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Structure of 2-(1,2-Dihydro-2,4-diphenylquinazolin-2-yl)aniline

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Abstract. $C_{26}H_{21}N_3$, $M_r = 375{\cdot}47$, monoclinic, $P2_1/n$ (non-standard space group), $a = 9{\cdot}3686$ (5), $b = 16{\cdot}8761$ (6), $c = 12{\cdot}4874$ (5) Å, $\beta = 90{\cdot}41$ (6)°, $V = 1974{\cdot}3$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}263$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 5{\cdot}47$ cm^{-1}, $F(000) = 792$, $T = 291$ K. Final $R = 0{\cdot}0458$ and $wR = 0{\cdot}0503$ for 2083 reflections. The 1,2-dihydroquinazoline ring system is not planar, the dihydropyrimidine ring being sofa-shaped. There is an intramolecular hydrogen bond between N3 positioned in the dihydropyrimidine ring and the amino group on the aniline moiety. This hydrogen bond elongates the phenyl–NH_2 bond length, and creates a sofa-shaped six-membered ring.

Introduction. The title compound was obtained by heating (433 K, 2 h) *o*-aminobenzophenone imine, which in turn was prepared from *o*-aminobenzonitrile and phenylmagnesium bromide as described by Bergman, Brynolf, Elman & Vuorinen (1986). The compound serves as a precursor for the preparation of phenyldibenzodiazocines. An X-ray investigation became necessary since MS, NMR and IR data did not present conclusive structural evidence.

Experimental. Pale yellow transparent flat prisms were obtained from an EtOH/H_2O mixture. Dimensions of examined crystal were 0·40 × 0·23 × 0·15 mm. The diffraction experiment was performed using a Stoe four-circle diffractometer with Cu $K\alpha$ radiation. $\omega/2\theta$ scan with scan width 1·02° and scan speed 1·20–3·60° min^{-1}. Lattice parameters from least-squares refinement of setting angles of 24 reflections, $22{\cdot}04 \le \theta \le 29{\cdot}60°$, 3725 observations in the range $2{\cdot}62 \le \theta \le 69{\cdot}58°$, $-11 \le h \le 11$, $0 \le k \le 20$, $0 \le l \le 14$. Three standard reflections were monitored every 180, min., max. variation in intensity 2·1 and −3·6%. *SHELXS*86 (Sheldrick, 1986) was used to solve the structure. Full-matrix least-squares refinement minimizing $\sum w(\Delta F)^2$ with *SHELX*76 (Sheldrick, 1976). Non-H atoms anisotropic. All H atoms were located in difference Fourier maps, of which the three H atoms attached to nitrogens were kept and their positions isotropically refined. Remaining H atoms in their calculated geometrical positions with isotropic group temperature factors, 332 parameters refined using 2083 reflections with $F \ge 4\sigma(F)$ gave $R = 0{\cdot}0458$ and $wR = 0{\cdot}0503$, $w = 1{\cdot}0944/[\sigma^2(F) + 0{\cdot}0004F^2]$, max. Δ/σ for any parameter 0·001, largest and smallest density in final ΔF syntheses 0·43 and −0·13 e Å^{-3}. Atomic scattering factors from Cromer & Mann (1968) for C and N, and from Stewart, Davidson & Simpson (1965) for H atoms. *PARST* (Nardelli, 1983) was used for the geometrical calculations.

Discussion. Table 1 presents atomic coordinates and isotropic thermal parameters, Table 2 lists bond lengths and angles.* Fig. 1 shows the numbering scheme and Fig. 2 gives a stereoview of the molecule including the intramolecular hydrogen bond. Figures were drawn with *PLUTO* (Motherwell & Clegg, 1978).

The dihydroquinazoline ring system is not planar; the dihydropyrimidine ring adopts a sofa conformation, and the largest displacement from the dihydropyrimidine plane is 0·275 (3) Å (C2). The benzene ring is essentially planar but still deviates significantly from planarity; the maximum deviation is 0·009 (3) Å (C4a). The dihedral angle between these planes is 9·63 (9)°.

