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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.124
Data-to-parameter ratio = 19.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

N-(8-Quinolyl)-o-(1,1,3,3-tetramethyl-guanidino)phenylamine

The molecular structure of the title compound, $C_{20}H_{23}N_5$, shows two strong intramolecular N—H$\cdots$N hydrogen bonds. In contrast, intermolecular interactions are weak.

Comment

The primary amino group in the ligand N-(8-quinolyl)-o-phenylendiamine can undergo several substitution reactions and a variety of Schiff base derivatives can be prepared to obtain hitherto unknown hexa- and octadentate ligands which provide access to multinuclear metal complexes.

(I)
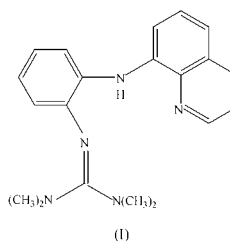
In the present study, we report the crystal structure of the title compound, (I), containing a highly basic tetramethyl-guanidine group, namely N-(8-quinolyl)-o-(1,1,3,3-tetramethylguanidino)phenylamine. The molecular structure (Fig. 1) displays the orientation of the quinolyl plane relative to the substituted phenyl ring. The angle between planes N3/C1–C9 and C10–C15 is 31.61 (5)° and the torsion angle C9—N2—C10—C11 is −17.3 (2)°. The C—N—C angle at the sp^3 atom N2 is enlarged to 129.9 (1)° and the two C—N bond lengths are similar [C9—N2 = 1.375 (2) Å and C10—N2 = 1.395 (2) Å]. The C16=N1 double bond measures 1.303 (2) Å, and the torsion angle C16—N1—C15—C10 between the guanidine moiety and the attached phenyl ring is −141.5 (1)°. This molecular geometry is accompanied by two intramolecular hydrogen bonds [N2—H2$\cdots$N1 with H$\cdots$N = 2.17 Å and N—H$\cdots$N = 106°, and N2—H2$\cdots$N3 with H$\cdots$N =
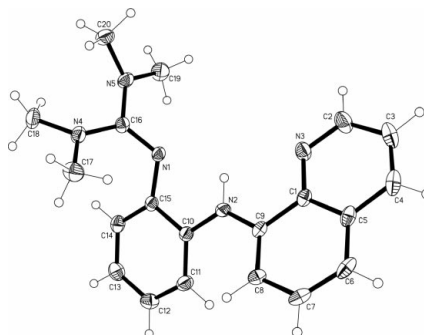
Figure 1
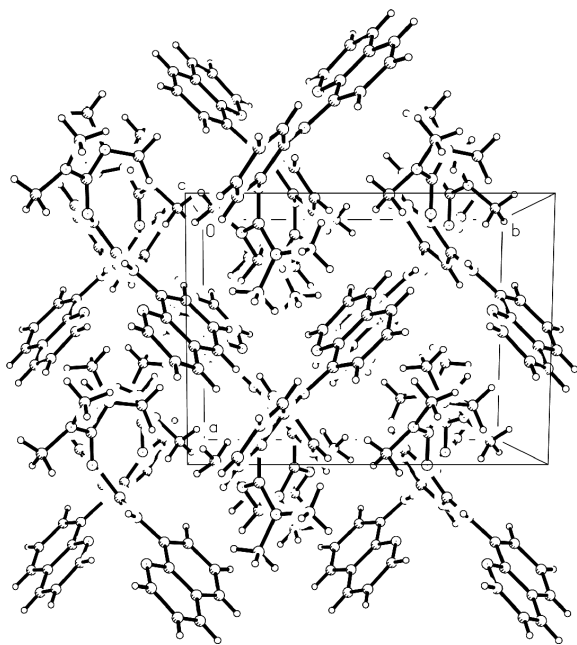The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

Figure 2
Crystal packing of (I), viewed along [001].

