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N-(3-Oxobutyl)-7-amino-4,8-dimethylquinoline

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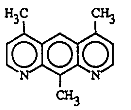
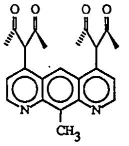
Abstract. $C_{15}H_{18}N_2O$, 4-[(4,8-dimethylquinol-7-yl)-amino]-2-butanone, $M_r = 242{\cdot}32$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}331$ (5), $b = 12{\cdot}369$ (3), $c = 8{\cdot}536$ (1) Å, $V = 1302$ (1) Å^3, $T = 296$ K, $Z = 4$, $D_x = 1{\cdot}236$, $D_m = 1{\cdot}20$ (3) Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}073$ mm^{-1}, $F(000) = 520$, $R = 0{\cdot}046$ for 1221 data with $F_o^2 > 3\sigma(F_o^2)$; $R = 0{\cdot}114$ for 2160 unique data. The quinoline moiety and the 7-amino group are nearly planar [maximum deviations 0·028 (2) and 0·042 (2) Å respectively] as well as approximately coplanar [dihedral angle 3·2 (2)°]. The conformation at the central C—C bond in the 3-oxobutyl moiety is *gauche* [N—C—C—C torsion angle −69·8 (3)°]. Bond distances and angles are normal, with the distances in the heterocyclic ring reflecting somewhat more marked alternation of single- and double-bond character. The most important intermolecular contact is a hydrogen bond between the amino group and the heterocyclic N atom at $\frac{1}{2} - x, \bar{y}, -\frac{1}{2} + z$: H···N, 2·51 (3); N···N, 3·286 (3) Å; N—H···N, 149 (2)°.

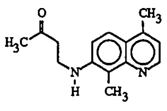
Experimental. We attempted to prepare 4,6,10-trimethylpyrido[3,2-*g*]quinoline [4,5,9-trimethyl-1,8-diazaanthracene, (I)] from 2,6-diaminotoluene using the general procedure of Campbell & Schaffner (1945) (condensation with methyl vinyl ketone, followed by cyclization and aromatization with $FeCl_3.6H_2O$). (I) was to be acetylated using the procedure of Nozawa (1986) to produce the bis(β-diketone) (II) for the construction of cofacial binuclear metal complexes (see for example Maverick, Ivie, Waggenspack & Fronczek, 1990; Bradbury, Hampton, Martone & Maverick, 1989). However, the title compound, the yellow intermediate (III), was isolated instead.



(I)



(II)



(III)

A crystal of (III) was grown from 2-propanol. An Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ source and graphite monochromator was used for data collection. Cell dimensions from least-squares fit to setting angles of 25 reflections having $10 < \theta < 11°$, ω–2θ scans, 0·69 to 4·0° min^{-1}, background and Lorentz and polarization corrections. No decay or absorption corrections necessary. Additional data collection and refinement parameters in Table 1.

Table 1. *Data collection and refinement parameters*

Crystal description	Yellow prism fragment, 0·32 × 0·42 × 0·45 mm	
T (K)	296	
θ range (°)	First octant: 1–30	Second octant: 1–25
Reflections measured	$0 \le h \le 17$	$0 \le h \le 14$
	$0 \le k \le 17$	$0 \le k \le 14$
	$0 \le l \le 12$	$-10 \le l \le -1$
	3346 total reflections in the two octants	
R_{int}	0·028	
Standard reflections	200, 040, 002	
Unique reflections	2160	
Reflections included	1221 [$I > 3\sigma(I)$]	
Parameters refined	200	
R (observed data)	0·046	
R (all data)	0·114	
wR	0·046	
S	2·248	
Δ/σ (max.)	0·06	
Max., min. residual (e Å^{-3})	0·19, −0·16	
Extinction	6(1) × 10^{-7}	

Table 2. *Coordinates and equivalent isotropic displacement parameters*

$U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3.$

	x	*y*	*z*	U_{eq}(Å^2)
O	0·1914 (2)	−0·3851 (2)	−0·1111 (3)	0·0991 (9)
N1	0·1467 (2)	0·1701 (2)	0·1542 (3)	0·0443 (6)
N2	0·1193 (2)	−0·1487 (2)	−0·1641 (3)	0·0485 (6)
C1	0·0974 (2)	0·2497 (2)	0·2256 (4)	0·0504 (8)
C2	−0·0155 (2)	0·2641 (2)	0·2347 (3)	0·0490 (8)
C3	−0·0819 (2)	0·1908 (2)	0·1646 (3)	0·0418 (7)
C4	−0·0341 (2)	0·1041 (2)	0·0830 (3)	0·0352 (6)
C5	−0·0929 (2)	0·0226 (2)	0·0044 (3)	0·0413 (7)
C6	−0·0446 (2)	−0·0583 (2)	−0·0733 (3)	0·0403 (7)
C7	0·0711 (2)	−0·0659 (2)	−0·0825 (3)	0·0363 (6)
C8	0·1335 (2)	0·0118 (2)	−0·0053 (3)	0·0369 (6)
C9	0·0826 (2)	0·0958 (2)	0·0787 (3)	0·0350 (6)
C10	−0·2039 (2)	0·2012 (3)	0·1772 (4)	0·062 (1)
C11	0·2554 (3)	0·0029 (3)	−0·0106 (4)	0·0556 (8)
C12	0·0628 (2)	−0·2299 (2)	−0·2548 (3)	0·0478 (8)
C13	0·0147 (2)	−0·3207 (2)	−0·1593 (3)	0·0454 (7)
C14	0·0968 (2)	−0·3964 (2)	−0·0872 (4)	0·0520 (8)
C15	0·0557 (3)	−0·4875 (2)	0·0093 (4)	0·0615 (9)