Bond lengths and angles in the dihydroquinazoline ring system are in fair agreement with related structures (Rogan & Williams, 1980; Hunter, Neilson & Weakley, 1985) except for N3—C4, 1·287 (3) Å. The above structures have a corresponding distance in the range 1·307–1·324 Å.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44533 (41 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates and isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = \frac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
N1	0·1138 (2)	0·8008 (1)	0·7486 (2)	3·82 (6)
C2	0·1996 (2)	0·7547 (2)	0·6731 (2)	3·37 (6)
N3	0·1074 (2)	0·6952 (1)	0·6204 (2)	3·88 (6)
C4	−0·0244 (3)	0·7120 (2)	0·6016 (2)	3·78 (7)
C4a	−0·0903 (3)	0·7867 (2)	0·6359 (2)	3·89 (7)
C5	−0·2231 (3)	0·8141 (2)	0·5991 (2)	4·76 (8)
C6	−0·2805 (3)	0·8831 (2)	0·6403 (3)	5·37 (10)
C7	−0·2070 (3)	0·9258 (2)	0·7177 (3)	5·27 (9)
C8	−0·0734 (3)	0·9006 (2)	0·7541 (2)	4·52 (9)
C8a	−0·0152 (3)	0·8310 (2)	0·7127 (2)	3·70 (8)
C21	0·2696 (2)	0·8063 (2)	0·5869 (2)	3·47 (6)
C22	0·3447 (3)	0·7711 (2)	0·5016 (2)	3·93 (8)
C23	0·4138 (3)	0·8201 (2)	0·4277 (2)	4·83 (9)
C24	0·4083 (3)	0·9012 (2)	0·4365 (3)	5·20 (9)
C25	0·3358 (3)	0·9367 (2)	0·5202 (3)	4·68 (8)
C26	0·2682 (3)	0·8886 (2)	0·5947 (2)	3·90 (7)
C21′	0·3134 (3)	0·7114 (2)	0·7391 (2)	3·61 (7)
C22′	0·4438 (3)	0·7468 (2)	0·7597 (2)	4·91 (8)
C23′	0·5466 (4)	0·7091 (2)	0·8205 (3)	5·75 (10)
C24′	0·5222 (4)	0·6355 (2)	0·8612 (3)	5·42 (10)
C25′	0·3926 (4)	0·5995 (2)	0·8419 (3)	5·88 (9)
C26′	0·2874 (3)	0·6374 (2)	0·7811 (2)	4·88 (9)
N27	0·3548 (3)	0·6896 (2)	0·4919 (2)	5·35 (8)
C41	−0·1091 (3)	0·6499 (2)	0·5443 (2)	4·32 (8)
C42	−0·0492 (3)	0·6097 (2)	0·4587 (3)	5·36 (9)
C43	−0·1243 (4)	0·5492 (2)	0·4072 (3)	6·62 (12)
C44	−0·2574 (4)	0·5284 (2)	0·4401 (4)	6·78 (13)
C45	−0·3178 (4)	0·5676 (2)	0·5241 (3)	6·91 (13)
C46	−0·2451 (3)	0·6288 (2)	0·5771 (3)	5·76 (10)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