2.25 Å and N—H⋯H = 103°]. The shortest non-bonding intermolecular contact is C4—H4A⋯N1($1 - x, y - \frac{1}{2}, \frac{1}{2} - z$) with H⋯N = 2.52 Å. (These values have been normalized for N—H = 1.03 Å and C—H = 1.08 Å, whereas those in Table 2 are uncorrected.) Fig. 2 shows the crystal packing of (I).

Studies are now in progress to examine the coordination properties of this ligand.

Experimental

Compound (I) was prepared by transformation of the primary amine functionality in its precursor into guanidine by reacting it with Vilsmeyer salt [(Me$_2$N)$_2$CCl]Cl, which, in turn, was obtained by the reaction of tetramethylurea with phosgene (Kantlehner *et al.*, 1984). The free base was obtained by deprotonation of the resulting hydrochloride using a two-phase system of MeCN/50% aqueous KOH. Suitable crystals were obtained by slow evaporation of a saturated solution in acetonitrile. ^{1}H NMR (500 MHz, CDCl$_3$, δ): 8.66 (d, 1H, J = 1.5, 4.1 Hz), 8.56 (s, 1H), 7.93 (d, J = 1.6, 7.3 Hz), 7.51–7.43 (m, 1H), 7.43 (d, 1H, 12 Hz), 7.30–7.23 (m, 2H), 7.03 (d, 1H, 8 Hz), 6.83–6.78 (m, 2H), 6.63–6.58 (m, 1H), 2.56 (s, 12H).IR (KBr, ν [cm^{-1}]): 3317 (m), 3032 (w), 3920 (w), 2920 (w), 2885 (w), 1607 (m), 1580 (s), 1562 (s), 1513 (s), 1375 (ms), 1148 (m), 1022 (ms), 815 (m), 740 (m).

Crystal data

C$_{20}$H$_{23}$N$_5$	D_x = 1.216 Mg m^{-3}
M_r = 333.43	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2544
a = 9.2842 (5) Å	reflections
b = 12.2427 (7) Å	θ = 2.2–25.8°
c = 16.3490 (9) Å	μ = 0.08 mm^{-1}
β = 101.463 (1)°	T = 120 (2) K
V = 1821.22 (17) Å^3	Prism, pale yellow
Z = 4	0.32 × 0.25 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4531 independent reflections
φ and ω scans	3494 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	R_{int} = 0.034
T_{min} = 0.922, T_{max} = 0.991	θ_{max} = 28.3°
14968 measured reflections	h = −11 → 12
	k = −16 → 16
	l = −21 → 21

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.049	$+ 0.3663P]$
$wR(F^2)$ = 0.124	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
4531 reflections	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
230 parameters	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N2—C9	1.3746 (16)	N4—C17	1.4474 (18)
N2—C10	1.3952 (16)	N4—C18	1.4543 (18)
N1—C16	1.3025 (16)	N5—C16	1.3616 (16)
N1—C15	1.4017 (16)	N5—C19	1.4494 (19)
N4—C16	1.3715 (17)	N5—C20	1.4564 (18)
C9—N2—C10	129.89 (11)	C16—N5—C20	121.62 (12)
C16—N1—C15	120.65 (11)	C19—N5—C20	115.91 (12)
C16—N4—C17	121.42 (12)	N1—C16—N5	119.63 (12)
C16—N4—C18	121.60 (12)	N1—C16—N4	125.68 (12)
C17—N4—C18	115.95 (12)	N5—C16—N4	114.67 (11)
C16—N5—C19	119.82 (11)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2⋯N1	0.88	2.22	2.6545 (14)	110
N2—H2⋯N3	0.88	2.29	2.6808 (16)	107
C4—H4A⋯N1^i	0.95	2.65	3.5818 (18)	167

Symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

H atoms were placed at calculated positions, riding on their parent C and N atoms, with isotropic displacement parameters U_{iso}(H) = $1.2U_{eq}$(C, N) and $1.5U_{eq}$(C methyl). All methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Bruker (2002). *SMART* (Version 5.62), *SAINT* (Version 6.02), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.

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