Structure of 6-Nitro-4-phenyl-1,2-dihydroquinazoline

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Abstract. $C_{14}H_{11}N_2O_2$, $M_r = 253.26$, monoclinic, $P2_1/n$, $a = 5.9155$ (6), $b = 10.132$ (2), $c = 19.252$ (5) Å, $\beta = 94.09$ (1)°, $V = 1150.9$ (4) Å^3, $Z = 4$, $D_x = 1.46$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.109$ mm^{-1}, $F(000) = 528$, $T = 296$ K, $R = 0.052$ for 1210 observed unique reflections. The phenyl group is tilted at an angle of 55.8 (7)° with respect to the quinazoline system.

Experimental. The title compound was prepared in the course of a program directed toward the synthesis of new heterocyclic systems for pharmacological evaluation. Its preparation entails condensation of 2-amino-5-nitrobenzophenone with hexamethylenetetramine in the presence of ethyl bromoacetate (Blazevic, Kolbah, Belin, Sunjic & Kajfez, 1979). Yellow crystal of dimensions $0.15 \times 0.25 \times 0.50$ mm, D_m not measured. Rigaku AFC-6R diffractometer with graphite monochromator. Lattice parameters determined using 18 centered reflections within $33.2 \le 2\theta \le 43.8°$. Absorption ignored; $(\sin\theta/\lambda)_{max} = 0.596$ Å^{-1}, range of *hkl*: $0 \le h \le 7$, $0 \le k \le 11$, $-22 \le l \le 22$. Three standard reflections monitored every 150 reflections with an average change in intensity of 0.5% over data collection, ω–2θ scans of $16.0°$ min^{-1} in ω, 2438 unique reflections, 1210 observed $[F_o^2 > 3\sigma(F_o)^2]$.

Structure solved by direct methods with *MITHRIL* (Gilmore, 1983), *DIRDIF* (Beurskens, 1984) and analysis of successive electron density maps. H atoms located in difference maps; constrained to idealized positions with isotropic $B = 1.2 \times B$ of bonded atom. $\sum w(F_o - F_c)^2$ minimized where $w = 1/\sigma^2(F_o)$. 172 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, $(\Delta/\sigma)_{max} = 0.00$, $R = 0.052$, $wR = 0.074$, $S = 1.92$. Final difference electron density excursions -0.21 and 0.30 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974) and programs used were those from the *TEXRAY* Crystallographic Software Package (Molecular Structure Corporation, 1985). Atom numbering for Tables 1 and 2, atom coordinates and bond distances and bond angles, follows that shown in Fig. 1.* Our primary

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* 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 51763 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters and equivalent isotropic temperature factors*

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j U_{ij}.$$

	x	*y*	*z*	$B_{eq}(\text{Å}^2)$
O1	0.8826 (5)	0.1986 (3)	0.4651 (2)	4.7 (2)
O2	0.8027 (5)	−0.0042 (3)	0.4394 (2)	4.7 (2)
N1	1.6387 (6)	0.0763 (3)	0.2655 (2)	3.6 (2)
N2	1.7231 (5)	0.3096 (3)	0.2872 (2)	3.0 (1)
N3	0.9201 (6)	0.0949 (4)	0.4351 (2)	3.5 (2)
C1	1.7568 (8)	0.1932 (4)	0.2458 (2)	3.6 (2)
C2	1.5630 (6)	0.3121 (4)	0.3289 (2)	2.4 (2)
C3	1.4115 (6)	0.2004 (4)	0.3390 (2)	2.4 (2)
C4	1.2363 (6)	0.2015 (4)	0.3827 (2)	2.7 (2)
C5	1.1100 (6)	0.0904 (4)	0.3915 (2)	2.8 (2)
C6	1.1589 (7)	−0.0273 (4)	0.3592 (2)	3.5 (2)
C7	1.3351 (7)	−0.0326 (4)	0.3174 (2)	3.4 (2)
C8	1.4642 (6)	0.0807 (4)	0.3056 (2)	2.7 (2)
C9	1.5391 (6)	0.4372 (4)	0.3678 (2)	2.4 (2)
C10	1.7227 (6)	0.4889 (4)	0.4077 (2)	3.0 (2)
C11	1.7075 (7)	0.6078 (5)	0.4418 (2)	3.7 (2)
C12	1.5067 (8)	0.6770 (4)	0.4377 (2)	4.1 (2)
C13	1.3226 (7)	0.6262 (4)	0.3989 (2)	3.8 (2)
C14	1.3378 (6)	0.5073 (4)	0.3646 (2)	3.2 (2)