Space group determined by systematic absences ($h00$, h odd; $0k0$, k odd; $00l$, l odd). Initial solution by direct methods (*SHELXS*86, Sheldrick, 1985). Refinement by full-matrix least squares based upon *F*, with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$, using the *Enraf–Nonius Structure Determination Package*

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(Frenz, 1985) and scattering factors of Cromer & Waber (1974). Non-H atoms refined anisotropically, H atoms located by ΔF synthesis and refined isotropically, except for those bound to C10, C11 and C15, which were placed in calculated positions with fixed isotropic displacement parameters ($U_{\rm iso}$ = 1·3 times $U_{\rm eq}$ for attached atom). Extinction coefficient $g = 6(1) \times 10^{-7}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Final R factors and residual electron densities in Table 1.

Refined fractional coordinates and isotropic equivalent displacement parameters are listed in Table 2. Fig. 1 is an *ORTEP* drawing (Johnson, 1976), showing the atom numbering and a *PLUTO*78 (Motherwell & Clegg, 1978) packing diagram appears in Fig. 2. Bond distances and angles, and torsion angles, are presented in Table 3.*

Table 3. *Bond distances* (Å) *and bond and selected torsion angles* (°)

O—C14	1·192 (4)	C4—C9	1·443 (3)
N1—C1	1·307 (4)	C5—C6	1·340 (4)
N1—C9	1·373 (3)	C6—C7	1·431 (4)
N2—C7	1·374 (3)	C7—C8	1·397 (4)
N2—C12	1·447 (4)	C8—C9	1·410 (3)
C1—C2	1·406 (4)	C8—C11	1·508 (4)
C2—C3	1·360 (4)	C12—C13	1·510 (4)
C3—C4	1·408 (4)	C13—C14	1·509 (4)
C3—C10	1·514 (4)	C14—C15	1·484 (4)
C4—C5	1·412 (3)		
C1—N1—C9	117·1 (2)	N2—C7—C8	120·9 (2)
C7—N2—C12	125·4 (2)	C6—C7—C8	118·6 (2)
N1—C1—C2	125·5 (3)	C7—C8—C9	120·1 (2)
C1—C2—C3	119·2 (3)	C7—C8—C11	119·0 (2)
C2—C3—C4	118·2 (2)	C9—C8—C11	120·9 (2)
C2—C3—C10	120·7 (2)	N1—C9—C4	120·9 (2)
C4—C3—C10	121·1 (2)	N1—C9—C8	118·4 (2)
C3—C4—C5	124·3 (2)	C4—C9—C8	120·6 (2)
C3—C4—C9	119·0 (2)	N2—C12—C13	114·6 (2)
C5—C4—C9	116·7 (2)	C12—C13—C14	114·7 (2)
C4—C5—C6	122·7 (2)	O—C14—C13	120·9 (3)
C5—C6—C7	121·3 (2)	O—C14—C15	121·1 (3)
N2—C7—C6	120·6 (2)	C13—C14—C15	117·9 (2)
C12—N2—C7—C6	4·3 (4)	C12—C13—C14—O	−2·5 (4)
C7—N2—C12—C13	−79·8 (3)	C12—C13—C14—C15	179·6 (3)
N2—C12—C13—C14	−69·8 (3)		

Related literature. *N*-[1-(2-Pyridyl)-3-oxobutyl]-4-bromoaniline (Cameron & McQuillan, 1973); proflavine hydrate (Achari & Neidle, 1976); hydrated proflavine hemisulfate (Jones & Neidle, 1975); 2-[3-(9-acridinylamino)propylamino]ethanol (Glusker, Gallen & Carrell, 1973).

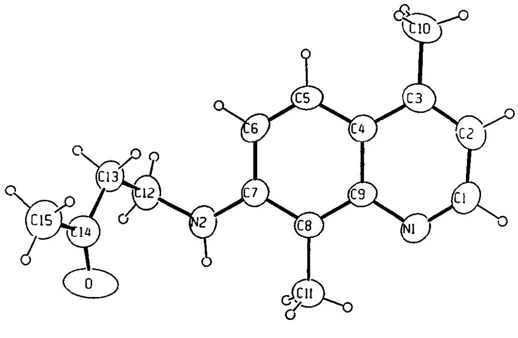

Fig. 1. *ORTEP* drawing (Johnson, 1976) of (III), representing H by circles of arbitrary radius and other atoms by 50% probability ellipsoids.

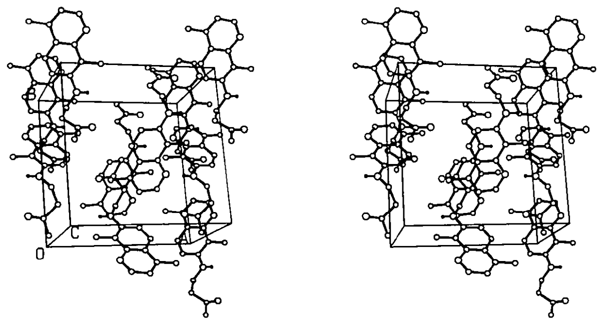

Fig. 2. *PLUTO*78 (Motherwell & Clegg, 1978) stereo packing diagram of (III). All H atoms except for H2N are omitted for clarity.

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* Lists of structure factors, anisotropic displacement parameters, distances and angles involving refined H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53409 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.