N1–H1	0·91 (3)	C22–N27	1·384 (4)
N1–C2	1·468 (4)	C23–C24	1·373 (5)
N1–C8a	1·382 (3)	C24–C25	1·387 (4)
C2–N3	1·475 (3)	C25–C26	1·391 (4)
C2–C21	1·535 (4)	C21′–C22′	1·382 (4)
C2–C21′	1·530 (4)	C21′–C26′	1·376 (4)
N3–C4	1·287 (3)	C22′–C23′	1·378 (5)
C4–C4a	1·469 (4)	C23′–C24′	1·362 (5)
C4–C41	1·493 (4)	C24′–C25′	1·376 (5)
C4a–C5	1·401 (4)	C25′–C26′	1·395 (5)
C4a–C8a	1·402 (4)	N27–H27*A*	1·06 (4)
C5–C6	1·383 (4)	N27–H27*B*	0·96 (4)
C6–C7	1·385 (5)	C41–C42	1·387 (4)
C7–C8	1·395 (4)	C41–C46	1·388 (4)
C8–C8a	1·396 (4)	C42–C43	1·395 (5)
C21–C22	1·411 (4)	C43–C44	1·362 (6)
C21–C26	1·393 (4)	C44–C45	1·367 (6)
C22–C23	1·401 (4)	C45–C46	1·400 (5)
C2–N1–C8a	117·9 (2)	C21–C22–N27	121·3 (3)
N1–C2–C21′	106·9 (2)	C21–C22–C23	118·9 (3)
N1–C2–C21	112·9 (2)	C23–C22–N27	119·7 (3)
N1–C2–N3	109·0 (2)	C22–C23–C24	121·2 (3)
C21–C2–C21′	110·4 (2)	C23–C24–C25	120·6 (3)
N3–C2–C21′	108·6 (2)	C24–C25–C26	118·7 (3)
N3–C2–C21	108·9 (2)	C21–C26–C25	122·0 (3)
C2–N3–C4	119·3 (2)	C2–C21′–C26′	121·0 (3)
N3–C4–C41	116·2 (2)	C2–C21′–C22′	120·3 (2)
N3–C4–C4a	122·7 (3)	C22′–C21′–C26′	118·7 (3)
C4a–C4–C41	121·2 (3)	C21′–C22′–C23′	121·1 (3)
C4–C4a–C8a	116·6 (2)	C22′–C23′–C24′	120·5 (3)
C4–C4a–C5	124·2 (3)	C23′–C24′–C25′	119·1 (3)
C5–C4a–C8a	119·2 (3)	C24′–C25′–C26′	120·8 (3)
C4a–C5–C6	120·2 (3)	C21′–C26′–C25′	119·7 (3)
C5–C6–C7	120·3 (3)	C4–C41–C46	121·7 (3)
C6–C7–C8	120·6 (3)	C4–C41–C42	119·7 (3)
C7–C8–C8a	119·2 (3)	C42–C41–C46	118·6 (3)
C4a–C8a–C8	120·4 (3)	C41–C42–C43	120·6 (3)
N1–C8a–C8	122·2 (3)	C42–C43–C44	120·6 (4)
N1–C8a–C4a	117·3 (2)	C43–C44–C45	119·4 (4)
C2–C21–C26	120·8 (2)	C44–C45–C46	121·1 (4)
C2–C21–C22	120·6 (2)	C41–C46–C45	119·6 (3)
C22–C21–C26	118·5 (2)		

The aminophenyl ring deviates slightly from planarity, maximum deviation being 0·007 (3) Å (C26); the remaining two phenyl rings are planar. The aminophenyl group is rotated out of the plane of the dihydroquinazoline system by 83·8 (1)°, the 2-phenyl ring by 57·9 (1)° and the 4-phenyl ring by 46·9 (1)°.

The hydrogens of the amino group (N27) are oriented so that H27*A* forms a hydrogen bond with N3 in the dihydropyrimidine ring. The bond lengths for this amino group are C22–N27 1·384 (4), H27*A*–N27 1·06 (4) and H27*B*–N27 0·96 (4) Å. The phenyl–NH_2 distance is about 0·3 Å longer than could be expected according to Voet & Rich (1970). An elongation of this kind was also observed by Rogan & Williams (1980), and proposed to be the result of the involvement of the amino group in a hydrogen bond. The hydrogen bond weakens the N lone-pair interaction with the π system of the phenyl ring, thus relaxing the bond length. This effect should be coupled with an increase in sp^3 character for the N atom, as proven by the angles in the amino group approaching tetrahedral values. In the case of the title compound the latter effect is not seen since these NH_2 angles are in the range 117–119 (2)°, indicating sp^2 character.

Furthermore, the hydrogen-bond lengths are shorter than those reported by Rogan & Williams (1980) for an

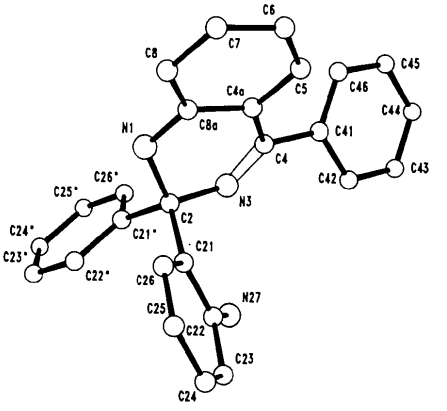

Fig. 1. Drawing of the molecule illustrating the numbering scheme. The unfilled bond indicates the double bond; H atoms are omitted for clarity.

