |
| S(1)—C(2)—C(3)—C(4) | 0.4 (2) | C(4)—C(5)—C(51)—C(56) | 54.1 (3) |
| N(1)—C(2)—C(3)—C(31) | -2.1 (4) | C(56)—C(51)—C(52)—C(53) | -0.1 (3) |
| C(2)—C(3)—C(4)—C(5) | -0.3 (3) | C(52)—C(51)—C(56)—C(55) | 1.5 (3) |
| C(31)—C(3)—C(4)—C(41) | -1.2 (3) | C(51)—C(52)—C(53)—C(54) | -0.8 (3) |
| C(2)—C(3)—C(31)—O(1) | -15.8 (3) | C(52)—C(53)—C(54)—C(55) | 0.4 (3) |
| C(4)—C(3)—C(31)—N(2) | -19.8 (3) | C(53)—C(54)—C(55)—C(56) | 0.9 (3) |
| C(3)—C(4)—C(5)—S(1) | 0.0 (2) | C(54)—C(55)—C(56)—C(51) | -1.9 (3) |

mal parameters of non-H atoms refined by full-matrix least-squares refinement on F using SHELX76 (Sheldrick, 1976). The H atoms located from a difference Fourier map were refined isotropically. At the final stage of refinement, weights were introduced resulting in R = 0.05, wR = 0.07, w = 7.3172 × 1/(σ²|F_o| + 0.000188|F_o|²). Shift/e.s.d. < 0.1 for 193 parameters. Final difference Fourier peaks less than 0.08 e Å⁻³. The atomic scattering factors used for all the atoms were as provided in the

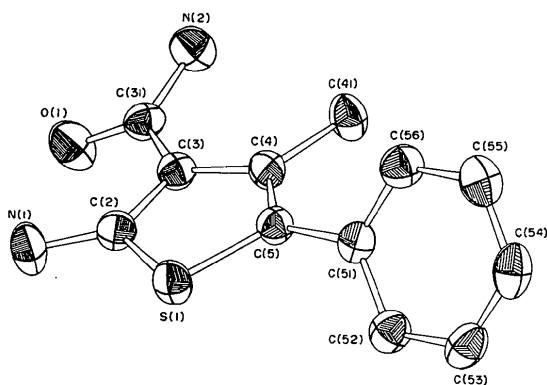


Fig. 1. Perspective view of the molecule showing atomic nomenclature.

SHELX76 program. Table 1* lists the final atomic coordinates and equivalent isotropic thermal parameters of non-H atoms. The numbering scheme and molecular connectivity are given in the perspective view of the molecule (Fig. 1). The bond lengths, bond angles and selected torsion angles are listed in Table 2.

Related literature. In 2,5-bis(4-nitrophenyl)-3,4-diphenylthiophene (Meester, Maldar, Hosmane & Chu, 1986) the dihedral angle between the phenyl and thiophene rings is -72.0° and that between the nitrophenyl and thiophene rings is 26.6° . The dihedral angle between the phenyl and thiophene

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, additional torsion angles, equations to selected least-squares planes, hydrogen bonding geometry and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53451 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1991). **C47**, 676–678

Structure of 2-Hydroxy-2-phenyl-2-phthalidylacetophenone

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(Received 17 March 1990; accepted 7 August 1990)

Abstract. $C_{22}H_{16}O_4$, $M_r = 344.4$, triclinic, $P\bar{1}$, $a = 10.684(2)$, $b = 9.455(1)$, $c = 9.492(1)$ Å, $\alpha = 104.18(2)$, $\beta = 90.02(2)$, $\gamma = 110.36(2)^\circ$, $V = 867.7(3)$ Å³, $Z = 2$, $D_x = 1.318$ Mg m⁻³, $\lambda(\text{Cu K}\alpha) = 1.5184$ Å, $\mu = 6.99$ cm⁻¹, $F(000) = 360$, room

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rings is 70.23° in 2-amino-3-ethoxycarbonyl-4-phenylthiophene (Joseph, Kannan & Parthasarathi, 1990).

Biological activities of 2-aminothiophenes and thieno[2,3-*d*]pyrimidine have attracted the attention of medicinal chemists (Nakanishi, Imamura & Maruyama, 1970; Wellings, 1972; Manhas, Sharma & Amin, 1972). It is noteworthy that compounds possessing the carboxamido moiety show a variety of pharmacological effects (Kleeman & Engel, 1982).

The authors wish to thank Professor H. Manohar and Mr K. Vyas, Department of IPC, Indian Institute of Science, Bangalore, India, for assistance in the computations and for useful discussions. This work was supported by a grant from UGC, New Delhi, India, under the Minor Research Project to one of us (PSJ), which is gratefully acknowledged.

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temperature, $R = 0.056$ for 2420 independent observed reflections. The two asymmetric centres at C(1) and C(2) respectively determine the *syn* relative configuration of the O(1) and O(2) atoms with a torsion angle O(1)—C(1)—C(2)—O(2) of $60.8(2)^\circ$.

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