Table 2. *Bond distances (Å) and bond angles (°)*

| O1–N3	1.226 (4)	C4–C5	1.368 (5)
O2–N3	1.227 (4)	C5–C6	1.385 (6)
N1–C8	1.333 (5)	C6–C7	1.363 (6)
N1–C1	1.440 (5)	C7–C8	1.405 (5)
N2–C2	1.284 (4)	C9–C10	1.387 (5)
N2–C1	1.446 (5)	C9–C14	1.385 (5)
N3–C5	1.449 (5)	C10–C11	1.378 (6)
C2–C3	1.465 (5)	C11–C12	1.377 (6)
C2–C9	1.484 (5)	C12–C13	1.376 (6)
C3–C4	1.381 (5)	C13–C14	1.379 (6)
C3–C8	1.418 (5)		
C8–N1–C1	122.4 (3)	C6–C5–N3	118.9 (4)
C2–N2–C1	119.8 (3)	C7–C6–C5	119.5 (4)
O1–N3–O2	123.1 (3)	C6–C7–C8	120.7 (4)
O1–N3–C5	118.3 (4)	N1–C8–C7	121.2 (4)
O2–N3–C5	118.6 (4)	N1–C8–C3	119.7 (3)
N1–C1–N2	115.8 (3)	C7–C8–C3	119.0 (3)
N2–C2–C3	123.6 (3)	C14–C9–C10	117.9 (4)
N2–C2–C9	115.6 (3)	C14–C9–C2	121.8 (3)
C3–C2–C9	120.7 (3)	C10–C9–C2	120.2 (3)
C4–C3–C8	118.8 (3)	C11–C10–C9	121.3 (4)
C4–C3–C2	124.6 (3)	C12–C11–C10	120.2 (4)
C8–C3–C2	116.4 (3)	C11–C12–C13	119.1 (4)
C5–C4–C3	120.6 (3)	C12–C13–C14	120.7 (4)
C4–C5–C6	121.3 (3)	C13–C14–C9	120.8 (4)
C4–C5–N3	119.9 (4)		
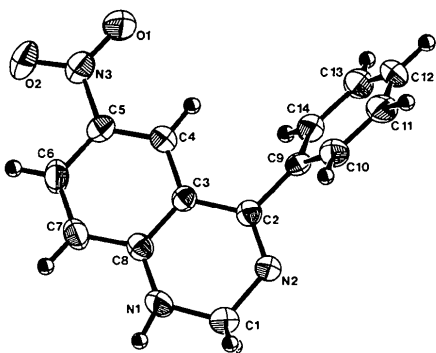
Fig. 1. *ORTEP* (Johnson, 1965) diagram of the molecule with thermal ellipsoids scaled at the 50% probability level.

Fig. 2. Packing diagram for $C_{14}H_{11}N_2O_2$.

purpose in elucidating the crystal structure (Figs. 1 and 2) of this compound was to determine whether the phenyl substituent is coplanar with the quinazoline nucleus. As can be seen from Fig. 1, the torsional angle N2—C2—C9—C10 is 55·8 (7)°, a rotation of the phenyl group that effectively takes it out of conjugation with the quinazoline system.

Related literature. In the crystal structure of the related compound 6-isopropyl-2,4-diphenylquinazoline (Hunter, Neilson & Weakley, 1985), the plane containing the C4-phenyl group is rotated by 63·9° with respect to the quinazoline ring system.

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3-Methyl-2,2,4,6-tetraphenyl-2,3-dihydro-1,3,5-triazine

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Abstract. $C_{28}H_{23}N_3$, $M_r = 401·5$, orthorhombic, *Pbca*, $a = 32·219$ (2), $b = 7·3562$ (4), $c = 18·390$ (2) Å, $V = 4358·6$ (5) Å^3, $Z = 8$, $D_x = 1·224$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 4·86$ cm^{-1}, $F(000) = 1696$, $T = 293$ K, $R = 0·060$ for 2691 unique observed reflections. In comparison with 2,2,4,6-tetraphenyl-2,3-dihydro-1,3,5-triazine N(3)—C(2) is lengthened by 0·035 (6) Å and the phenyl ring at the 4-position is rotated out of the plane composed of N(1), N(3), C(4), N(5) and C(6) by the introduction of a methyl group at the 3-position.

Experimental. The title compound was prepared from 2,2,4,6-tetraphenyl-2,3-dihydro-1,3,5-triazine (Maeda, Kihara & Ishimura, 1985). Recrystallization from benzene–hexane gave colorless column-like crystals; crystal dimensions 0·2 × 0·15 × 0·25 mm, Rigaku AFC-4 diffractometer, Cu $K\alpha$ radiation, graphite

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