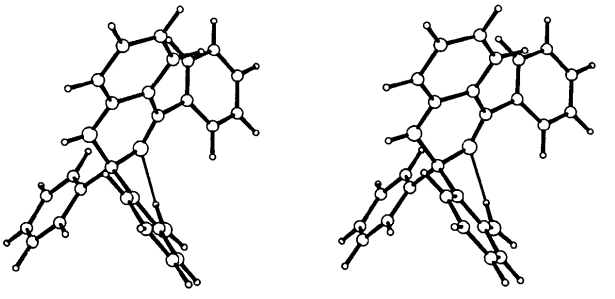

Fig. 2. Stereographic perspective view with the intramolecular hydrogen bond drawn with a thin line.

*inter*molecular bond of the NH_2—N (sp^2) type, N3···H27*A* 2·07 (4), N3···N27 2·830 (4) Å and N3···H27*A*—N27 127 (3)° for the title compound, and N···H 2·19 (5), N···NH_2 3·030 (4) Å, N···H—N 171 (2)° for the compound referenced. The intramolecular hydrogen bond completes a six-membered ring adopting a sofa conformation. The packing arrangement in the crystal shows no contacts less than the sum of the van der Waals radii.

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Structure of Dithieno[3,4-*b*:3′,4′-*d*]thiophene

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Abstract. $C_8H_4S_3$, $M_r = 196·31$, monoclinic, $P2_1/n$, $a = 15·137$ (6), $b = 5·943$ (3), $c = 9·022$ (4) Å, $\beta = 97·1$ (2)°, $V = 805$ (1) Å^3, $F(000) = 400$, $\mu = 8·07$ cm^{-1}, $D_x = 1·62$ g cm^{-3}, $Z = 4$, Mo $K\alpha$ radiation, $\lambda = 0·71073$ Å, $T = 293$ K, $R = 3·8\%$ based on 1112 independent reflections. The structure consists of the packing of planar discrete molecules. The geometric features as compared with thiophene analogues and a dithienothiophene isomer evidence a variation in 'aromaticity', according to a previously discussed trend. No short contacts are observed in the crystal packing.

Introduction. In the field of electroconducting polymers, polyheterocyclic systems have recently achieved major consideration because of their stability in air and processability and also in view of their relatively easy preparation by electrosynthesis, thus providing directly doped highly conducting materials. With the double purpose of elucidating conduction models suitable for these polymers and envisaging potential aplications, we have prepared polydithieno[3,4-*b*:3′,4′-*d*]thiophene (Bolognesi, Catellani, Destri, Zamboni & Taliani, 1987), a polymer exhibiting interesting optical and electronic properties. The understanding of these properties and their relation to the conduction process cannot disregard the structural evidence of the polymer. Moreover, *ab initio* calculations for determining electronic parameters, namely band gap, ionization potential, band width and electronic affinity, require the accurate knowledge of the system geometry. Therefore we report on the structure of the monomer dithieno[3,4-*b*:3′,4′-*d*]thiophene (DTT1) with particular reference to the analogue dithieno[3,2-*b*:2′,3′-*d*]thiophene (DTT).

Experimental. The synthesis of the title compound was carried out according to the literature (De Jong & Janssen, 1971) and the product was crystallized from a solution of 50% aqueous ethanol. In Table 1 the experimental details and the crystal data are summarized. Intensity data were collected on a Nonius CAD-4 computer-controlled diffractometer with a randomly-oriented crystal; they were subsequently corrected for Lorentz and polarization effects and an experimental absorption correction was applied (North, Phillips & Mathews, 1968). The absorption correction values range from 0·91 to 1·00. A linear correction for decay (*ca* 10% during the collection) was also applied. Unit-cell parameters and their e.s.d.'s were determined by an accurate centring of 25 strong reflections (θ range 5–12°) and then refined by least squares. The structure was solved by direct methods (*MULTAN*; Main, Lessinger, Woolfson, Germain & Declercq, 1977), using the solution with the highest figure of merit from 64 different sets (2500 relationships, $E_{min} = 1·4$). A subsequent Fourier map revealed the positions of all

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